# Interfacial Tension Measurement near Wax Appearance Temperature

By

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Dissertation submitted in partial fulfillment of

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# CERTIFICATION OF APPROVAL

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(13144)

A project dissertation submitted to the

Petroleum Engineering Programme

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In partial fulfilment of the requirement for the

BACHELOR OF ENGINEERING (Hons)

(PETROLEUM ENGINEERING)

Approved by,

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(Dr Khalik M Sabil)

UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

September 2013

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

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MOHAMMAD ABDUL HAKIM B MUSTAFFA

# ABSTRACT

This report includes the whole summary of the study of interfacial tension (IFT) measurement near wax appearance temperature (WAT) which is the title of final year project. The scope of this study is to measure three different parameters which are wax appearance temperature (WAT), density and interfacial tension (IFT) using three different equipment which are micro differential scanning calorimeter, anton paar density meter and spinning drop tensiometer.

In the literature review, it will explain about the interfacial tension effect on the temperature and other parameters, the oil-water flow behavior, wax appearance temperature and how to determine it by the past research which is by using various methods.

Besides, the problem statement of this study is to experimentally study the IFT trending near wax appearance temperature which is related to the problem statement which is lacking of experimental data of interfacial tension measurement near wax appearance temperature.

Through the methodology, this experiment will have three experiments need to be carried out which are: determining the wax appearance temperature of three different crude oils, density of oil samples, and interfacial tension of three different crude oils near wax appearance temperature. All the equipment used for this experiments is available in UTP laboratory and do not need the outside facilities.

The results that acquired from this experiment is still reasonable and the accuracy is high enough even for wax appearance temperature experiment which just repeated two times. From the result, it is shown that, interfacial tension is directly proportional to the temperature near WAT. While the density is inversely proportional to the temperature. The IFT trending that acquired is different to each other which might be due to the different composition of the crude oils.

Recommendation that can be provided here is, to carry out the experiments with more trials, repeat it using different field or region waxy crude oil and use another equipment to carry out all three experiments. As a conclusion, IFT trending every waxy crude oil is different to each other and it tends to decrease as temperature decrease.

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Mohammad Abdul Hakim Bin Mustaffa

Petroleum Engineering Department

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# CHAPTER 1

# INTRODUCTION

## 1.1 Background Study

Interfacial tension is defined as the cohesive energy present at an interface arising from the imbalance of forces which could lead to accumulation of energy. Interfacial tension happen when a molecule near an interface has different molecular interaction than an equivalent molecule within the bulk fluid. Regarding to the definition, it can be said that when IFT is low, drops tend to be smaller and flat.

For a long time ago, there are various technique that have been used in order to determine the interfacial tension (IFT) between two immiscible fluids. A lot of method has been proposed to get the most accurate value while using the easiest way in determining it. Methods that have been proposed also have their specific condition and parameter so that it will fit the required condition of that particular well or field.

Generally, in measuring interfacial tension in fluid-fluid system, there are two famous techniques that have been widely used which are pendant drop and spinning drop. Pendant drop is used to measure IFT between liquid-liquid phase and liquid-gas phase. However, spinning drop method can only measure IFT for liquid –liquid phase only. When using pendant drop, hanging pendant drop is used if the pendant drop is denser than the external phase, while inverted pendant drop is used for the drop phase which is less dense than the external phase. Therefore, in this project, spinning drop method will be used as the method to determine IFT.

Normally, IFT is expressed as mN/m which is exactly equivalent to the older units of dyne/cm (Dandekar). An interfacial measurement that detects changes over a time is known as dynamic IFT. According to Rajayi and Kantzas (2011), thermal recovery of bitumen causes numerous changes in oil-sand reservoirs and in fluid properties (viscosity, density of each fluid, and IFT and wettability of the aqueous/hydrocarbon and rock/fluid interfaces). The changes in these properties affect the production of oil sand; therefore, it is important to study them.

Similar to more conventional resource at lower pressures and temperatures, effective exploitation of depth greater than 10 000 ft, natural gas resources requires accurate description of key reservoir rock and fluid properties, including gas-water interfacial tensions to quantify the capillary pressure based vertical distribution of gas-in-place and the potential recoverable gas (Shariat, Moore, Mehta, & Van Fraassen, 2012). This shows that IFT measurement is important in oil and gas industry.

There are a lot of conditions considered in measuring interfacial tension which would satisfy the condition of the actual reservoir such as temperature and pressure. It shows that temperature have effect on the IFT measurement by plotting graph and the trend of the graph showed differences between high temperature and low temperature (Rajayi & Kantzas, 2011). Wax appearance temperature (WAT) is also considered as one of the condition where IFT could be measured. Wax appearance temperature is the particular temperature when wax crystallize during cooling process. Since there are a lot of method could be used to measure WAT, micro differential scanning calorimeter (µDSC) is the method will be used in order to calculate WAT in this project.

Transferring or movement of hydrocarbon that has been produced from well to the terminal is normally through the pipeline. For waxy oil, the wax starts to deposit in the pipeline due to the temperature drop. Temperature drops because the pipeline is located under the ocean which the temperature is lower than normal temperature. Generally, there would be multiphase fluid flow like oil and water or oil and gas at the same time in the pipeline and for some model, interfacial tension is required in calculating the pressure drop through a pipeline. That is why this study is conducted.

For Petalas and Aziz Correlation which is one of the model for multiphase flow correlation is applicable for vertical flow. Frictional pressure loss and hydrostatic pressure differences are accounted as this correlation. Each combination of gas and liquid rates are characterized by many flow regimes and one of them is bubble flow (FAST Piper, 2013). For calculating hydrostatic pressure differences of bubble flow the volumetric gas fraction is:

$$E_G = \frac{V_{SG}}{V_t}$$

Which $V_t$ is the translational bubble velocity:

$$V_t = C_0 V_m + V_b$$

Where $C_0$ is assumed to be 1.2 and $V_b$ is given as with σ is the interfacial tension value:

$$V_b = 1.41 \left[ \frac{g\,(\rho_L - \rho_G)\,\sigma}{\rho_L^2} \right]^{1/4} \sin\theta$$

The value of EG is characterized by the range where:

$$0 \le E_G \le C_G = \frac{V_{SG}}{V_m}$$

Once the volumetric gas fraction (EG) has been calculated, it is then used to calculate the mixture density (ρm). The mixture density can now be used to calculate the pressure change due to the hydrostatic head for the segment of pipe being investigated.

$$\Delta P_{HH} = \rho_m \frac{g}{g_c} \sin\theta$$

In measuring the wax appearance temperature of fluid, oil that contain wax is the key in this project. Higher content of wax in the oil would be better. However, choosing the crude oil subjected to the availability of waxy crude oil types in the UTP laboratory. Therefore, as long as the crude oil is waxy and available in UTP laboratory, it will be used as the materials for this experiment.

## 1.2 Problem Statement

Due to temperature decrease, wax deposition might happen in the pipelines during transportation. For modelling of multiphase fluid flow in those pipes, interfacial tension values is required. Based on literature on this project, there are lacking of experimental study for interfacial tension near wax appearance temperature.

In addition, literature reviews also shows that available prediction model like Parachor Method and Gradient Theory are not accurate even at normal condition. That is why this study is important to be conducted. Moreover, this project might be pilot project so that it could be used as guidance for next experiment that related in order to make improvement or reference. Therefore, highlighting the significance of measuring IFT near WAT is required so that it is worth to carry out.

## 1.3 Objectives

The main objective of carrying out this project is to experimentally study the trend of interfacial study (IFT) near wax appearance temperature (WAT). Below is the side objective that keeping the main objective is achieved:

1. To study the concept of interfacial tension and wax appearance temperature and get whole information on what factor affect IFT and WAT significantly
2. Measuring the wax appearance temperature and density for different crude oils.
3. Measuring interfacial tension for different crude oils near wax appearance temperature
4. Analysing the result to find the interfacial tension trend near wax appearance temperature.

## 1.4 Scope of Study

The scope of study for this project will be mainly related to the experiments conducted at the lab in order to get the measurement of interfacial tension near wax appearance temperature by using variety of method which is available at UTP. For this project, two major measuring experiments will be conducted as this project required the researcher to measure the wax appearance temperature and interfacial tension.

This project will be measuring the interfacial tension between two immiscible fluids which are oil and water. Therefore, there are three samples of oil to be used in this experiment and the oil sample should contain wax and high level of wax percent would be better. Therefore, the waxy crude oil that have been chosen are Dulang, Sepat 7 and Puteri. For this study, three samples is adequate to get a more reliable data.

During conducting this experiment, the parameter that should be concern in this project is the temperature. Therefore, the temperature range in measuring IFT near WAT is exactly within the boundary of wax appearance temperature. This temperature is determined by conducting the first experiment which is measuring WAT.

In calculating the wax appearance temperature, micro differential scanning calorimeter (µDSC) is used since it is available in UTP. The equipment can detect when the crystallization happening and at what temperature. While the second experiment, measuring IFT is measured using the spinning drop method which is also available in UTP laboratory. The spinning drop is used to measure the interfacial tension between two liquids unlike pendant drop which is capable of measuring IFT between liquid and gas as well. While Anton Paar Density meter is used to determine the density of crude oil samples that will be used for this experiments.

## 1.5 Feasibility Study

This project will be based on the study of determining the best method in measuring interfacial tension and wax appearance temperature. After picking up the best method, next thing to be carried out is to run the experiments to get the expected results. The tools and devices used in this project may not be exactly the same with the one that applied in the industry, but enough to make sure the objectives is met and proved. While conducting this project, there are some constraints that might need to be aware of, such as:

**Time**

The time given to complete final year project (FYP) is two 14-weeks semesters. At the end of the period given, a detail presentation will be conducted, and submission of final report to the coordinator.  In order to meet the objectives in time, the project will be focused on analysing the methods of measuring those two parameters; interfacial tension and wax appearance temperature. Almost of the time might also be spent on conducting the experiments to get the expected results.

**Tools**

After analysing all the methods that has been used in the industry, one best method will be chosen which is fit all the required parameters and conditions. However, that particular method might not be available at UTP and buying the new one might be uneconomical. Therefore, ascertaining the facilities available at UTP is crucial so that the objectives of this experiment is achieved.

## 1.6 Significance of Study

In this project, it is stressed more on measuring the interfacial tension near wax appearance temperature and there should be reason why is it so important to measure IFT near WAT. In industrial process, there are lot of process which involve two phase and create interface like emulsification, coating, and flotation. The physical properties of that phases is quantified using interfacial tension value using the contact angle.

There is a lot of previous study and journal proposed that temperature has effect on the interfacial tension. According to Flock, LE and Gibeau (1986), the effect of temperature is much greater than the effect of pressure. Flock identified that a decreased in IFT with an increase in temperature up to 90˚C for a bitumen-water system. This findings show that the same result might happen towards oil-water or oil-gas system which is strongly support why this experiments need to be carried in order to ensure either the result will turn out the same or not.

Other than that, the temperature between regions in the well is different, as example, the temperature in the wellbore and at the surface is very different. Consequently, it will affect the wax appearance temperature between the regions.

Besides, the measurements of interfacial tension at reservoir conditions are critical both as an indication of the IFT changes at reservoir harsh condition and for minimum miscibility pressure (MMP) measurement if gas is used as the EOR agent (Fars EOR Tech, n.d.). This recent technique is called Vanishing Interfacial Tension (VIT) as an alternative to very expensive and time consuming slim tube/rising bubble apparatus methods. However, in this project, it concerns more on the transportation of crude oil which involve pipeline either under the seabed or on land. The temperature of pipeline under the seabed could be as low as 25°C.

# CHAPTER 2

# LITERATURE REVIEW

## 2.1 What Is Interfacial Tension?

Interfacial tension happen whenever immiscible phases coexist in a porous medium as essentially all processes of interest, surface energy related to the fluid interfaces influences the saturations, distributions, and displacement of the phases. Molecules that are well below the surface attracted equally in all directions owing to cohesive forces, and their movement therefore tends to be unaffected by cohesive forces.

This force which is a tensile force is quantified in term of surface tension, Sigma, the force acting in the plane of the surface per unit length of the surface. Surface tension can be visualised as shown in the figure below. The term surface tension usually is reserved for the specific case in which the surface is between a liquid and its vapour or air. If the surface is between two different liquids, or between a liquid and a solid, term interfacial tension is used.

The surface tension of water in contact with its vapour at room temperature is about 73 dynes/cm while the IFT's between water and pure hydrocarbons are about 30-50 dynes/cm at room temperature. Mixtures of hydrocarbons such as crude oil will have lower IFT's that depend on the nature and complexity of the liquids. IFT and surface tensions are relatively strong functions of temperature.

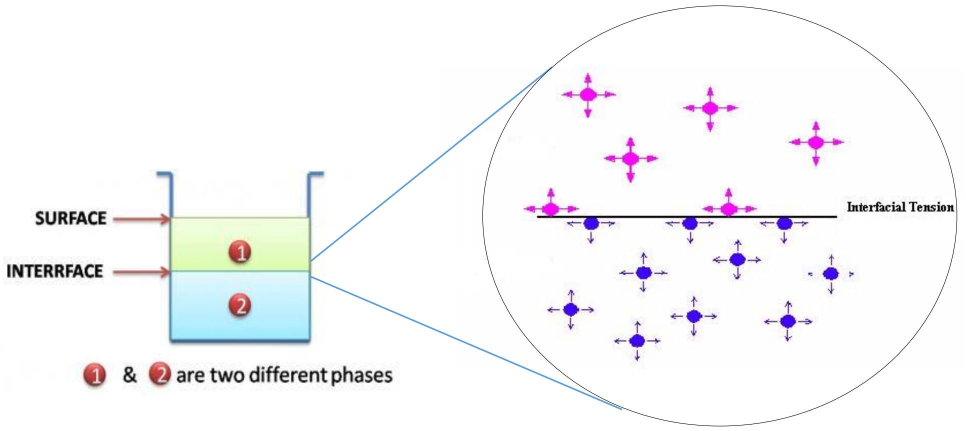


Figure 1 - Interfacial Tension

## 2.2 Measuring Interfacial Tension

Nowadays, it is very importance to determine interfacial tension for understanding of oil recovery mechanism. There are two popular technique that normally used by researcher in measuring interfacial tension and they are pendant drop and spinning drop method. Since pendant drop measurement could permits continuous study of interfacial phenomena without mechanical interference, it is considered as an important practical technique.

Over more than a century, there are various technique of measuring interfacial tension between two immiscible fluid phases have been proposed. There are five group in classical interfacial tension measurement methods which have been widely used by a lot researcher (Drelich, Fang, & White, 2002). Those five groups are direct measurement using a microbalance, measurement if capillary pressure, analysis of capillary-gravity forces, gravity-distorted drops and reinforced distortion of drop.

GROUP I: Direct Measurement Using a Microbalance

Interfacial tension between liquid and liquid produce excess of energy associated with unsaturated intermolecular interactions which tends to adopt geometries that minimize the interfacial area and this tendency can be interpreted as a physical force per unit length applied in the plane of the interface.

In order to measure the interfacial tension directly, plate, ring, rod or other probe of simple shape is used. If the probe is completely wetted by one of the liquids, this liquids will adhere to the probe and climb as the result of capillary force, increasing the interfacial area and leading to a force tending to pull tending to pull the probe toward the plane of the interface (Drelich, Fang, & White, 2002).

In direct measuring using microbalance, there are two principle techniques used for direct measurement of interfacial tension and they are Wilhelmy plate technique and Du Nouy ring method.

GROUP II: MEASUREMENT OF CAPILLARY PRESSURE

Interfacial tension can also be expressed as the work required to create a unit area of interface at a constant temperature, pressure and chemical potential. Since it is never been negative for interfaces between immiscible phases, interfacial tension always tends to decrease the area of interfaces.

Maximum bubble pressure method is the most commonly used and probably the oldest method for this group of interfacial measurement.

GROUP III: ANALYSIS OF THE BALANCE CAPILLARY AND GRAVITY FORCES

The most famous method for this group of interfacial tension measurement are capillary rise method and drop volume or weight techniques. Measuring of the interfacial tension is based on the height of meniscus in a known radius of round glass tube. If the tube diameter is small, the shape of the meniscus will be spherical while if the diameter of the tube diameter is big, the meniscus is not spherical. Even though capillary rise method could be claimed as the best and the most accurate method, there are still disadvantage of this method which is the technical problem.

Besides, drop volume or weight is also located under this group of measurement which the weight or volume of the drop falling from capillary with radius of r is measured. The number of droplets released from the capillary can be measured precisely using the modern instrumentation nowadays. Measuring interfacial tension using this kind of method is very simple yet sensitive to vibration on the other side.

GROUP IV: ANALYSIS OF GRAVITY-DISTORTED DROPS

This group of measurement is the most famous group since it is consist of pendant drop method and sessile drop method. Two parameters of the pendant drop that should be experimentally determined are the equatorial diameter D and the diameter d at the distance D from the top of the top (Drelich, Fang, & White, 2002). The interfacial tension then calculated using equation. Like other methods, pendant drop method requires clean needle to obtain a good quality and reproducible results. This needle was made of stainless steel and glass that are relatively easier to clean using acids.

Sessile drop method is based on the analysis of the profile of the drop sitting on a solid substrate and it is recommended that substrates used in sessile drop measurements be poorly wetted by the drop.

GROUP V: REINFORCED DISTORTION OF DROP

The last group applies two methods of spinning drop method and micropipette. Spinning drop method implies that gravitational acceleration has a very little impact on the shape of a fluid drop suspended in a liquid. This kind of method has successful in the measuring of ultralow interfacial tension as low as $10^{-6}$.

However, micropipette technique directly measure the interfacial tension of micro-sized droplets. The interfacial tension is calculated form the minimum pressure at which the droplets extends a hemispherical protrusion into the pipette.

Since pendant drop tends to become small and flat at low IFT, not every method is applicable to determine the interfacial tension of the phases. Due to the difficulties, there is one new method has been developed, tested and compared with the result of the old method. This difficulties is due to the diameter of the drop (Ds) at the elevation of equator diameter (De) from the apex is strongly affected by the presence of the tip (Guo & Schechter, 1997).

For the past six decades, IFT information from the drop shape was extracted from six type of method likes shape factor method, inflection method, regression method, direct method, spline-fitting method and growing-drop method. This new method introduced was more accurate for low level of interfacial tension (IFT < 1mN/m).

There are two main parameters required to assess an enhanced oil recovery (EOR) technique in terms of its efficiency which are interfacial tension and surface (wettability) alteration (Stukan & Abdallah, 2012). Contact angle measurement on reservoir rocks could be measured best using novel protocol of determining the effect of water composition on surface alteration using much simpler and less preparation sensitive.

During high temperature of the thermal recovery of bitumen will cause numerous change in fluid properties including IFT. Therefore, Rotenberg et al. (1983) proposed a new computational procedure for determining values of IFT and contact angle from the shape of axisymmetric fluid interfaces (ADSA) (Rotenberg, Boruvka, & Neumann, 1983). MicroCT scanner is required to study the contact angle of a drop of water or a bubble of gas inside the oil phase so that it is possible to see through the oil as the bulk fluid and investigate the IFT and contact angle of the water as a sessile drop on a quartz plate.

Measuring interfacial tension using spinning drop tensiometer M6500 is more specifically used to determine IFT value between surfactant and oil. It can also determine the optimal surfactant's concentration, optimal salinity and type of additives so that oil is miscible in the surfactant solution (Hsia, 2013).

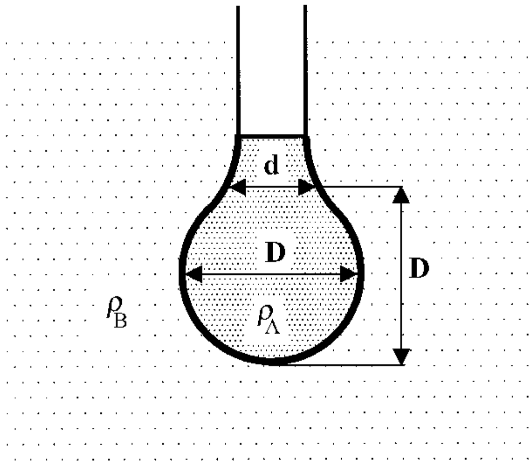


Figure 2 - Pendant Drop

## 2.3 Wax Appearance Temperature

Wax appearance temperature (WAT) is the temperature at which visible crystallization occurs and it depends on the waxes and the chemical nature of the non-waxy part of the crude oil, termed the hydrocarbon matrix (Mustafa, et al., 1996). This process normally occurs during the cooling process of oil transportation and during the fluid flow in the well. Therefore, it should be some experiments with complete procedure on how to determine the temperature of wax starts to crystallise.

## 2.4 Determination of Wax Appearance Temperature

Numerous of ways available to determine the temperature when wax stats to crystallize. One of the way to determine the WAT is by using differential scanning calorimeter (DSC). DSC is operated using a computer-controlled apparatus. Melting point and heat of melting is use for calibration of temperature and heat flow which the apparatus is flushed with argon then. The temperature range for cooling experiments is between 80-20 ˚C for heavy crude oil.

Other than that, *thermomicroscopy* requires a microscope that equipped with polarized light and phase contrast device and a hot stage unit in order to operate between the temperature ranges of 300˚C to -70 ˚C. Deviation of signal will be obtained when crystal appeared due to the variation of light intensity transmitted to the sample. This process is run using an apparatus called photomonitor.

Besides, WAT could also be determined using the fluid properties of viscosity using a method named viscometry. For a Newtonian region, kinetic viscosity is used to measure using glass capillary viscometer tubes. Hile for non-Newtonion region, apparent viscosity will be used and it is measured using rotational viscometer equipped with a cooling bath, temperature programmer, scanner and plotter. Between those three methods, the thermomicroscopic and viscometriy is the best method in measuring the WAT since.

Standard American Society for Testing and Materials (ASTM) method depends on the visual observation of the wax crystal which it requires a transparent fluid or if not, it will not be measured. In order to measure WAT of black oil or opaque, Cross Polar Microscopy and Light Transmittance are the best. Not to forget, DSC and Viscometry can also measure the WAT for black oil.

However, Cross Polar Microscopy depends on the size of the wax crystal, while Light Transmittance affected by the number of crystal. Both number and size of the wax crystal is depends much on the cooling rate.

PMAC company who is the flow assurance specialist provides the technologist with a capability to analyse live crude, at operational conditions, not only to provide the dynamic result for WAT but also for WDT ( Wax Dissolution Temperature: the temperature in which in necessary to dissolve the solid wax back into back into solution). The apparatus is modular and can be extended or enhanced to a certain capabilities.

There are two types of oil in measuring WAT, which are live oil and dead oil. Live oil means the oil that contains solution gas which is under elevated pressure while dead oil is subjected to atmospheric conditions (Uba, Ikeji, & Onyekonwu, 2004). Dead oil does not contain gas. Measurement of WAT could be used towards both type of oil but it is preferable to use dead oil in making estimation of flow assurance related to live oil because it is cheaper.

Figure 3 - PMAC Wax Appearance Temperature Apparatus

## 2.5 Waxy Crude Oil And Where Are They?

Paraffin wax and naphthenic hydrocarbon is the wax that contain in the petroleum crudes. Wax can appear in various state depending on the temperature and pressure of its surrounding. When wax reach its WAT, it will crystallise. If the paraffin wax crystallise, it is known as macrocrystalline wax, while microcrystalline is name for naphthenes whenever it is crystallised.

Waxy crude is divided into two types which are clean waxy crude oil and regular waxy crude oil. Clean waxy crude is the crude oil which consist only hydrocarbon and wax as its only heavy component (University of Illinois). However, regular waxy crude is not clean and there is some addition of asphaltene, resin and etc. which they are not crystallize during cooling process and even has no definite freezing point.

Waxy oil also turns out to be more wanted since they normally have low sulphur contents which grow some interest from an environmental viewpoint (Sifferman, 1979). Since wax is exposed as difficult to handle goods, there are lot of research done to improve flow of oil especially in the pipeline. Crude oils from China, Australasia and many other parts of the world, containing waxy crystal colloidal asphaltenes, possess distinct non-Newtonian flow properties which depend strongly on the shear and thermal history and result in special difficulties being encountered in all types of viscometers (Wardhaugh, Boger, & Tonner, 1988).

Most Australian crude oil contain high proportions of low molecular weight waxes and are pipelined at temperatures well below the pour point (Wardhaugh, Boger, & Tonner, 1988). While, one field in northeast Brazil named Pinauna Field is located shallow waters under operation of El Paso Oleo e Gas do Brasil Ltda. It was found that the API of that field is 34.5 and has peculiar characteristic under environmental conditions, due to its high paraffin conten, which provides the oil with pour point and wax appearance temperature, approximately, 105˚F and 140˚F respectively (Viana, de Goes Monteiro, & El Paso, 2011).

## 2.6 Oil-Water Flow Behaviour

Understanding the aspects of fluids flow behaviour in reservoir is mostly done by using laboratory corefloods. The mostly derived parameters from the corefloods is the oil-water relative permeability. It is also the only way to calculate for all of the rock-fluid interactions in the mathematical models developed to explain the reservoir flow phenomena. Oil-water relative permeability that calculated using reservoir cores will be analysed for discern wettability check through Craig's broad rules-of-thumb.

Values of the connate water saturations, water saturations at crossover-point of relative permeability and the end-point water relative permeability at residual oil saturation are used in that rules as base. However, the end-point oil relative permeability at connate water saturation decrease as the system becomes more oil-wet (Rao, 2002). Other than relative permeability is mainly a fluid conductance parameter in multiphase flow which depends on a few variables like wettability, rock pore structure, fluid-fluid interfacial tension, and fluid saturations.

Rao (2002) also described that it is preferable to make an independent measurement of wettability, than to rely on relative permeability interpretation alone. It is also important to obtain representative reservoir rock samples, and to restore them to native state wettability before being used in laboratory corefloods.

## 2.7 Oil-Gas Volumetric Behaviour

Correlating the volumetric behaviour of naturally occurring hydrocarbon mixtures from a single geographical area is possible with sufficient accuracy to be of industrial value. Throughout the heterogeneous region, the formation volume is larger than and distinct from the liquid formation volume. However, at the bubble point they become identical and remain so throughout the condensed liquid region, while at the dew point the liquid formation volume vanishes.

In order to get the samples of trap gas and trap liquid, forged steel container is used. It is equipped with valve ports at both ends were thoroughly purged by flushing with a large excess of the fluids collected. Once arrived at the laboratory, test will be run to check any sign of leakage and subsamples were obtained by withdrawing the hydrocarbons through drying chambers packed with granular calcium chloride into appropriate subsample containers.

During the withdrawal process, pressure within the liquid-sample containers was maintained above the initial trapping pressure by using ethylene glycol. To ensure the vaporization of any condensate that may have been present, the trap gas was withdrawn from the original sample container at a temperature above 200°F. The compositions of the trap liquid and trap gas for each field were determined by fractional distillation by forwarding the subsamples to industrial laboratories.

**2.8 Density**

Density is actually the volumetric mass density of a substance is its mass per unit volume. The symbol that normally used for density is ρ which is pronounced as rho. Normally, density is calculated using below equation:

$$\rho = \frac{m}{V}$$

where ρ is the density, m is the mass and V is the volume.

In oil and gas industry, there are two things called light crude oil and heavy crude oil. Light crude oil is the crude oil that has a low density and flow freely at room temperature. It normally has low viscosity, low specific gravity, and high API gravity due to the presence of high proportion of light hydrocarbon fraction. Commonly, it has low wax content. In term of commercial, light crude oil sold at higher price than heavy crude oil because the percentage of gasoline and diesel fuel is higher when converted into products.

However, for heavy crude oil, it is very viscous and not easily flow at room temperature even under normal pressure condition. It is recognized as heavy due to its specific gravity or density which is higher. Transportation of heavy crude oil is also contributing to some complexity and that is why it is lower in price when compared to light crude oil. There are two main types of heavy crude oils which are those that have more than 1% sulphur and those that have less than 1% sulphur.

# CHAPTER 3

# METHODOLOGY

## 3.1 Research Methodology

This project will focus more on the measurement of interfacial tension of two immiscible fluid like oil and water. Various variables and parameters need to be familiar with like interfacial tension, contact angle, cooling process of wax and other else. In order to gain more understanding before conducting the experiments, the author did research on them by reading any related reading materials towards interfacial tension measurement near wax appearance temperature. Information regarding this project is numerous since it is not a new thing in oil industry.

The experiments on this projects will consist of two significant experiments which are determining the wax appearance temperature using micro differential scanning calorimeter (µDSC) and measuring interfacial tension between two immiscible fluid using spinning drop method. Measuring the interfacial tension and determining wax appearance temperature might not bring much problems since the technology is available in Universiti Teknologi PETRONAS laboratory.

### 3.1.1 Materials

In order to conduct this experiments, there are materials required so that the objective of the study could be accomplished. For this experiments, it would only requires two materials which are Malaysian crude oils and distilled water as the solvent for conducting experiments. The crude oil required should have contain wax in order to determine its wax appearance temperature. However, Malaysian crude oil is not really significant in wax content except for certain fields. Since this experiments would require at least three sample, Dulang, Puteri, and Sepat 7 are the crude oils that might be chosen for this experiments.

In UTP laboratory, there are only one types of crude oil available which is Dulang crude oil, while the other crude oils need to be requested from oil refinery. For distilled water as the second material, it can be easily obtained from the laboratory. Below is the table of details regarding the materials:

| Type | Supplier |
|---|---|
| Dulang | Available in UTP Laboratory |
| Puteri | PETRONAS Penapisan (Melaka) |
| Sepat 7 | PETRONAS Penapisan (Melaka) |

Table 1 - Materials Details

### 3.1.2 Parameters

Throughout this experiments, there are some parameters that should be concerned and focused so that the result obtained at the end of the experiments is accurate and reliable. Pressure and temperature are two major parameters which control the result of this experiments especially the temperature. For the pressure, both IFT and WAT experiments is conducted at the atmosphere pressure as per equipment requirement.

For measuring wax appearance temperature, the temperature will be set from the crude oil appear as completely solid until it starts to crystallize. The temperature of oil starts to crystallise need to be recorded as that is the WAT of that oil. However, during calculating IFT, the temperature used to calculate it is the temperature near the wax appearance temperature.

### 3.1.3 Equipment

There are three major equipment used in this experiments. Every equipment has its own function in contributing to the accomplishment of objectives. Below are the equipements and its function:

- Micro Differential Scanning Calorimeter (µDSC)
    - Measuring Wax Appearance Temperature (WAT)
- Spinning Drop Tensiometer
    - Measuring Interfacial Tension (IFT)
- Anton Paar Density Meter
    - Measuring density of oil sample

## Equipment Working Mechanism

### Measuring Wax Appearance Temperature (WAT) using Differential Scanning Calorimeter (DSC)

µDSC will require the user to set the upper limit and lower limit of the temperature. The scanning rate is also need to be set either at heating zone or cooling zone. After that, the machine will provide the exothermic graph which will be used for WAT determination. It measures the heat flow from or to the sample when the sample is heated or cooled. For measuring wax appearance temperature, heat will flow away from sample as the cooling process to let the oil sample crystallise. It requires only a small amount of sample for DSC analysis.

Figure 4 - Crude oil sample above WAT (left) and crude oil sample below WAT (right)

Figure 5 – micro Differential Scanning Calorimeter

### Measuring Interfacial Tension (IFT) using Spinning Drop Method

This technique relies on the fact that gravitational acceleration has little effect on the shape of a fluid drop suspended in a liquid, when drop and the liquid are contained in a horizontal tube spun about its longitudinal axis. When rotational velocities (ω) is low, it takes ellipsoidal shape. When ω is high, it turns out to be cylindrical and at this time the radius of the cylindrical is recorded for IFT measurement.


Figure 6 - Spinnig Drop Tensiometer

**Measuring Density using Anton Paar Density Meter**

The measurement cell (as in the figure) oscillates at several frequencies in two modes. This allow the damping due to the sample to be measured and properly corrected. When the standard is calibrated, the density is a function of the oscillation period and the damping.

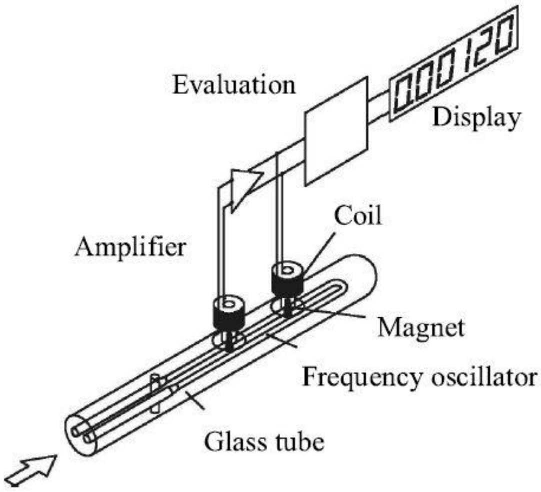


Figure 7 - Schematic Diagram of Anton Paar Density Meter

## 3.2 Project Work

**PRELIMINARY RESEARCH**

- Study on research paper
- Understand the concept and theory

**EXPERIMENTS**

- Booking of Lab Session for conducting experiments
- Preparation of apparatus and tools
- Determining the Wax Appearance Temperature (WAT) using micro Differential Scanning Calorimeter (µDSC)
- Measuring the density of oil sample using Anton Paar Density Meter
- Measuring the Interfacial Tension (IFT) near WAT using Spinning Drop Tensiometer
- Results Gathering

**DISCUSSION**

- Discuss, analyse and interpret the findings and results
- Compile all the related result and produce in hardcopy & softcopy form

### 3.3 Procedure of Experiment

#### 3.3.1 WAT Measurement Using Micro Differential Scanning Calorimeter

1. Preparation is made once a sample of crude oil is ready.
2. Oil will be transferred into the thermally safe container.
3. The crude oil is than heated in an oven for 2 hours at 80˚C. It is to make sure all the suspended wax melt.
4. After fully heated, sample of certain micro litre is injected onto DSC stainless steel pan.
5. O Ring and lid are inserted on top of pan and forming a seal by compression.
6. Maximum and minimum temperature is set up with the desired cooling rate.
7. Wait for the machine to complete the test and do analysis regarding the result.
8. Clean up and maintenance.

#### 3.3.2 IFT Measurement Using Spinning Drop Method

1. Set up the spinning drop tensiometer by switching on all the controls.
2. Calibrate all the component of the apparatus.
3. Load the solvent into the sample tube using syringe and put into the tensiometer
4. Inject the oil-droplet into the solvent and start rotation at desired speed.
5. Measure the IFT by mesasuring the oil droplets diameter from microscope eyepiece.
6. Determine the interfacial tension and run for analysis on the result obtained.
7. Turn off the rotation speed, remove the sample tube and switch off all the controls
8. Clean the sample tube

#### 3.3.3 Density Measurement Using Anton Paar Density Meter

1. Switch on the anton paar density meter and let it on for 15 minutes.
2. Heat up the crude oil sample using heater until it melts and mix very well. (If the sample is crude oil, if not just proceed with step 3)
3. Transfer the sample into the density meter using syringe.
4. Make sure there is no bubble in the U-tube to avoid any error of results.
5. Set the temperature to the required temperature. Press start and wait for the density meter to analyse the sample and show the results.
6. Clean up and maintenance.

### 3.4 Calculation of Experiment

Throughout this experiment, the only calculation involved is the calculation of interfacial tension. By using the Anton Paar density meter, it determines the density of the oil sample and spinning drop tensiometer would give the rotational velocities as

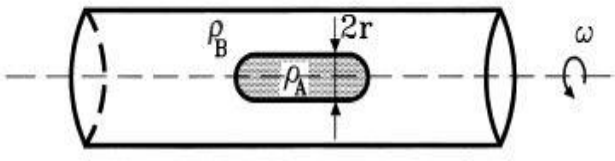


Figure 8 - Sample of Oil Drop using Spinning Drop

well as the diameter of the oil sample in order to calculate interfacial tension. With the equation provided, the interfacial tension can be calculated from the equation below:

$$\gamma = \frac{1}{4} r^3 \Delta\rho\omega^2$$

Equation 1 - Interfacial Tension Equation

### 3.5 Analysis of Data

After all the data has been gathered, it is ready to go for analysis of data. Since the main objective of this project is to experimentally study the trending of interfacial tension measurement near wax appearance temperature, it is obviously showing that the analysis of data would include making a graph in order to see the trending of the graph.

In order to set the temperature range, the reference point need to be set first which WAT is chosen. Interval of five and 10 °C above and below the WAT is used to set as the temperature near WAT. As the temperature range has been set, the interfacial tension can be find. After IFT has been found, plotting of graph using that data would show the trending of the IFT near WAT for every crude oils and combined trending of all crude oils into one graph can also be made.

## 3.6 Gantt Chart

### Final Year Project I – Gantt Chart

| ACTIVITIES | WEEK | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | **1** | **2** | **3** | **4** | **5** | **6** | **7** | **8** | **9** | **10** | **11** | **12** | **13** | **14** |
| Topic Selection | | | | | | | **MIDSEM BREAK** | | | | | | | |
| Preliminary Research Work | | | | | | | | | | | | | | |
| Submission of Extended Proposal | | | | | | | | | | | | | | |
| Proposal Defence | | | | | | | | | | | | | | |
| Preparation of Lab Work | | | | | | | | | | | | | | |
| Submission of Interim Draft Report | | | | | | | | | | | | | | |
| Submission of Interim Report | | | | | | | | | | | | | | |

Table 2 – FYP I Gantt Chart

**Final Year Project Ii – Gantt Chart**

| Training Activities | Week | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 |
| Preparation of Materials and Equipments | ■ | | | | | | | | | | | | | |
| Determining WAT using µDSC | | ■ | ■ | | | | | | | | | | | |
| Density Measurement | | | ■ | ■ | | | | | | | | | | |
| IFT Measurement | | | | ■ | ■ | | | | | | | | | |
| Data Gathering | | | | | ■ | ■ | | | | | | | | |
| Analysis of data | | | | | ■ | ■ | | | | | | | | |
| Submission of Progress Report | | | | | | ■ | ■ | ■ | | | | | | |
| Submission of Draft Report | | | | | | | | | ■ | ■ | ■ | ■ | | |
| Oral Presentation | | | | | | | | | | ■ | ■ | ■ | ■ | |
| Submission of Technical Paper | | | | | | | | | | ■ | ■ | ■ | ■ | ■ |
| Submission of Dissertation | | | | | | | | | | ■ | ■ | ■ | ■ | ■ |

Table 3 - FYP II Gantt Chart

# CHAPTER 4

# RESULT & DISCUSSION

Earlier in the problem statement, it has been stated that this project lack of experimental study regarding the interfacial tension near wax appearance temperature. Therefore, the result of this study could not be compared with the previous study. However it can be used as the reference for the future study. This study has been completed within 28 weeks provided by the coordinator. This project has two major experiments which are determining wax appearance temperature and measuring the interfacial tension near wax appearance temperature. Both of the data are presented in the table and graph format. The same goes to the determination of density for interfacial calculation purpose which the data is also presented in table form.

As all the data has been gathered and interpreted, analysis of data will be carried out using the graph to see the trending of interfacial tension near wax appearance temperature. As the trending of provided by graph, it shows that the objective of this study is met.

The discussion about the data gathered are explained at the below of the graph. It is easier to gain understanding with direct explanation provided below the table or figure provided.

## 4.1 Determination of Wax Appearance Temperature Using Micro Differential Scanning Calorimeter

Determining the wax appearance temperature using micro differential scanning calorimeter is easy since all the analysis of data is made by the computer and software. However, the interpretation part involve the understanding of exothermic concept. Exothermic means the process of getting rid of heat during the cooling process and wax appearance temperature means the temperature at which the samples starts to crystallize during the cooling process.

Endothermic is not related to WAT determination since it is happening during the heating process. Data provided by the software regarding the exothermic and endothermic is in graph format. Interpretation of the graph will show the WAT of the samples.

### 4.1.1 Dulang Wax Appearance Temperature Determination

**First Trial**

Experiment: WAT Dulang

Apparatus: Micro DSCVII CS Evol.

Initial Mass: 50 (mg)

Molar Mass: Not Applicable

Cell: Standard Hastelloy

Zone Number: 2

Zone: Cooling zone

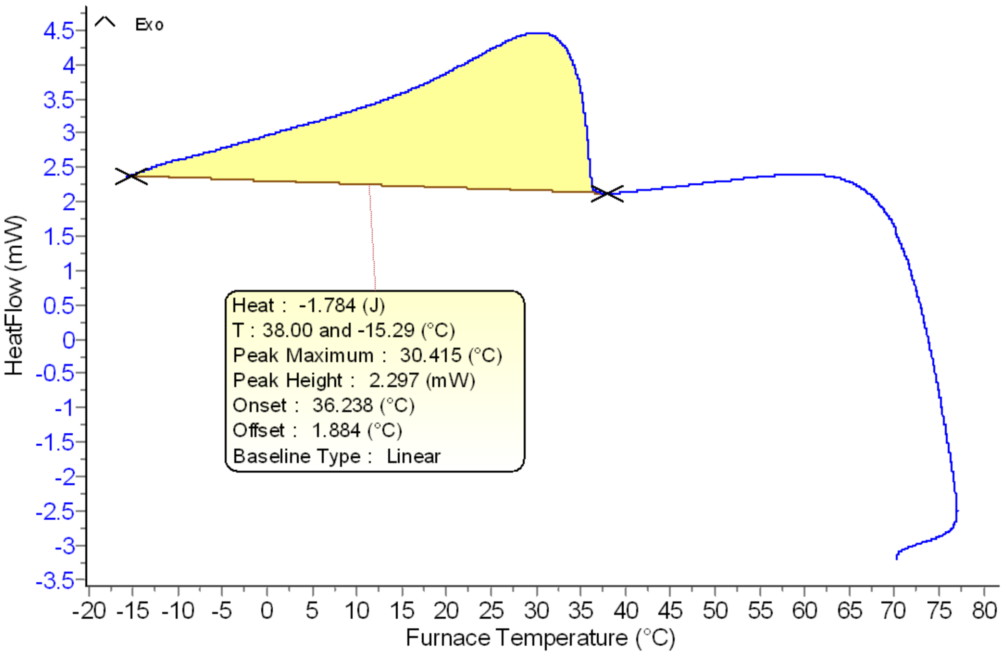


Figure 9 - Exothermic graph of Dulang First Trial

**Discussion:**

Above is the data that has been interpreted from a raw data. According to the above data, the process of cooling starts from 70°C until -15°C and exothermic region is shown by the yellow region. From the information box, the onset temperature is the temperature at which sample starts to crystallize. Therefore, the WAT for Dulang is 36°C. Other parameter provided in the box is not under the concern of this experiment.

**Second Trial**

Experiment: Dulang TRIAL 2 24.10.2013

Apparatus: Micro DSCVII CS Evol.

Initial Mass: 21 (mg)

Molar Mass: Not Applicable

Cell: Standard Hastelloy

Zone Number: 2

Zone: Cooling zone

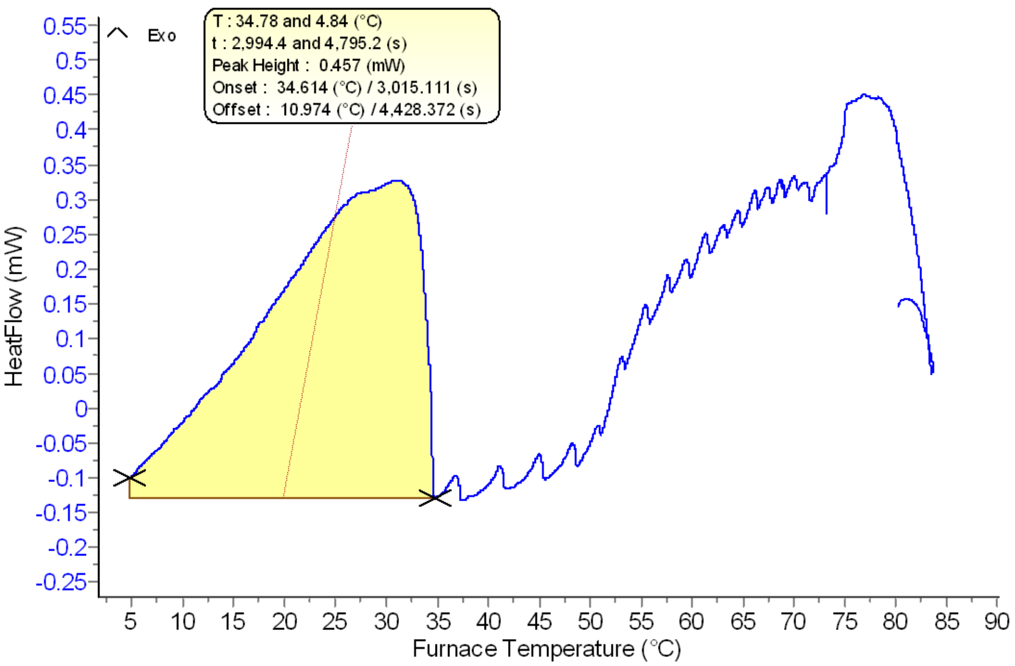


Figure 10 - Exothermic Graph of Dulang Second Trial

**Discussion:**

From the information box provided, it is clearly stated that for the second trial, the onset temperature of the exothermic region is 34°C. Before the exothermic region, there a lot of fluctuating graph from 85°C until 34°C and that is noise which is contributed from the experiments. This noise can be ignored since the results still give reasonable reason to compare with the first trial. In order to get a better result, average of this results is used to consider as the WAT of Dulang crude oil.

**4.1.2 Sepat 7 Wax Appearance Temperature Determination**

**First Trial**

Experiment: Sepat 7 WAT

Apparatus: Micro DSCVII CS Evol.

Initial Mass: 100 (mg)

Molar Mass: Not Applicable

Cell: Standard Hastelloy

Zone Number: 2

Zone: Cooling zone

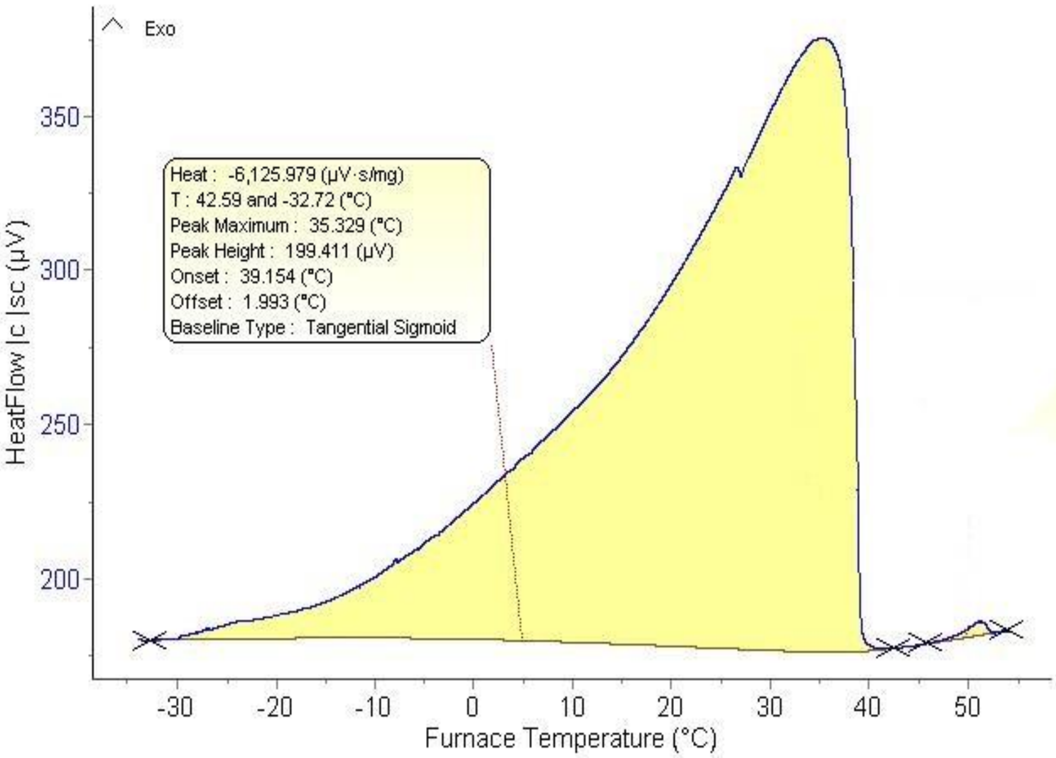


Figure 11 - Exothermic Graph of Sepat 7 First Trial

**Discussion:**

By referring to the information box, the onset temperature of the exothermic region is 39°C which is slightly higher than the onset temperature of Dulang crude oil sample meaning that the WAT of Sepat 7 is higher than the WAT of Dulang. However, the difference is small which just 4°C if the average value of Dulang crude oil is taken.

**Second Trial**

Experiment: Sepat 7 TRIAL 2 25.10.2013

Apparatus: Micro DSCVII CS Evol.

Initial Mass: 42 (mg)

Molar Mass: Not Applicable

Cell: Standard Hastelloy

Zone Number: 2

Zone: Cooling zone

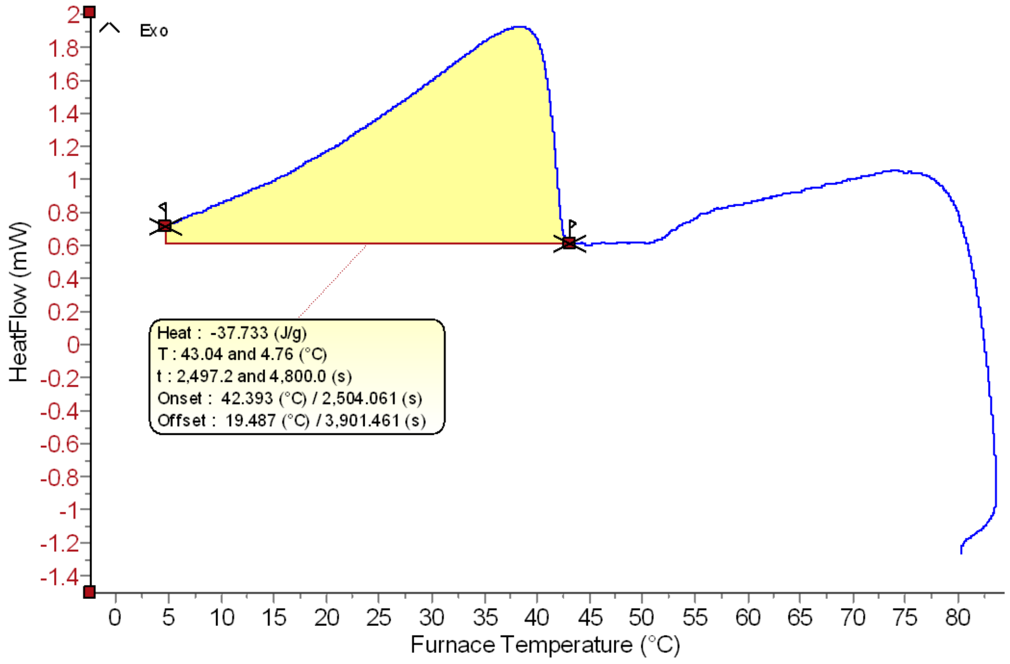


Figure 12 - Exothermic Graph of Sepat 7 Second Trial

**Discussion:**

According to the information box in the figure above, the onset temperature which showing the WAT of this sample is 42 °C. The result is still reasonable since the difference with the previous trial is still small which just 3°C. If the average value between these two is taken, it would be 40.5°C.

### 4.1.3 Puteri Wax Appearance Temperature Determination

**First Trial**

Experiment: PUTERI TRIAL 1 21.10.2013

Apparatus: Micro DSCVII CS Evol.

Initial Mass: 90 (mg)

Cell: Standard Hastelloy

Zone Number: 2

Zone: Cooling zone

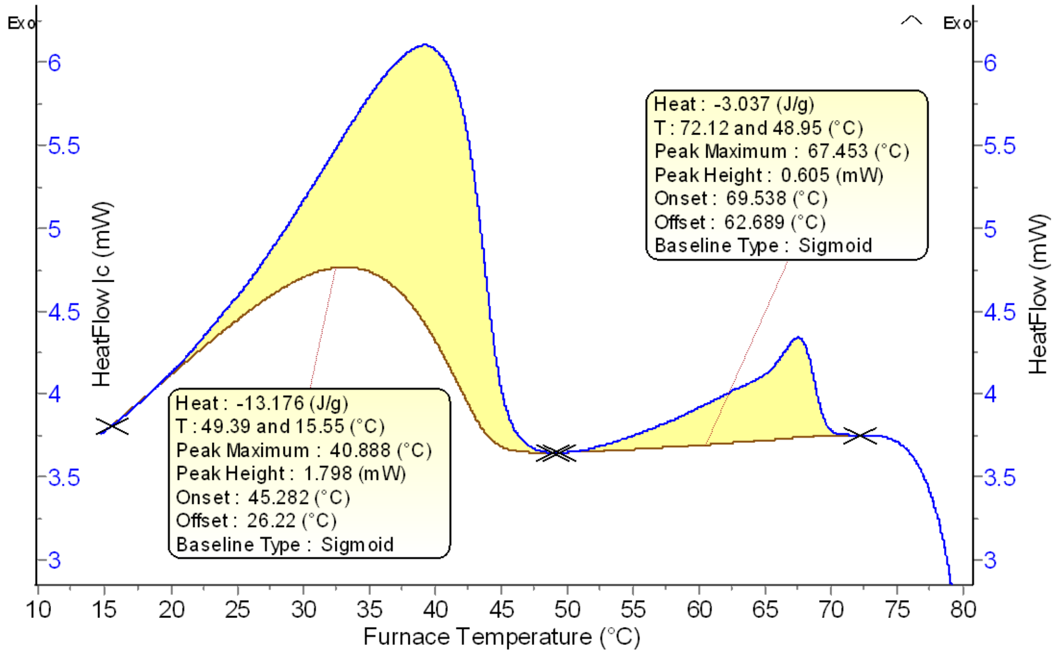


Figure 13 - Exothermic Graph of Puteri First Trial

**Discussion:**

After interpretation process, the onset temperature of the first trial of Puteri crude oil is 45°C. Among all three crude oil samples, this sample has the highest value of WAT. That is why it takes longer time and higher temperature to be melted before conducting this experiment. It is also more difficult to be cleaned after conducting the experiment.

**Second Trial**

Experiment: Puteri TRIAL 2 25.10.2013

Apparatus: Micro DSCVII CS Evol.

Initial Mass: 37 (mg)

Molar Mass: Not Applicable

Cell: Standard Hastelloy

Zone Number: 2

Zone: Cooling zone

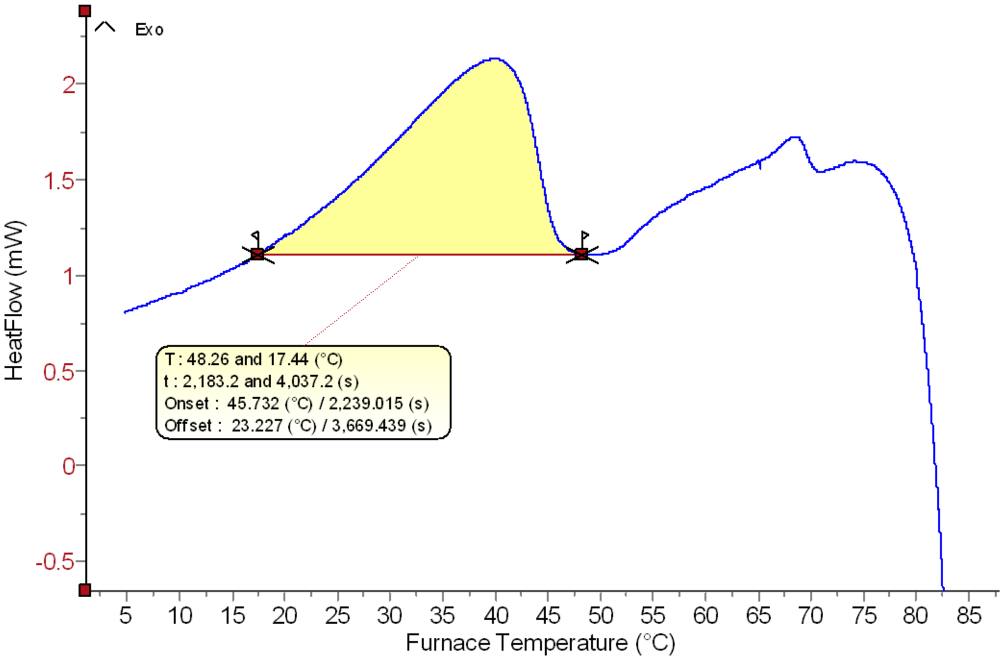


Figure 14 - Exothermic Graph of Puteri Second Trial

**Discussion:**

From the figure above, it shows that the value of WAT for the second trial of Puteri crude oil is also 45°C. For this sample, there is consistency in shape between these two trials. However, the second trial is a zoom out version of the first trial since the shape is not much different. Therefore, if the average value of this sample is the same since it gives same value for both trial.

### 4.2 Determination of Density Using Anton Paar Density Meter

In order to calculate the value of interfacial tension of crude oil samples, the value of their density are required so that it can be substituted into the formula. The formula has been shown in the calculation part previously. For this part of experiment, the samples involved are Dulang cude oil, Speat 7 crude oil, Puteri crude oil and distilled water. Distilled water is used as the solvent during the interfacial tension experiment and it required the value of density for solvent and sample.

Since the objective of this experiment is to experimentally study the interfacial tension trending near wax appearance temperature, the temperature is the variable that going to be controlled. All the value is determined three times and average value for all three trials has been calculated as in the table. Below is the data for density value of four samples at various temperature:

#### 4.2.1 Dulang Crude Oil Density

| Dulang | |
|---|---|
| Temperature | Density (g/cm$^3$) |
| 30°C | 0.82667 |
| 35°C | 0.82326 |
| 40°C | 0.81984 |
| 45°C | 0.81643 |
| 50°C | 0.813 |
| 55°C | 0.80958 |
| 60°C | 0.80617 |

Table 4 - Dulang Crude Oil Density

#### 4.2.2 Sepat 7 Crude Oil Density

| Sepat 7 | |
|---|---|
| Temperature | Density (g/cm3) |
| 30°C | 0.84039 |
| 35°C | 0.83694 |
| 40°C | 0.83352 |
| 45°C | 0.83009 |
| 50°C | 0.82664 |
| 55°C | 0.82318 |
| 60°C | 0.81974 |

Table 5 - Sepat 7 Crude Oil Density

### 4.2.3 Puteri Crude Oil Density

| Puteri | |
|---|---|
| Temperature | Density (g/cm3) |
| 30°C | 0.8717 |
| 35°C | 0.8679 |
| 40°C | 0.86403 |
| 45°C | 0.85716 |
| 50°C | 0.85325 |
| 55°C | 0.84972 |
| 60°C | 0.84576 |

Table 6 - Puteri Crude Oil Density

### 4.2.4 Distilled Water Density

| Distilled Water | |
|---|---|
| Temperature | Density (g/cm3) |
| 30°C | 0.99398 |
| 35°C | 0.99071 |
| 40°C | 0.98761 |
| 45°C | 0.98359 |
| 50°C | 0.98209 |
| 55°C | 0.9777 |
| 60°C | 0.97219 |

Table 7 - Distilled Water Density

**Discussion:**

Among all four samples, Puteri crude oil has the highest value of density and it is also the highest wax appearance temperature value recorded. It can be said that high density sample give high wax appearance temperature value.

### 4.3 Determination of Interfacial Tension of Three Crude Oils near The Wax Appearance Temperature

Interfacial tension of three crude oils are determined using the spinning drop method. The spinning drop method would use spinning drop tensiometer which is the result will be calculated automatically after all the required information is keyed in into the software. Before conducting this experiment, the variable of this temperature has been set which is the temperature.

In order to set up the required temperature, the reference to set the temperature is the wax appearance temperature which are different for every crude oil. It means every crude oil has different range of temperature. As example, if the WAT of X oil is 20°C, the interval between higher and lower temperature of that WAT is 5°C. Therefore, it will be 25°C and 15°C. The highest and lowest limit of the temperature set is 10°C interval from WAT. So for X oil, its upper and lower limits are 30°C and 10°C.

Below is the results of interfacial tension among all three crude oils used at the temperature near the WAT:

#### 4.3.1 Dulang Interfacial Tension Near WAT

| Dulang | |
|---|---|
| **Temperature (°C)** | **IFT (mN/m)** |
| 45 | 18.8856 |
| 40 | 18.1600 |
| 35 | 14.5429 |
| 30 | 13.0745 |
| 25 | 12.9096 |

Table 8 - Dulang IFT near WAT

#### 4.3.2 Sepat 7 Interfacial Tension Near WAT

| Sepat 7 | |
|---|---|
| **Temperature (°C)** | **IFT (mN/m)** |
| 50 | 19.5494 |
| 45 | 18.9302 |
| 40 | 17.7416 |
| 35 | 17.4244 |
| 30 | 17.5399 |

Table 9 - Sepat 7 IFT near WAT

### 4.3.3 Puteri Interfacial Tension Near WAT

| Puteri | |
|---|---|
| **Temperature (°C)** | **IFT (mN/m)** |
| 55 | 26.0281 |
| 50 | 25.5428 |
| 45 | 21.5955 |
| 40 | 21.1763 |
| 35 | 21.6039 |

Table 10 - Puteri IFT near WAT

**Discussion:**

The column that have red square around it indicating that the column is the wax appearance temperature of that crude oil with its interfacial tension value. Therefore, the value above and below the wax appearance temperature column is the temperature near the wax appearance temperature.

## 4.4 Summary of Results

With all the results provided before, here is the summary of the result that can be used as a quick reference.

### 4.4.1 Wax Appearance Temperature Value of Three Samples

| Dulang | | | Sepat 7 | | | Puteri | | |
|---|---|---|---|---|---|---|---|---|
| Trial No | WAT Value (°C) | Average Value (°C) | Trial No | WAT Value (°C) | Average Value (°C) | Trial No | WAT Value (°C) | Average Value (°C) |
| 1 | 36 | 35 | 1 | 39 | 40.5 | 1 | 45 | 45 |
| 2 | 34 | | 2 | 42 | | 2 | 45 | |

Table 11 - WAT Value of Three Samples

### 4.4.2 Density Value of Four Samples

| Dulang | | Sepat 7 | | Puteri | | Distilled Water | |
|---|---|---|---|---|---|---|---|
| Temperature | Density (g/cm3) | Temperature | Density (g/cm3) | Temperature | Density (g/cm3) | Temperature | Density (g/cm3) |
| 30°C | 0.82667 | 30°C | 0.84039 | 30°C | 0.8717 | 30°C | 0.99398 |
| 35°C | 0.82326 | 35°C | 0.83694 | 35°C | 0.8679 | 35°C | 0.99071 |
| 40°C | 0.81984 | 40°C | 0.83352 | 40°C | 0.86403 | 40°C | 0.98761 |
| 45°C | 0.81643 | 45°C | 0.83009 | 45°C | 0.85716 | 45°C | 0.98359 |
| 50°C | 0.813 | 50°C | 0.82664 | 50°C | 0.85325 | 50°C | 0.98209 |
| 55°C | 0.80958 | 55°C | 0.82318 | 55°C | 0.84972 | 55°C | 0.9777 |
| 60°C | 0.80617 | 60°C | 0.81974 | 60°C | 0.84576 | 60°C | 0.97219 |

Table 12 - Density Value of Four Samples

### 4.4.3 Interfacial Tension near Wax Appearance Temperature for Three Crude Oils

| Dulang | | Sepat 7 | | Puteri | |
|---|---|---|---|---|---|
| Temperature (°C) | IFT (mN/m) | Temperature (°C) | IFT (mN/m) | Temperature (°C) | IFT (mN/m) |
| 45 | 18.8856 | 50 | 19.5494 | 55 | 26.0281 |
| 40 | 18.1600 | 45 | 18.9302 | 50 | 25.5428 |
| 35 | 14.5429 | 40 | 17.7416 | 45 | 21.5955 |
| 30 | 13.0745 | 35 | 17.4244 | 40 | 21.1763 |
| 25 | 12.9096 | 30 | 17.5399 | 35 | 21.6039 |

Table 13 - IFT near WAT for Three Crude Oils

## 4.5 Analysis of Results

### 4.5.1 Dulang Interfacial Tension Trend near Wax Appearance Temperature

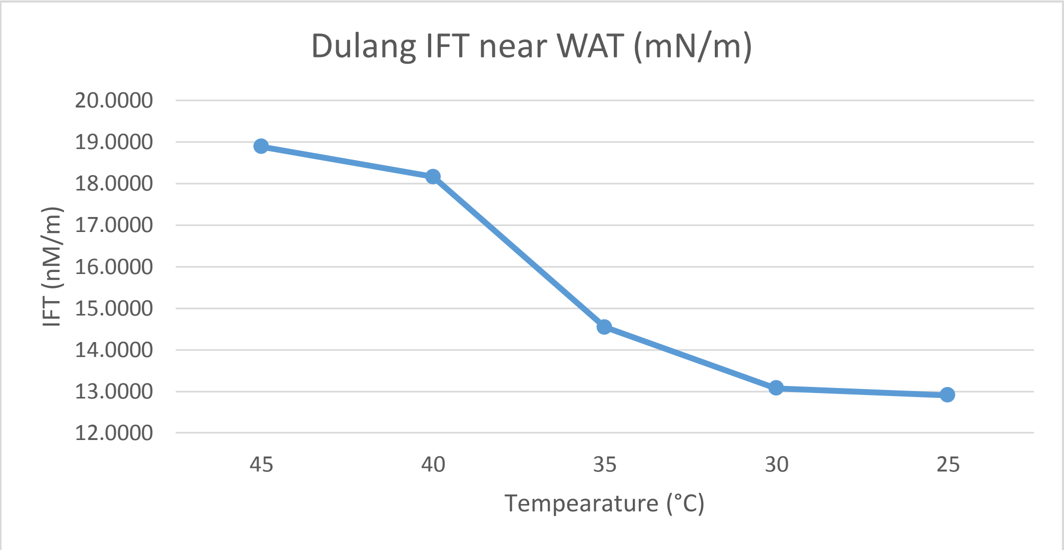


Figure 15 - Dulang IFT Trend near WAT

**Discussion:**

Looking at this graph, we can say that the interfacial tension value for temperature above wax appearance temperature is increasing as the temperature increase while the interfacial tension value decreasing for temperature below wax appearance temperature.

### 4.5.2 Sepat 7 Interfacial Tension Trend near Wax Appearance Temperature

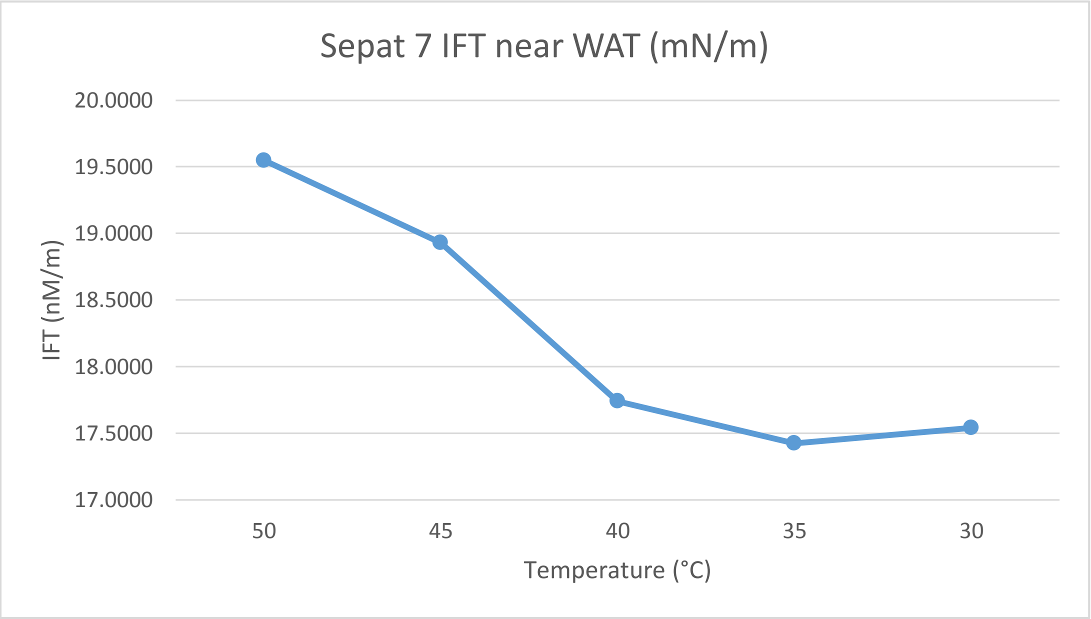


Figure 16 - Sepat 7 IFT Trend near WAT

**Discussion:**

From the graph, it shows that the interfacial tension value for the temperature above WAT is increasing while it turns to be decreasing below wax appearance temperature except for the 30°C. At 30°C, the temperature increase back but with very small amount in value. There is no specific reason can be said at the moment because there are no literature review saying why it turns to be like that.

#### 4.5.3 Puteri Interfacial Tension Trend near Wax Appearance Temperature

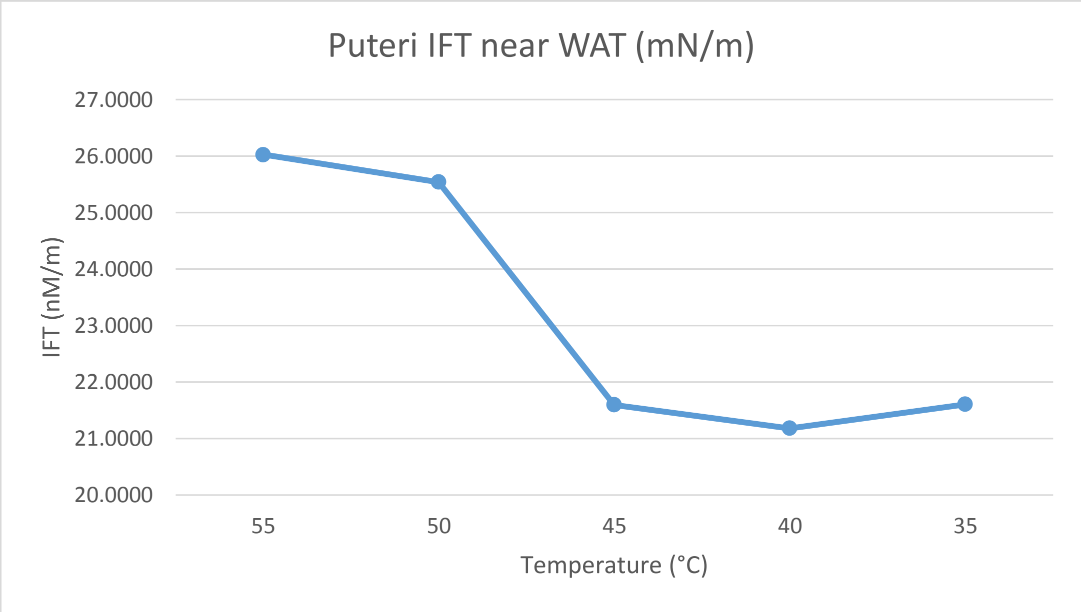


Figure 17 - Puteri IFT near WAT

**Discussion:**

Puteri crude oil has the same trend as Sepat 7 which the interfacial value is increasing above the wax appearance temperature while it is decreasing below wax appearance temperature but increase back at 35°C which the reason of why it increasing back is not yet discovered.

### 4.5.4 Trending For Three Crude Oils

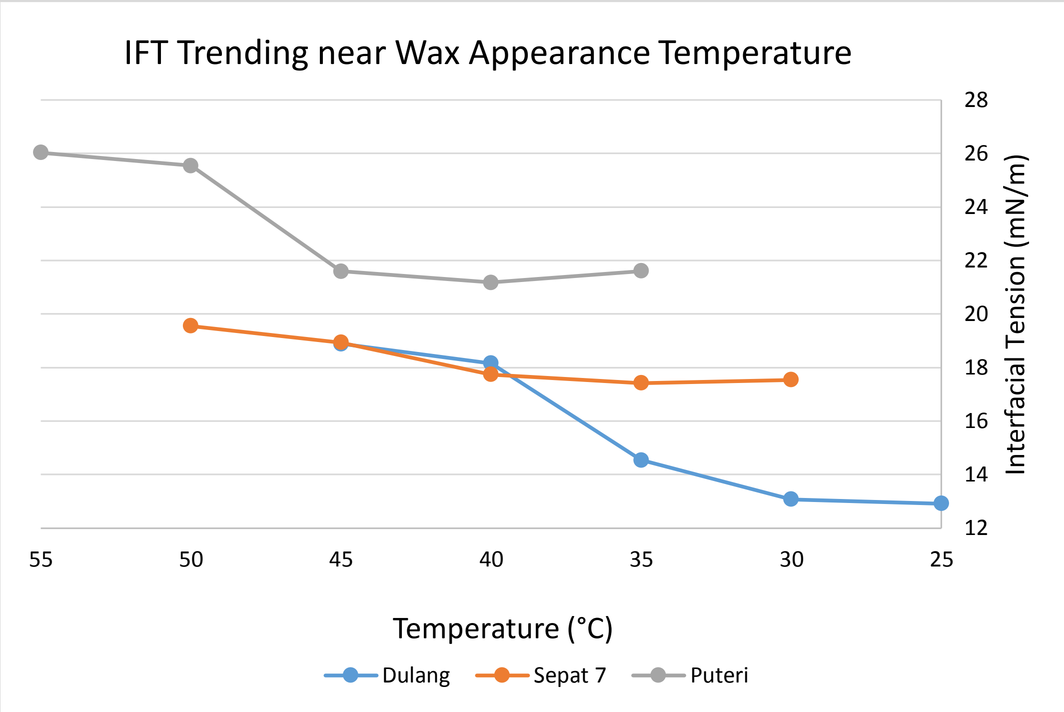


Figure 18 - IFT Trending near WAT for Three Crude Oils

**Discussion:**

Between the trending of these graph, Dulang is the only crude oil that has the trend of interfacial tension is directly proportional to the temperature. However, for other crude oils, it turns out to increase back at the lowest limit of temperature. The reason it increase back at the lowest limit is might be the crude oil is already completely turn to solid as the spinning drop tensiometer could not calculate interfacial tension between liquid and solid. It can only measure the IFT of liquid and liquid only. However, that inference is not yet proven since there is no experimental study carried out.

# CHAPTER 5

# CONCLUSIONS AND RECOMMENDATIONS

## Conclusion

As a conclusion, the objectives set up for this research has been accomplished which are basically to experimentally study the trend of interfacial measurement near wax appearance temperature. Besides, the problem of literature review on the interfacial tension trending near wax appearance temperature that lacking can be overcome by carrying out this study which might give something to compare with or serve as a reference for a future study.

Regarding to the objective to experimentally study the trending of the interfacial tension measurement near wax appearance temperature, it would give some help regarding the issue on calculating the pressure drop for some models of multiphase flow and serve as the reference or guideline towards those who is planning to conduct the study with same scope. In order to achieve the main objective, there are some side objective that keeping this study on its track.

As long as this study undergone, there are four side objectives set up for the smoothness of this study, and all four objectives is really helpful to get to the main objective. Puteri crude oil records the highest value of wax appearance temperature among all crude oils with 45°C. Next trials of this sample still recording the same value but not for other samples. Even the value for other sample do not record the same value for the second trial, the difference is still within the acceptable limit which is not more than 5°C.

From the second side objectives of determining the density of crude oils and distilled water, it shows that as the temperature increase, the interfacial tension value of sample decrease. Therefore, it really shows that temperature has effect towards density.

Last two side objectives can be said as the most important part of this study as it will provide the interfacial tension value and the trending of the interfacial tension of the crude oils in order to achieve the main objective of the study.

**Recommendations**

After all the experiments and analysis have been conducted as well as the objective has been achieved, recommendation regarding this project could be provided so that next work which is related to this project can use this project as guidance or reference. The recommendations that can be provided regarding this study are:

i. Carry out the interfacial tension measurement for the the sepat 7 and puteri crude oils using another interfacial tension determination equipment. The interfacial tension value for below wax appearance temperature is a little bit doubtful. The reason is because based on this study, the interfacial tension value of this two crude oil increasing even the temperature decreasing not same as the Dulang crude oils. This recommendation will help on verifying the inference that if the phase of crude oil already completely turns into solid state, the spinning drop tensiometer could not measure the interfacial tension value accurately anymore.

ii. Carry out this experiment with another type of crude oil. The selection of crude oil is important because it require the crude oil to contain wax since this experiment concern is temperature around the wax appearance temperature. If possible, use the crude oil outside of Malaysia because it may give another findings or results.

iii. Carry out the wax appearance temperature with more trials in order to get more accurate result of wax appearance temperature. Author cannot carry out the experiments with more trials due to the time constraint and the equipment is fully utilised by the postgraduate student since the equipment is under research custody.

If this three recommendations can be carried out, it would give a better and accurate explanations. Thorough experiments and investigation can lead to more understanding on how the improvement can be achieved. Thus, this analysis can give great impact to the industrial applications.

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