

# **Thermal Shrinkage and Gas Void Formation of Waxy Crude Oil –Gas Voids Study**

By

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(10754)

Dissertation submitted in partial fulfillment of

There requirements for the

Bachelor of Engineering (Hons)

(Mechanical Engineering)

SEPTEMBER 2011

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**Certification of Approval**

**Of research project**

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Mechanical Engineering Programme

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September 2011

## 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 of persons.

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## ABSTRACT

This paper discussed on the project entitled, “Thermal shrinkage and gas voids formation of waxy crude oil –gas voids study”. It consists of the project’s background, objectives, and problem statements, the relevance of the project, the literature review, the methodology which is the flow of the project and lastly the expected result of this project. In this project the author reported a study of the gas voids formed in gelled waxy crude which is a waxy crude oil which undergoes thermal shrinkage or cooling process.

The study is about gathering all possible information about the gas voids for further study which will lead to the result of compressibility of the gelled crude. The main objective of this project is merely to prove the compressibility of the gelled crude since gelled crude problem occur during operation shutdown and been assumed that gelled crude behave as incompressible high viscous fluid. After certain shutdown period or non-operating period the petroleum remains in the pipeline and undergoes thermal shrinkage due to low seabed temperature and subsequently leading to formation of gelled crude. Since they assumed that gelled crude behaves as incompressible high viscous fluid, high pressure pump is needed to move the gelled crude or to make the gelled crude to flow in the start-up process after certain shutdown period.

At the end of this research, if sufficient information on the gas voids is obtained successfully further explanation could be mode on the compressibility and subsequently the pressure needed in the startup process can be reduced thus reducing the cost for facilities such as the pumps. In order to provide such information, the crucial data to be considered are the volume of the gas voids over liquid volume, the pressure of the gas voids, effect of cooling rate on the gas voids volume and lastly the composition of the gas voids itself. The experimental setup for this project is about the simulation of cooling and gelling of the static petroleum crude in the pipeline which located on the seabed with some pressure reading device and the gas chromatography for the gas composition analysis purpose.

## ACKNOWLEDGEMENT

First and foremost, highest gratitude to Allah the almighty for upon His guidance and will, had blessed the author with good health and mind in order to complete the Final Year Project (FYP) successfully within the time given.

The author wishes to express his utmost appreciation to his project supervisor Dr. Azuraen Jaafar for having faith in the author to be her FYP student and guiding the author throughout the research. Her continuous support since the beginning until the end of the research is highly appreciated.

The author also hopes to send his deepest appreciation to Mechanical Engineering Department of Universiti Teknologi PETRONAS (UTP) for excellent support in terms of providing invaluable knowledge, information and laboratory facilities for the whole period of project especially to the lab technicians Mior Rosgiazhar Mior Sofli and Muhammad Hazri Ahmad Shahpin for the technical advice as well as Jaspreet Kaur for research advance knowledge.

Special thanks to the author's family and fellow colleagues for their support and help throughout the completion of this research

Last but not least, to all who's involved in making this research a success, thank you for your help, motivation and encouragement. This will be a memorable experience and the author hopes that it will bring beneficial output to others as well. The author sincerely appreciates all your kindness.

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## CHAPTER 1

### INTRODUCTION

#### 1.1 Background study

Nowadays, petroleum is one of the energy sources that have been widely used in the world. Petroleum is merely utilized to generate energy in most vehicles and the petroleum by-product can be made into plastic. Petroleum is defined as a natural yellow-to-black flammable liquid hydrocarbon found beneath the earth's surface. Hydrocarbon is an organic compound made up of carbon and hydrogen atoms. The petroleum can be found in the earth crust particularly on the land called "onshore" or under the sea which is termed as "offshore". Petroleum is normally found in the reservoir therefore petroleum drilling process has to be done in order to take out the petroleum which is now called "crude oil" to the surface. To start the drilling process the platform need to be set up first since the drilling rig is part of the platform.



Figure 1: Different types of platform

The above picture shows the example of the platform for the petroleum drilling and production process. The main focus of this final year project is more into discussing the offshore petroleum production process. During normal operation, the crude oil which is taken out from the reservoir will be transported to storage through a pipeline. This thermal shrinkage and gas void formation of waxy crude oil –gas voids study is actually based on the problem that occurs at offshore during the emergency or maintenance shutdown.

The study starts when the crude oil is found to be trapped in the pipeline during the shutdown for certain period of time until the plant started its next operation. The low seabed temperature affects the crude oil in the pipeline and the crude oil will undergo thermal shrinkage.

Normally the low seabed temperature will cause the crude to gel up or form wax at Wax Appearance Temperature (WAT). The gelled crude needs high start-up pressure in order to start the crude flow for the transporting purpose compared to the liquid crude oil. Using the assumption that the gelled crude behaves as an incompressible high viscous fluid (Michele Margarone, 2010), the facility such as pump designed to have superior horse power is then needed. High horse power equipment means high Capital Expenditure (CAPEX) to spend for the drilling and production process. This study is mainly focused on finding the relationship between gas voids present during the gelled up process and the compressibility of the gelled crude. If the gelled crude is compressible, this means the CAPEX can be reduced and as the result, the profit gained will be higher.

## **1.2 Problem statement**

In order to accurately predict the pressure to restart the gelled crude oil, it is important to take into consideration the multi phases of fluid present in the gelled crude during the thermal shrinkage. The gas voids formed during cooling maybe affect the compressibility of the gelled crude. Therefore, the study of thermal shrinkage and gas voids formations in the gelled crude requires further qualitative and quantitative analysis.

## **1.3Objective**

To study the formation of gas voids in gelled crude by measuring the size and pressure of the gas voids and also the effect of the cooling rate to the gas voids volume.

#### **1.4The Relevance of the Project**

This study will mainly discuss the characteristic of the gas voids and the relationship between the gas void and the compressibility of the gelled crude. If this study manages to provide new information and finding proves that the gelled crude is compressible, it can be a new guideline for all the oil and gas operating company in designing the platform that can give more profit with less cost and the most important is to reduce energy usage. Since cost and profit are of the utmost importance in every industry especially in the Oil and Gas industry, the outcome from this study will give a lot of benefits to the industry in terms of profit and cost benefit.

#### **1.5 The feasibility of the project**

This project is about the experimental simulation of the thermal shrinkage of waxy crude oil in the pipeline that lay on the seabed under non operating condition which means the petroleum crude remains in the pipeline statically. In conducting this project, the tool needed is a custom made crude tank. The crude tank will be used to store the crude during the experiment thus simulating a pipeline which is cylinder in shape. For the laboratory facilities, the water bath or chiller has been provided and the other main equipment needed to be used is a Gas Chromatography analyzer for the gas voids composition analysis purpose. For the time and cost allocation, the experimental setup complies to the allocated time and cost as it does not demand such expensive tools and only a short period of time is needed to conduct the experiment. For the source of references, there are a lot of journals to study and the most important is project supervisor can be consulted each time any problem encountered.

## CHAPTER 2

### LITERATURE REVIEW

The title of this project reflects the study of the gas void that happens during the thermal shrinkage of the crude oil. Thermal shrinkage is experienced during cooling that the crude oil undergoes. In the real situation thermal shrinkage happens to the crude oil in the pipeline naturally as the temperature of the seabed is low depending on the geographical difference. Based on the study, the petroleum crude contains paraffin ( $C_nH_{2n+2}$ ) and if the carbon atoms contained are more than eighteen the petroleum crude is then known as waxy crude oil (C.W Sum, 2011). During the thermal shrinkage or cooling process of this crude oil, wax or gelled crude will start to form at the Wax Appearance Temperature (WAT) and the wax starts to deposit at the pipeline forming a solid layer that decrease the available flow area (Ekweribe et al, 2008).

When the plant is about to restart the production operation, a high pressure is needed in order to move the gelled crude compared to the crude oil that can easily flow in the pipeline using the assumption that gelled crude behaves as an incompressible high viscous fluid (Michele Margarone , 2010). Since high pressure is needed to move the gelled crude, a huge amount of money needs to be invested for the high horse power pump at the platform. A study on the thermal shrinkage entitled “Novel Approaches to Waxy Crude Restart: Part 1: Thermal shrinkage of waxy crude oil and the impact for pipeline restart” had come out with a result which proves that there were gas voids produced by cooling process of the crude oil in the flow line (Phillips, David A., 2010). The gas voids appearance may affect the compressibility of the gelled crude since there are spaces for the gelled crude to move after some amount of pressure applied.

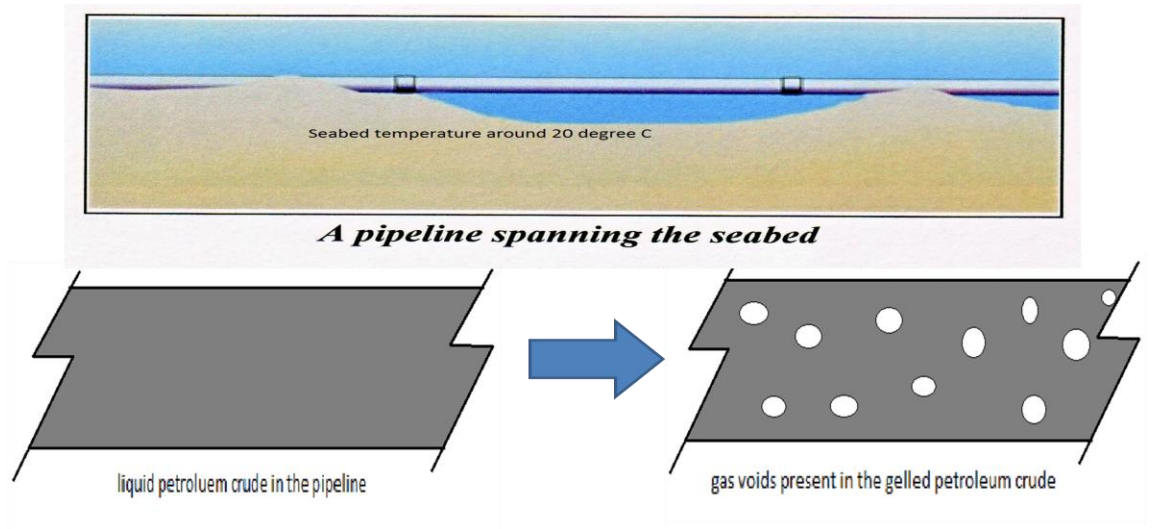


Figure 2: Gas voids formation diagram

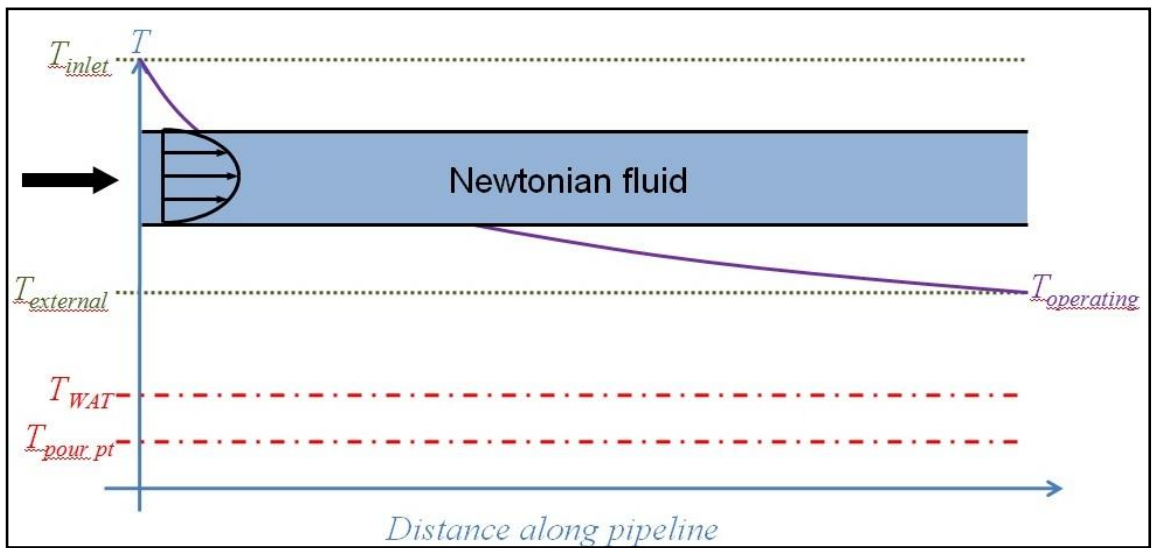


Figure 3: Newtonian crude before thermal shrinkage

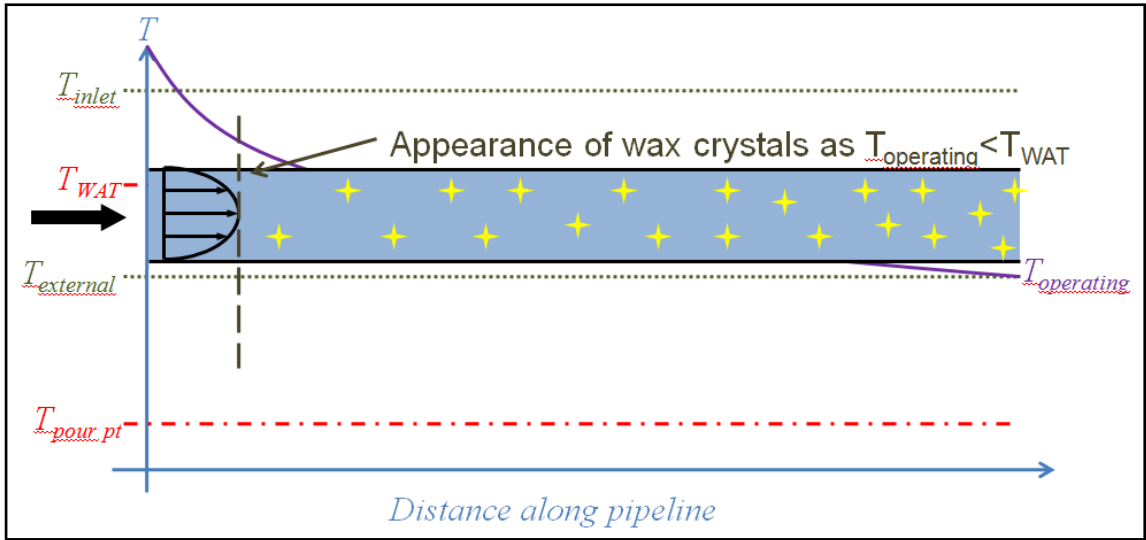


Figure 4: Wax formation start during thermal shrinkage

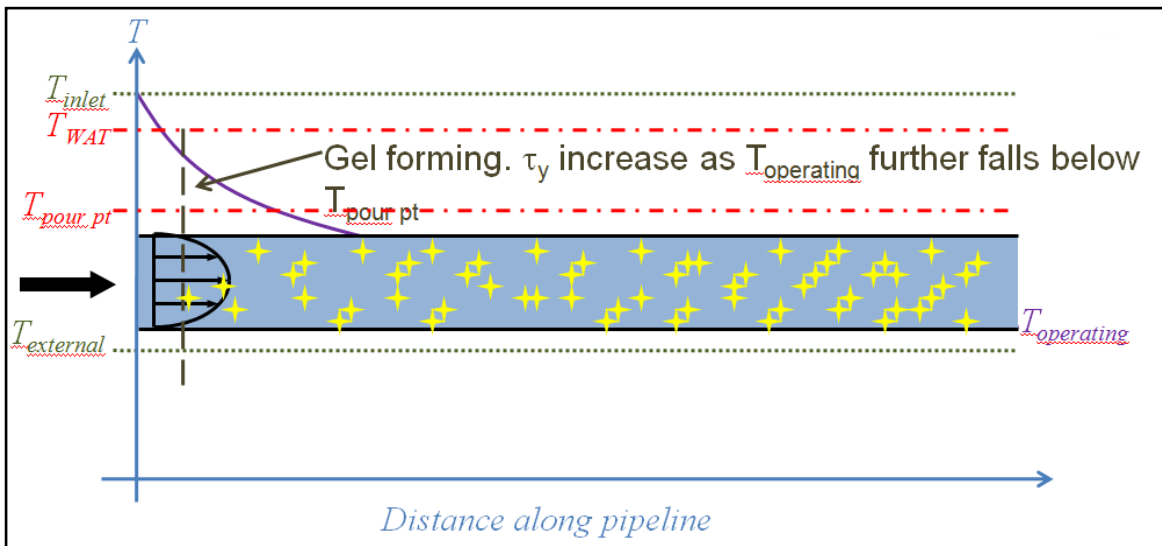


Figure 5: Wax form solid layer

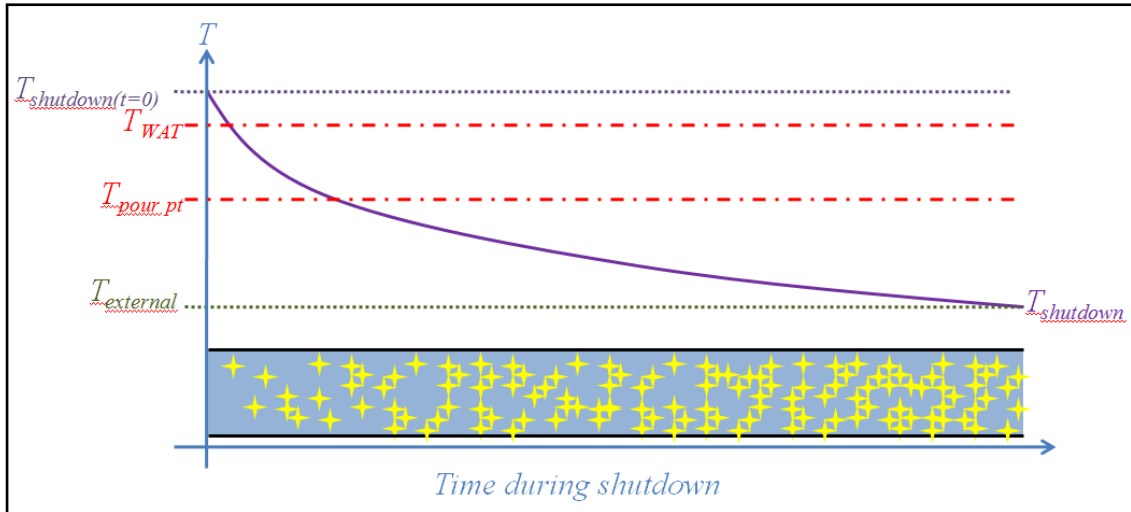


Figure 6: Wax occupy in the pipeline

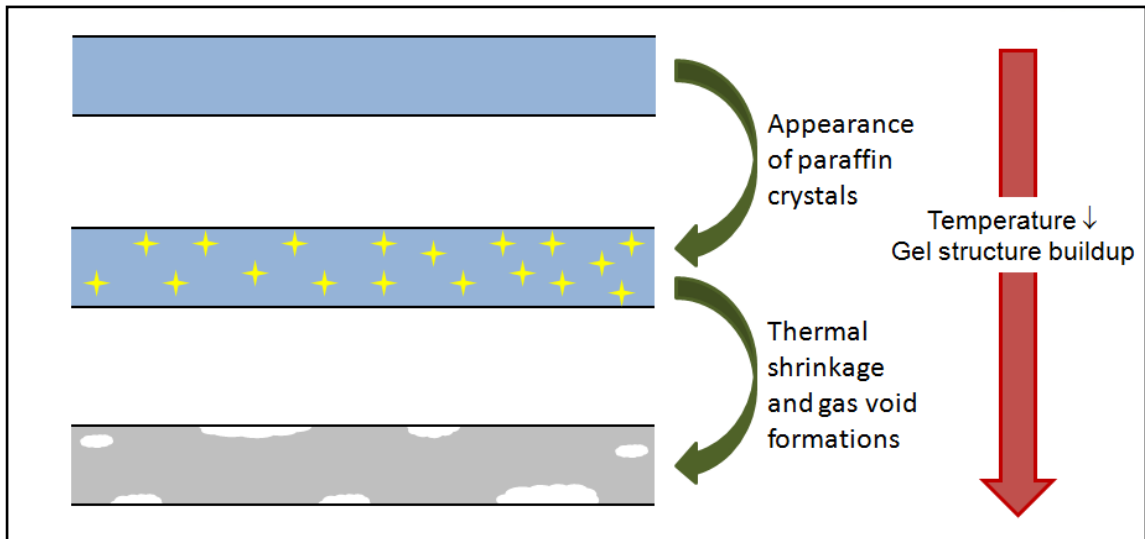


Figure 7: Wax formation stage

This study will employ certain parameters such as the size of the gas voids form at constant and variable cooling rate as well as the pressure of the gas voids itself. From the results of the past studies, a further study will be focused on the gas voids that are present as the crude oil which undergoes thermal shrinkage.



## CHAPTER 3

### METHODOLOGY

There are a few steps to be taken in order to get the expected result of the study and these steps will be improved for a better result.



Figure 8: Experimental procedure

### 3.0.1 Procurement

The first process after all the required documentation has been done is the procurement which involves purchasing of raw materials to fabricate the crude tank since the fabrication will be done manually and not by any fabricator. The procurement process is quite hard since the material selection is based on the daily usage and we still need to fill the requirement of a good crude tank which is needed to be made from glass so that it acts as a good thermal conductor and the transparent glass allow us to see any changes of the crude oil physical properties during the heating and cooling process.

The chosen valve is easily available in the market and suitable for the tank which is not too big in size so that the gas in the valve will not interfere with the result thus it is possible to reduce the error of the pressure reading and gas voids volume. The material that had been selected for the tank cover is the transparent plastic cap which can fit the glass opening and allow the observation of the physical crude oil properties from above. Silicon sealant had been chosen as the sealer for the crude tank since it is mainly used to seal glassware. Lastly, a flexible tube is used in order to connect the crude tank to the differential pressure meter perfectly without any leakage.



Figure 9: Raw material of crude tank

### 3.0.2 Fabricating

For the experiment setup, the fabricating process will be done manually by the author since the fabricating process by the private fabricator is not highly feasible in terms of economy and time constraint. After the procurement process is completed, the crude tank was fabricated. The below procedures lay-out the fabricating of the crude tank processes:

1. The tank cap is drilled to the valve end size to make sure the valve fit the cap perfectly.
2. Then, the valve is placed at the right location which is on top of the drilled tank cap.
3. The valve been attached to the container cap and tighten with socket.
4. Crude been poured into the tank after ready.



Figure 10: Old crude tank

### 3.0.3 Crude Oil Preparation

Originally, the crude oil is stored in a container for a long time thus it allows the crude to freely separate into the heavy part and the light part of the oil. To ensure homogeneity, the crude needs to be heated up to 80 degree Celsius in a water bath for 2 hours. At 80 degree Celsius, the crude will form Newtonian fluid and 2 hours agitation process is needed to make sure the crude is mixed together nicely and become homogeneous crude. After the 2 hours of heating and agitation has elapsed, the crude is then poured into smaller containers to store it for the experiment.



Figure 11: Petroleum Crude container

### 3.0.4 Experimental Setup Training

Before commencing the experiment, there are some training needed in order to operate the equipment such as Vivo RT 2 water bath which is the main equipment that will be used in this experiment. The training has been conducted by the lab technician and all the safety measures were taught to ensure the experiment run safely. Vacuum meter is the important device that will be used in the experiment. This device will give the gauge pressure within the gas voids.



Figure 12: Major equipment of the experiment

### 3.0.5 Penara Crude Properties Experiment Using Rheometer

Penara Crude sample preparation:



Figure 13: Crude tank immerse in the water bath

- 1) Prior to measurements using the rheometer, the Penara blend in the small container was soaked in a water bath at 80°C for 2 hours. However, the tests which were conducted on gelled sample (small sample volume left to gel to replicate IFP loading procedure) had shown a little effect of the sample initial condition.
- 2) The sample was loaded on the peltier plate set to 80°C to avoid gelling upon loading on the peltier plate and to allow the 300 micron gap to be achieved for the measurements. It is important that sufficient amount of sample is loaded onto the peltier plate. Under-loading and over-loading may result in incorrect measurements done.
- 3) It is also important that the head is lowered gradually to avoid squeezing out the sample from under the geometry leading to a void in the centre of the geometry.
- 4) It is also crucial to ensure that the instrument is calibrated regularly prior to the test as relatively heavy geometry (stainless steel) is used for the measurements. Without regular calibration, the low viscosity values at temperature  $> T_{WAT}$  will not be accurate (sometimes giving negative values)

### 3.0.6 Gas Voids Volume Measurement Process

The idea of measuring the gas voids volume is to have the info about how much the gas voids will present after the thermal shrinkage process per total crude volume. Below is the procedure to measure the gas voids volume.

1. The crude tank was immersed in the water bath at temperature 80 degrees Celsius for 30 minutes.
2. The Newtonian crude been poured into glass for better observation process.
3. The cooling process with two different cooling rate to 25 degree Celsius starts right after 30 minutes of heating.
4. Gas voids formation was observed.
5. Measurement of gas voids size was done by taking the diameter and the height of gas voids.
6. Gas voids volume over total crude volume is calculated.



Figure 14: Gas voids formation

### **3.0.7 Gas Voids Pressure Measurement**

The main point of the experiment is to know the gas voids pressure. If the gas voids pressure is lower than the atmosphere pressure, this will give a good sign which lead to prove the compressibility of the gelled up waxy crude oil. Proper steps are crucial since the pressure maybe too small for the pressure meter to read and error will lead to failure of getting the intended result which is the gas voids pressure. Below is the procedure for gas voids pressure measurement:

1. Immersed the crude tank in the water bath.
2. Open the valve properly to make sure the air can flow from tank to atmosphere.
3. Heating process started at 80 degree Celsius for 30 minutes.
4. Close the valve properly to make sure no air can flow into the tank after 30 minutes of heating.
5. Cooling process started and cools the tank down to 25 degree Celsius.
6. The immersed into the 25 degree Celsius water for 30 minutes.
7. Vacuum meter is use to measure the pressure.
8. Valve is opened and the gas voids pressure is measured.
9. The experiment repeated 3 times and the average reading calculated.



### **3.0.8 Error Reduction Steps**

There are some error reduction steps that need to be considered in order to get the perfect result thus making this experiment a successful one. Since this experiment deal with a small change in pressure so the experiment setup must be the finest setup. Below are the error reduction steps that need to be taken.

1. The valve must be small to reduce the air contamination which will disturb the gas chromatography result.
2. There is no leakage of gas so that the pressure of the gas voids will be more accurate.
3. The pressure meter hose will be modified to make it shorter to reduce the air contamination that will disturb the gas voids pressure result.
4. The hose that connects the tank and the pressure meter will be transparent in color to make sure that there are no crude in the hose during the pressure measurement process otherwise the pressure meter will break.

### 3.0.9 Crude Tank

Since the procurement process has already been completed, the fabrication process is then ready to be taken place with the lab technician supervision. The new raw materials that had been purchased are the threaded end valve, transparent tube for differential pressure meter connection purpose and lastly threaded end glass container. Below are the new specifications of the new tank.

1. Using threaded cap glass container for better pressure resistance.
2. Using threaded valve to make sure better grip and no more leakage.
3. Transparent tube to make sure the tube is clean from crude before pressure reading process because during the experiment, the crude tends to flow into the tube as it expands during heating.



Figure 15: New crude tank parts

### 3.0.10 Gas Chromatography

Gas chromatography is the method utilized to determine the composition of certain gases. For this experiment the gas chromatography is used to find the composition of the gas voids as the research focus on the gas voids study. The findings of this gas voids composition may lead to a better idea of the gas voids and the compressibility of the gelled up waxy crude oil. Below is the procedure of extraction of gas for gas chromatography.

1. Heating process started at 80 degree Celsius for 30 minutes in the water bath while the valve is open.
2. Cooling process started and cools the tank down to 25 degree Celsius and the valve is close properly to avoid gas voids loss.
3. The tank immersed into the 25 degree Celsius water for 30 minutes.
4. Syringe was used in order to extract the gas from the tank after the valve is open and will be sealed using silicon glue.
5. Gas from syringe will be transferred into tank in the gas chromatography machine and result will be documented.



Figure 16: Syringe for gas voids storage

### 3.1 Experimental Setup

The simulation of the crude oil gelled up will be conducted in this experiment. The crude oil will be stored in the tank and will be immersed in the cooling tank. There are several variables that are taken into consideration in order to achieve the objective of the project:

1. Cooling temperature
2. Cooling rate

The variables controlled by the cooling equipment such as Vivo RT2 water bath. The equipment will be considered based on the availability at the laboratory and the best equipment will be chosen to ensure that the result is accurate. The crude oil will be stored in a custom-made tank that will be fabricated based on the dimension of the cooling equipment.

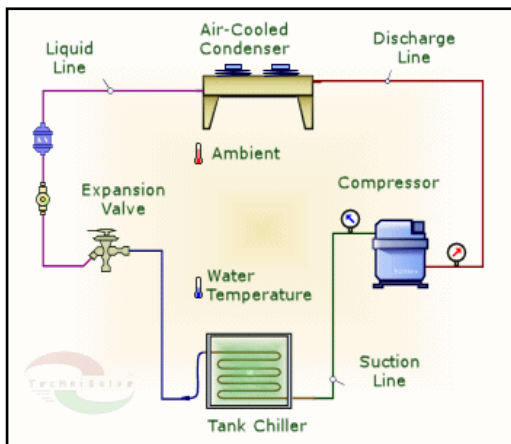


Figure 17: Diagram of water

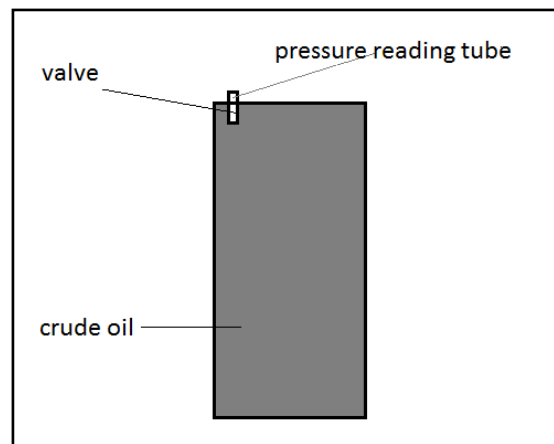


Figure 18: Illustration of crude tank

### 3.1.1 Gas Chromatography

Gas chromatography (GC) means gas separation process without decomposition for the gas compound analysis purpose. Usually the GC is used for the testing of the purity of a substance by finding out the relative amount of gas component. The working principal of this GC involves the sample of liquid been vaporized and injected onto the head of the chromatographic column. The sample will be vaporized to the gas state and been transported by the inert gas through the column to the detector.

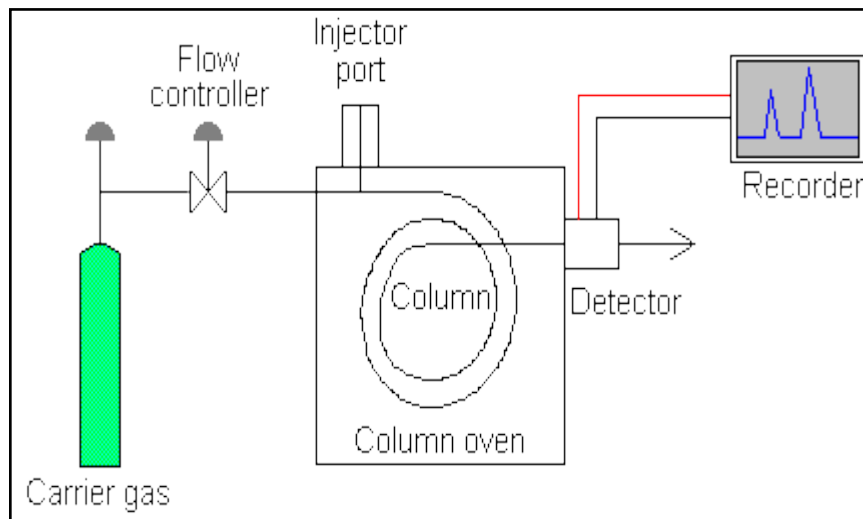


Figure 19: Diagram of gas chromatography machine

The inert gas acts as the carrier gas which has to be chemically inert. The common inert gases used are nitrogen, helium, argon, and carbon dioxide. The carrier gas will vary dependent on the type of detector which is used. The carrier gas system also contains a molecular filter for the water and other impurities removal purpose. The significant of the GC with this gas voids study is to observe the composition of the gas voids that form after the thermal shrinkage of waxy crude oil.

### 3.2 Experimental tools

The tools, equipment and materials that are going to be employed in this project are:

1. Custom crude tank
2. Crude oil
3. Thermo hake
4. Water chiller
5. Pressure meter



Figure 20: Vivo RT2 water bath

3.3 Designing the crude tank

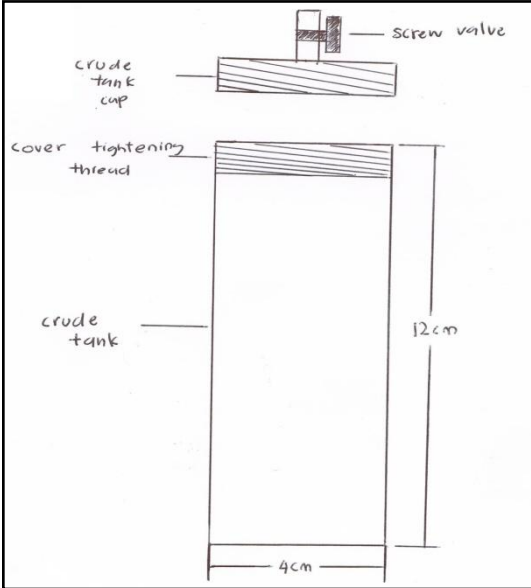


Figure 21: Raw sketch of crude tank

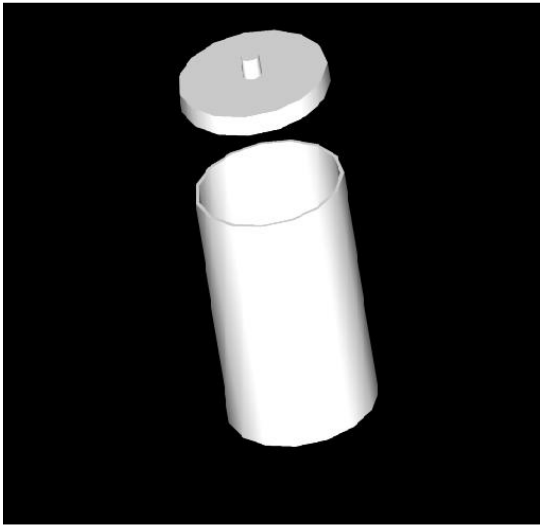


Figure 22: AutoCAD tank image

Crude tank size is 4cm diameter and 12cm length based on bath size opening is 12.5 x 11.5cm (W x L) & bath depth is 19cm.

#### Dimension

- Diameter 4cm, height 12cm (approximate volume 150ml)
- To simulate the pipeline ( long cylinder)

#### Material

- Borosilicate glass (usually used to make laboratory glassware)
- Thin Glass to transfer the heat from the crude tank surrounding.
- To make sure gas voids form can be observe easily.

#### Valve

- Screw type valve located at the center of the cap (the similar one used at the end of a burette)
- To seal the tank and allow the pressure different between inside the tank and atmosphere

#### Crude Tank Cap

- To seal the crude tank tightly to avoid leaking using tightening thread



After Fabrication



Figure 23: High pressure resistant crude tank

The actual crude tank that will be used in the experiment must fulfill the requirement of a crude tank such as good thermal conductivity, high pressure resistant and has a valve to control the pressure.

### 3.4 Gantt Chart

Activity	FYP1				FYP2			
	May	June	July	August	Sept	Oct	Nov	Dec
Project study	█	█						
Proposal submission		█						
Proposal defense			█					
Experimental setup			█	█	█			
Experimental work					█	█		
Analysis of result						█	█	
Final documentation								█

Table 1: Gantt chart

### 3.5 Key Milestone

Key Milestones	FYP1				FYP2			
	May	June	July	August	Sept	Oct	Nov	Dec
Experimental setup plan			•					
Crude tank design				•				
Crude tank fabricating complete						•		
Experiment complete								•
Data analysis								•

Table 2: Key milestone

### 3.6 Expected Result

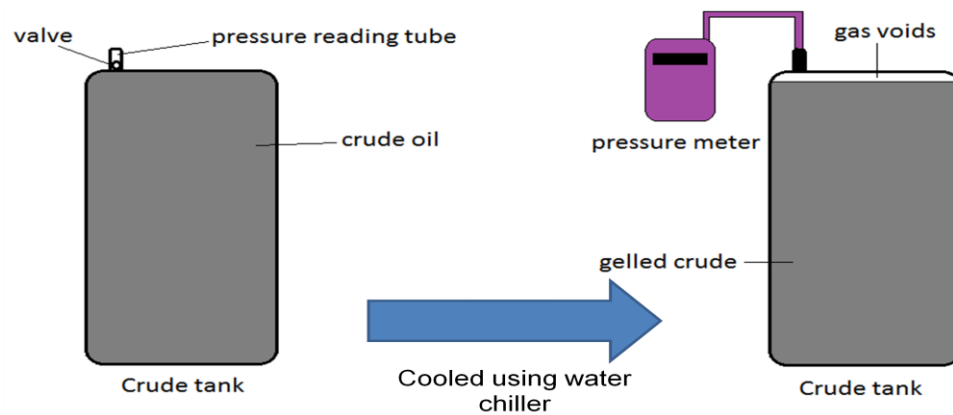


Figure 24: Expected result illustration

The expected results to be obtained at the end of this study are:

1. The crude properties
2. Effect of the cooling rate to the gas voids present
3. Pressure of the gas voids
4. Composition of gas voids

## CHAPTER 4

### RESULT AND DISCUSSION

#### 4.1 Penara Crude Properties

In this experiment, Rheometer is use to find the Wax Appearance Temperature (WAT) and the Pour Point for the Penara crude in order to find the best temperature for heating and cooling process.

##### Rheological Measurements of Penara Crude (Roughened Plate)

All the rheological measurements were conducted using an AR G2 controlled stress Rheometer by TA Instrument.

Geometry used: 4-cm roughened parallel plate

Gap setting: 300  $\mu\text{m}$

Steps:

- 1) Conditioning step – a sample was heated at 80°C for 30 seconds
- 2) Conditioning step – a sample was left to rest for 2 minutes
- 3) Temperature ramp step – a constant shear rate was applied with temperature reduced from 80°C to 40°C at a cooling rate of -1°C/min. Data sampling was taken every 30 s.

Steps 1-3 were conducted for constant shear rates of 50s<sup>-1</sup>.It is also important that the geometry is released upon raising the head at the end of each test as the crude is in gelled state at the end of the test.

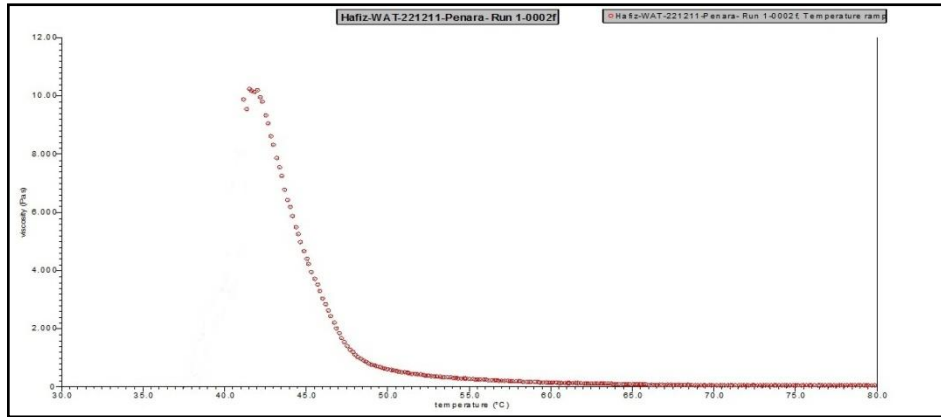


Figure 25: Penara crude Rheometer result

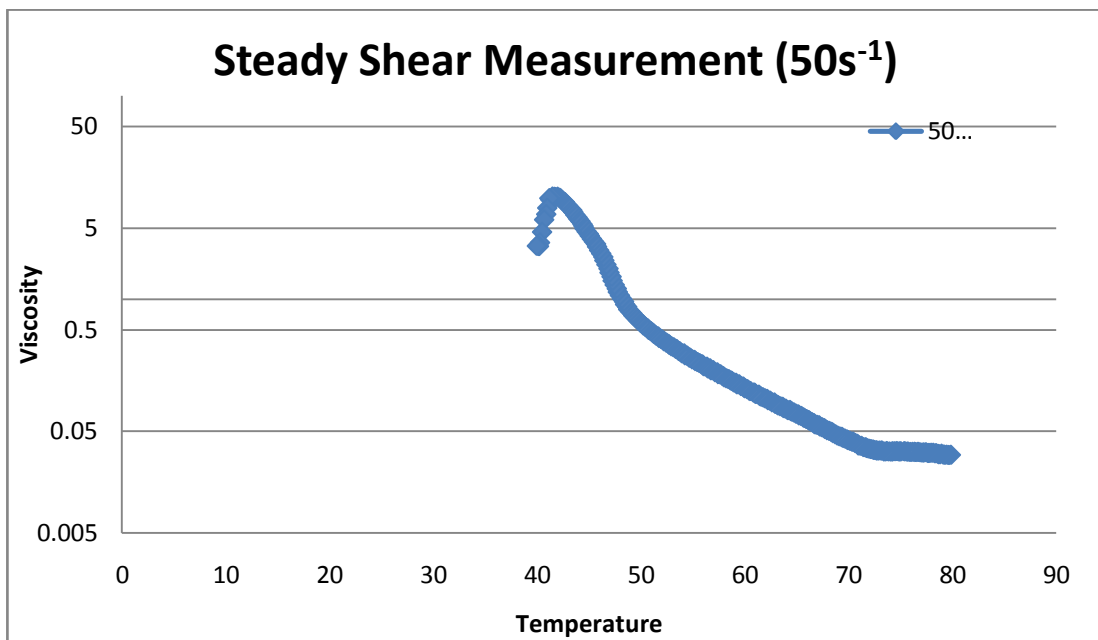


Figure 26: Viscosity VS Temperature

**Table 2.1: Partial List of PETRONAS Carigali Fields with WAT & Pour Pt Data**

Field	Fluid	Location	WAT/Cloud Pt (°C)	Pour Point (°C)	Wax Content (wt%)
Angsi	Oil	Malaysia			
Anding Utara -1	Oil	Malaysia			
J4	Oil	Malaysia			
D21	Oil	Malaysia			
Penara	Oil	Malaysia	68	51	24
Sepat	Oil	Malaysia			
Malikai	Oil	Malaysia			
Diyaberkir	Oil	Turkmenistan			
Thang Long	Oil	Vietnam			
Topaz	Oil	Vietnam			
Chinguetti	Oil	Mauritania			
Adar	Oil	Sudan			
Owez	Condensate	Turkmenistan			
Rehmat	Condensate	Pakistan			
Mehar	Condensate	Pakistan			
Mashrykov	Condensate	Turkmenistan			

Table 3: Crude properties (confidential)

From the table and the result of the Rheometer for the Penara crude, it is shown that the Penara wax Appearance Temperature is 68 degree Celsius and the pour point is 51 degree Celsius. This shows that Penara crude is heavy oil or black oil which is very thick and has a high viscosity. Since the pour point is 51 degree Celsius which is quite high, the cooling process will take shorter time for crude to form wax. Base on this criterion, the cooling temperature of the crude will be 25 degree Celsius since it is low enough to produce the wax from the crude. After knowing the properties of this crude, a better procedure will be designed in order to get the best result for every experiment.

## 4.2 Gas Voids Percentage

### 4.2.1 Total Crude Volume



Figure 27: Before thermal shrinkage

It is important to find the total crude volume for the comparison purpose. The comparison will be based on the crude volume before and after the thermal shrinkage which is the cooling process. By knowing the volume difference of the crude before and after the thermal shrinkage process, the percentage of the gas voids volume can be calculated. The calculation of the total crude volume or the volume of Newtonian crude before the thermal shrinkage process is done by using the mathematical model.

$$\text{Total crude volume} = \pi r^2 h$$

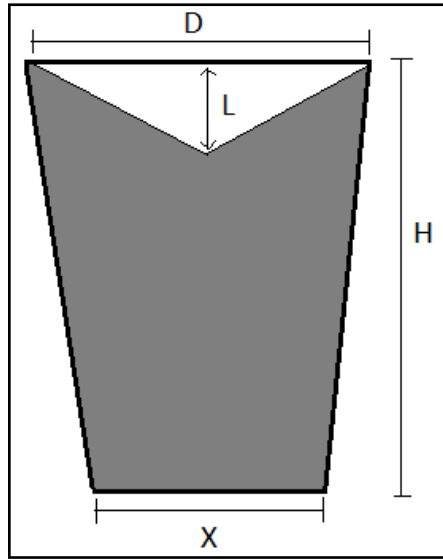


Figure 28:2D diagram of crude container

$$\text{Where } r = \frac{X+D}{4}$$

$$D = 6.8 \text{ cm} \quad X = 5.8 \text{ cm} \quad H = 7.5 \text{ cm}$$

$$r = \frac{6.8 + 5.8}{4}$$

$$r = 3.15 \text{ cm}$$

$$\text{Total crude volume} = \pi (3.15)^2 (7.5)$$

$$\text{Total crude volume} = 233.8 \text{ cm}^3$$

#### 4.2.2 Total Gas Voids Volume

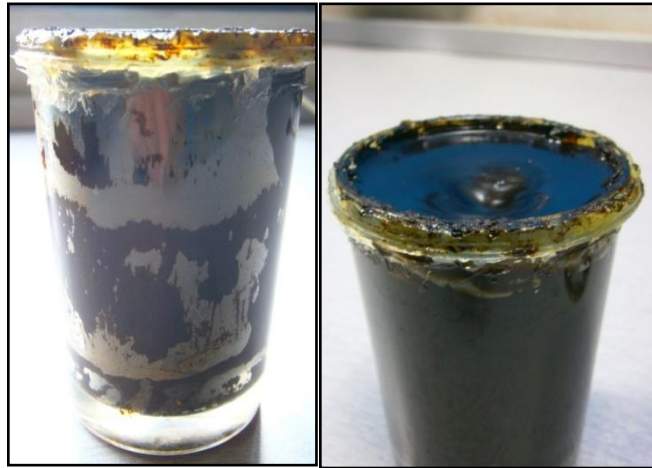


Figure 29: After thermal shrinkage

Based on the figures shown above, after the thermal shrinkage of the crude oil, the crude will turn into the wax. The shrinkage of the crude will cause the formation of the gas voids which fills up the empty space. The geometry of the gas voids is not consistent and it is hard to do the volume calculation since the gas voids formation is not fixed in one place since the crude is very thick which the viscosity is high thus the gas void cannot migrate to the upper part of the container.

The geometry of the gas voids that form at the upper part of the container is slightly triangle and based on this scenario an assumption was then made. The gas voids geometry is triangle by considering the gas voids that form in the other area as well. The calculation has been made by two sets of experiment and each experiment had three repetitions for the average reading purpose.

To find the percentage of the gas voids volume, we need to find the total gas voids volume first. The gas volume will be divided by the total crude volume at the end of the experiment.



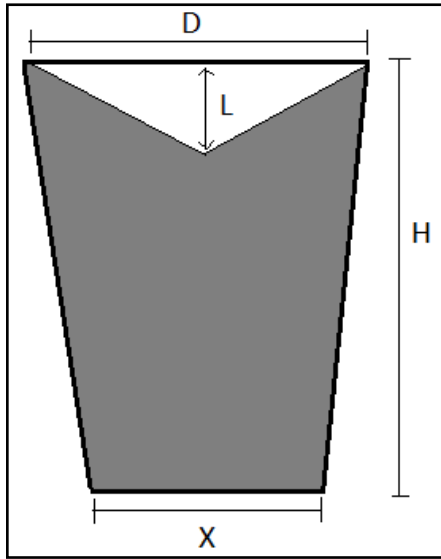


Figure 30: 2D diagram of crude container

$$D = 6.8 \text{ cm} \quad X = 5.8 \text{ cm} \quad H = 7.5 \text{ cm}$$

### **Percent of Gas Voids Volume for Cooling At 25 Degree Celsius Using Ambient Temperature of Laboratory**

For the first set of experiment, the cooling at 25 degree Celsius had been done by cooling the crude using the laboratory room temperature. L, which is the different of wax height before and after cooling process is taken for three times and the average is calculated by using mathematical model.

$$\text{Average } L = \frac{l_1 + l_2 + l_3}{3}$$

The reading for the L:

$$1^{\text{st}} \text{run} = 1.7 \text{ cm}$$

$$2^{\text{nd}} \text{run} = 1.3 \text{ cm}$$

$$3^{\text{rd}} \text{run} = 1.5 \text{ cm}$$

$$\text{Average L} = \frac{1.7\text{cm} + 1.3\text{cm} + 1.5\text{cm}}{3}$$

$$\text{Average L} = 1.5 \text{ cm}$$

$$\text{Total gas voids volume} = \pi r^2 h$$

$$\text{Where } h = L_{\text{avg}} \text{ and } r = \frac{D}{4}$$

The gas voids cone shape will be converted into cylinder for easier calculation.

$$\text{Total gas voids volume} = \pi (1.7)^2 (1.5)$$

$$\text{Total gas voids volume} = 13.6 \text{ cm}^3$$

$$\text{Gas voids percentage} = \frac{13.6}{233.8} * 100$$

$$\text{Gas voids percentage} = \mathbf{5.8 \%}$$

The average gas voids formation percentage for the cooling process using 25 degree Celsius of the ambient temperature of the laboratory is 5.8%.

### **Cooling Rate**

$$\text{Cooling rate} = \frac{80 \text{ degree Celsius} - 25 \text{ degree Celsius}}{43 \text{ minutes}}$$

$$\text{Cooling rate} = 1.3 \text{ degree Celsius /minute}$$

## **Percentage of Gas Voids Volume for Cooling In the Water Bath Using Water as Cooling Medium**

The reading for this experiment is shown below:

$$1^{\text{st}} \text{ run} = 1.6 \text{ cm}$$

$$2^{\text{nd}} \text{ run} = 1.7 \text{ cm}$$

$$3^{\text{rd}} \text{ run} = 1.6 \text{ cm}$$

$$\text{Average } L = \frac{1.6\text{cm} + 1.7\text{cm} + 1.6\text{cm}}{3}$$

$$\text{Average } L = 1.63 \text{ cm}$$

$$\text{Total gas voids volume} = \pi r^2 h$$

$$\text{Where } h = L_{\text{avg}} \text{ and } r = \frac{D}{4}$$

The gas voids cone shape will be converted into cylinder for easier calculation.

$$\text{Total gas voids volume} = \pi (1.7)^2 (1.63)$$

$$\text{Total gas voids volume} = 14.5 \text{ cm}^3$$

$$\text{Gas voids percentage} = \frac{14.5}{233.8} * 100$$

$$\text{Gas voids percentage} = 6.2 \%$$

## **Cooling Rate**

$$\text{Cooling rate} = \frac{80 \text{ degree Celsius} - 28 \text{ degree Celsius}}{90 \text{ minutes}}$$

$$\text{Cooling rate} = 0.6 \text{ degree Celsius /minute}$$

### 4.3 The Effect of Gas Voids Volume to the Different Cooling Rate



Figure 31: Slow cooling rate (Right) and Fast cooling rate (Left)

The result of the effect of the cooling rate to the gas voids present volume, the gas voids volume is slightly different which is 5.8 % for using 25 degree Celsius ambient temperature of laboratory which is fast cooling rate of 1.3 degree Celsius temperature drop/minute and 6.2 % for using water as cooling medium for 1 hour which is a slow cooling rate of 0.6 degree Celsius temperature drop /minute. The obvious difference is the gas voids formation geometry. For the experiment using 25 degree Celsius of the ambient temperature of the laboratory, the formation of the gas voids scattered since the crude forms wax quickly and the gas voids cannot move to the surface due to the high viscosity of Penara crude. For the experiment using water as a cooling medium, the gas voids formation geometry is better and the shape is almost a perfect triangle. That is the most obvious difference between the high and low cooling rates.

#### 4.4 Gas Voids Pressure



Figure 32: Before pressure reading

For the gas voids pressure measurement, the experiment is done by using the crude tank that is fabricated for high pressure resistant purpose so that the pressure of the gas voids will be precisely taken. After heating the crude tank that fill with the crude at 80 degree Celsius for 30 minutes in order to make the crude Newtonian fluid like operating condition, the valve is closed and the crude tank was immersed in the 25 degree Celsius water for the thermal cooling process for a period of 30 minutes to make sure the simulation of the real situation is successful. After many trials using different pressure meter such as differential pressure meter and manometer, the gas voids volume can be concluded as a below than atmosphere pressure.

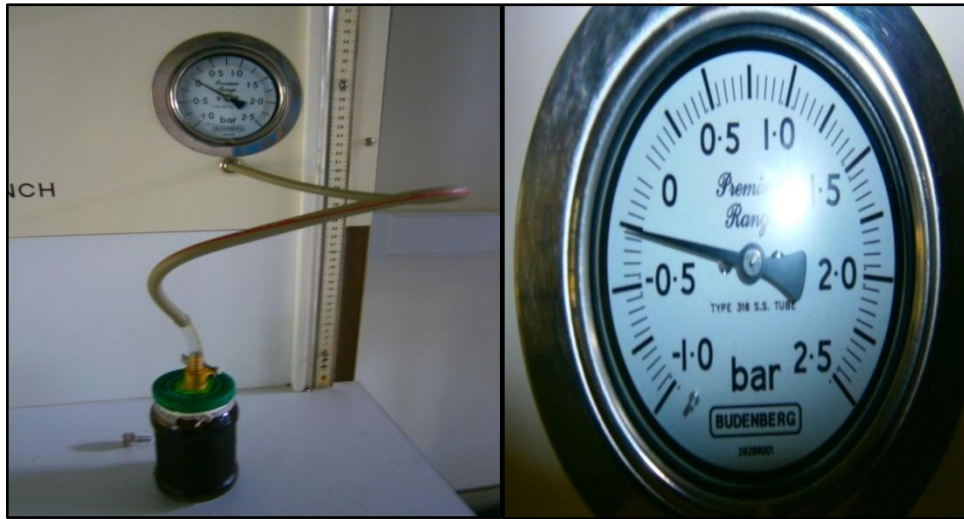


Figure 33: After pressure reading

After knowing the gas voids pressure is a below than atmosphere, the vacuum meter is used in order to get the reading. There are some modifications in order to use the vacuum meter since the meter located on the Pressure measurement bench. The tube that can fix the vacuum meter input hole is used and connected to the crude tank directly. In order to get the reading, the valve must be opened and once the vacuum meter reading is stable, then the meter reading is taken. After three times doing the same experiment the reading are as below:

1<sup>st</sup> run: -0.22 bar Gauge

2<sup>nd</sup> run: -0.21 bar Gauge

3<sup>rd</sup> run: -0.26 bar Gauge

$$\text{Average gas voids pressure} = \frac{(-0.22) + (-0.21) + (-0.26)}{3}$$

$$\text{Average gas voids pressure} = -0.23 \text{ bar Gauge}$$

## 4.5 Gas Composition

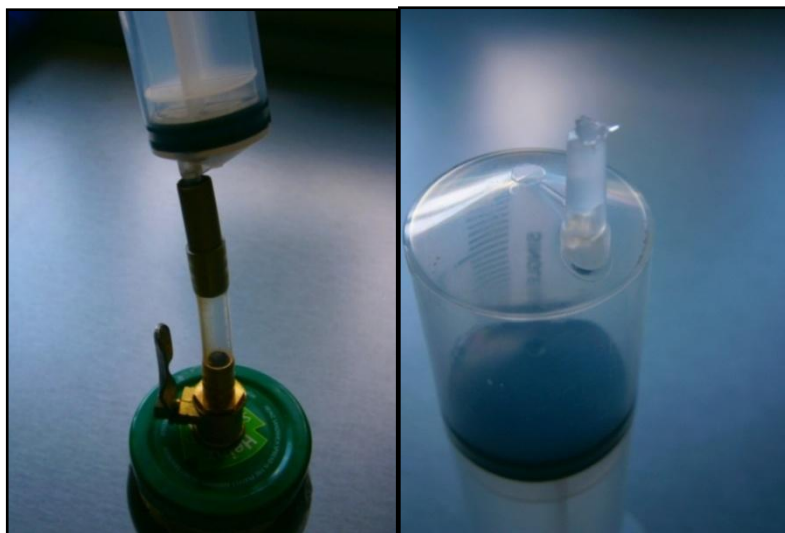


Figure 34: Gas voids suction and storage process

By referring to the previous experiment, it is quite hard to take out the gas voids since the pressure is negative which means that it is below. This results in a sudden surge of flow from outside if there is any opening although it is a small one. The steps that need to be taken are to attach a 30 mL syringe before opening the valve. The syringe moves down which means the crude tank sucks the air from the syringe into the crude tank. The syringe is then pushed to give some pressure to the crude tank and then the tank's valve is closed.

The empty syringe is attached back to the tank and the valve is opened then the syringe moves upward indicating that the air from the crude tank is moving into the syringe due to a lower pressure. Lastly, the syringe is sealed using silicon glue and the Gas chromatography process is conducted. After two sets of data from Gas chromatography were obtained using the same air from the syringe, the resultant gas appears the same which is only air. There is no other gas since the gas analyzer is used to find the other gases that are contained in that gas voids.

Sample Information

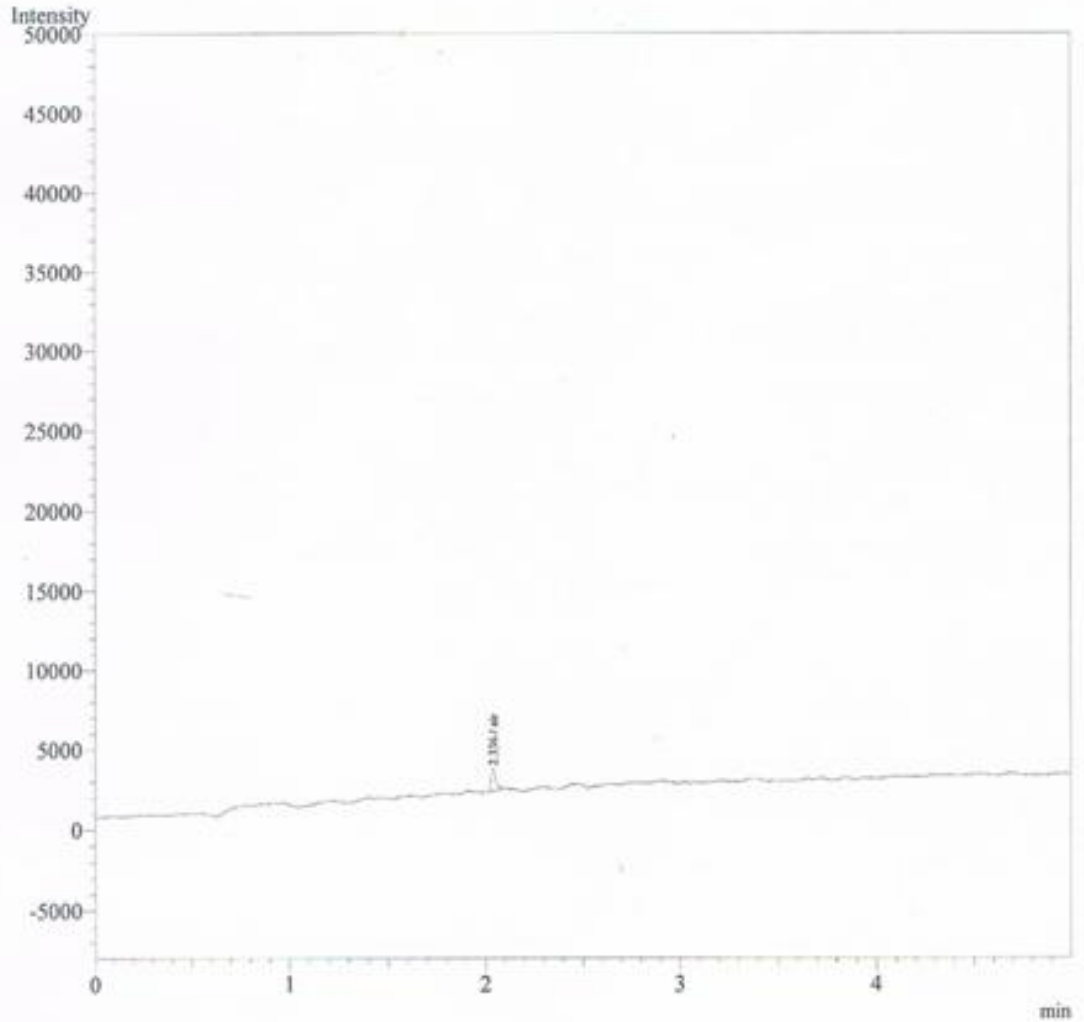
Analysis Date & Time : 14/12/2011 11:39:54 AM

Sample Name : air

Sample ID :

Data Name : C:\GCsolution\Data\2011\hafiz hizam\air.gcd

Method Name : C:\GCsolution\Data\2011\calib 3 comp 100%air.gcm



Peak#	Ret.Time	Area	Height	Conc.	Units	Mark	Name
1	2.336	2094	1385	0.000	%		air
Total		2094		0.000			

Figure 35: First Gas Chromatography trial result



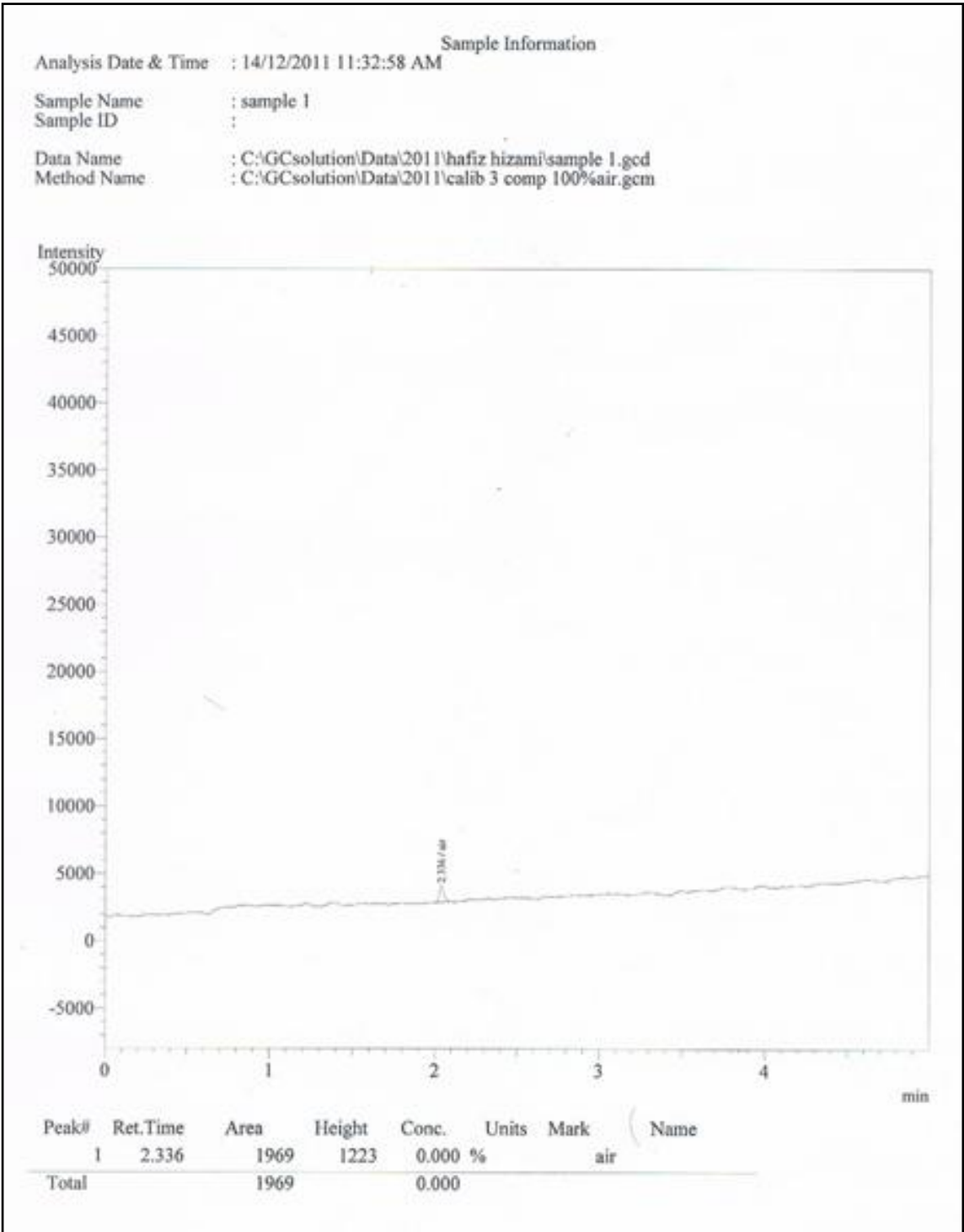


Figure 36: Second Gas Chromatography trial result

## CHAPTER 5

### CONCLUSION AND RECOMMENDATION

#### 5.1 Conclusion

The main purpose of this research is to study the gas voids that form after the thermal shrinkage of the waxy crude oil. There are a lot of things that needs to be studied about the gas voids and the focus are on the percentage of gas voids volume, the pressure of the gas voids, the effects of the cooling rate to the gas voids volume and lastly is the composition of the gas voids itself.

The percentage of the gas voids does not shown any difference between fast cooling rate which is using 25 degree Celsius ambient temperature of laboratory and slow cooling rate which is using water as cooling medium for 1 hour where the percentage are 5.8% and 6.2% of the total volume respectively.

The pressure of the gas voids is negative which is under the atmospheric pressure. The average reading of the gas voids pressure is -0.23bar. Relating to the problem statement of this project, below the atmospheric pressure means that there are potential for compressing the gelled up crude.

Lastly, the composition of the gas voids is the part of the research and the result shows that gas voids only contain air. After conducting gas chromatography experiment for two times, it was shown that the there is no other gas in the gas voids instead of air.

In conclusion, most of the gas voids properties have high potential to achieve the goal which is to prove that the gelled up crude can be compressed and the assumption that gelled up crude behave as incompressible high viscous fluid should be revised in the future.

## **5.2 Recommendation**

There are some findings and challenges faced throughout conducting the research project. Therefore, some recommendations can be implemented so that there will be improvement for further study.

To make the study of the crude and gas voids easier, some properties need to be considered to fit the crude to the facility provided. The Wax Appearance Temperature, Pour Point and Viscosity of the crude must be considered first.

High Wax Appearance Temperature and high Pour Point need facility that can provide heat around the experimental setup since ambient room temperature will cause the crude to form wax.

For the gas voids study, high viscosity crude must be avoided since the high viscosity of crude causes the gas voids to trap between the gelled up crude and it is hard to calculate the actual volume of gas voids.

## LITERATURE REFERENCES

1. "Petroleum Waxes" G. A. Mansoori, H. Lindsey Barnes and Glen M. Webster, Chapter 19
2. "Fuels and Lubricants Handbook", ASTM Int'l, West Conshohocken, PA, Pages 525-558, 2003.
3. Vinay, G., Wachs, A., & Frigaard, I. (2009). Start-up of Gelled Waxy Crude Oil Pipelines: A New Analytical Relation to Predict the Restart Pressure. Asia Pacific Oil and Gas Conference Exhibition. Society of Petroleum Engineers.
4. Michele Margarone, SPE, Gianpaolo Borghi, SPE, & Sebastiano Corra, SPE, Eni Exploration and Production (2010). One Dimensional Modelling and Experimental Validation of Gelled Waxy Oils Restart. North Africa Technical Conference and Exhibition, 14-17 February 2010, Cairo, Egypt
5. C.W Sum. Petroleum Exploration, Nature Of Petroleum –Pt 1 (2011).
6. Phillips, David A., Forsdyke, Ivor N., McCracken, Ian R Ravenscroft, Paul D. Novel Approaches to Waxy Crude Restart: Part 1: Thermal shrinkage of waxy crude oil and the impact for pipeline restart, Journal of Petroleum Science and Engineering (2010), doi: 10.1016/j.petrol.2010.11.009
7. A. Jaafar, S.A. Sulaiman, I. Ismail, Thermal Shrinkage And Yield Stress Of Statically And Dynamically Cooled Waxy Crude (2011)