

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

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Dissertation submitted in partial fulfillment of  
the requirements for the  
Bachelor of Engineering (Hons)  
(Mechanical Engineering)

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## **CERTIFICATION OF APPROVAL**

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Approved by,

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May 2012

## **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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MOHD NAZMI B MOHAMAD SAIFULL BAHRI

## **ABSTRACT**

This report discussed on thermal shrinkage and gas voids formation of waxy crude oil. The objectives will be mainly on the effect of final temperature to the gas voids volume, pressure and composition and the result of aging process to the formation of gas voids. This report begins with the background study of the formation of gas voids inside pipeline. The limitations of current practice commonly lead to the adoption of wide margins of safety. This result in the needs to use high pressure in order to clear up the gelled crude oil formed within the pipelines. Literature reviews on the project composed of the behaviors of the formation of gas voids which is being done at the initial phase of the project. The methodologies and results from some journals are collected and compared. Next is the experimental work. During this phase, the simulation of actual problem faced is being conducted through experimental procedures and equipment in laboratory. A sample is tested with different parameters which are final temperature of 20°C, 15°C and 10°C. For the experiment regarding the effect of aging period, the sample is tested with aging period of 0 and 30 minutes at the temperature of 20°C. From here, analysis is done with respect to three respective parameters which are gas voids volume, pressure and composition. The findings are the gas voids pressure is lesser than the ambient pressure and it contains air properties. The second one is the volume of the gas voids increase as the final temperature of the crude decreases. Lastly, with regards to limited parameters it is observed that is no effect of aging process to the volume and pressure of gas voids.

## ACKNOWLEDGMENT

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## CHAPTER 1

### PROJECT BACKGROUND

#### 1.1 Background of Study

Petroleum currently has become a great deal in operating machines and has a great demand from every country in the world. It can be acquired either on land or under the seabed. It is transported using pipeline from the earth crust to the surface. In the oil and gas industry, about 20% of the petroleum reserves produced and pipelined are crudes containing large proportions of high molecular weight compounds, also known as waxy crude oils<sup>[1]</sup>.

During the maintenance or emergency situation, the crude oil inside of the pipeline will be in static condition. Thus, temperature within the pipeline drops significantly due to external factors and this lead to the formation of paraffin crystals. This occurs due to the temperature reduced to seabed temperature which is below the Wax Appearance Temperature (WAT).

The crude oil that undergoes thermal shrinkage will eventually cause the gel crude to form gas voids. The limitations of current practice commonly leads to the adoption of wide margins of safety leads to the usage of high pressure in order to clear the gelled crude out.

Thus the facility such as pump designed to have superior horse power is then needed and will then eventually increase both the capital and operating costs. Reducing the pressure needed will lead in reducing the cost and then will eventually make the operator to gain more profits compared to existing conditions.

## **1.2 Problem Statement**

### **1.2.1 Problem Identification**

Nowadays, high restart pressure is being applied in order to clear the formation of gelled waxy crude oil. The gas void formation inside pipeline that appeared during thermal shrinkage or cooling of crude oil may affect the compressibility of gelled waxy crude. Aside of that, Gelled crude oil will plug the pipeline cause the issue of restarting. Plugged Pipeline requires large amount of restart pressure. Therefore, the study of the thermal shrinkage and void formation in gelled crude could possibly determine the accurate restart pressure.

### **1.2.2 Significance of the Project**

This study contributes in identifying the accurate restart pressure if the gas void compressibility could be identified.

## **1.3 Objective and Scope of the Study**

- To study the formation of gas voids in gelled crude and also the effect of the final temperature to the gas voids volume.
- To assess the evolution of the gas voids when the gel is aging.

The scope of study is mainly to study the behavior of the gas void inside the waxy crude oil as well as its characteristic in term of volume, pressure and components.

#### **1.4 Relevancy 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. The success of this study will contribute finding that prove the gelled crude are compressible, thus reducing energy usage as well as can give more profit with less cost.

#### **1.5 Feasibility of the Project within the Scope and Time Frame**

This project is about the experimental simulation of the thermal shrinkage of waxy crude oil in the pipeline that lay on the seabed under not operating condition which means the petroleum crude remains in the pipeline statically. In doing this project, we will use the U-tube to monitor the behavior of crude oil as it undergoes its temperature reduce to seabed temperature. The U-tube will be used to store the crude oil during the experiment. For the laboratory facilities, the water bath or chiller has been provided and the other main machine need to be used is Gas Chromatography machine for the gas voids composition analysis purpose.

For the time and cost allocation, the experimental setup is being prepared and this experiment only consume a reasonable period of time to be completed. 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

#### 2.1 Thermal Shrinkage and Gas Void Formation

This project is mainly about the study of the gas void that happens during the thermal shrinkage of the waxy crude oil. 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 <sup>[2]</sup>. Pipelines transporting waxy crude oils may be shut down for normal operational reasons and also for emergency reasons for example like line damage, power failure or even earthquake <sup>[3]</sup>. Thermal shrinkage occurs as the crude oil is subject to cooling process due to the low temperature of seabed during static condition.

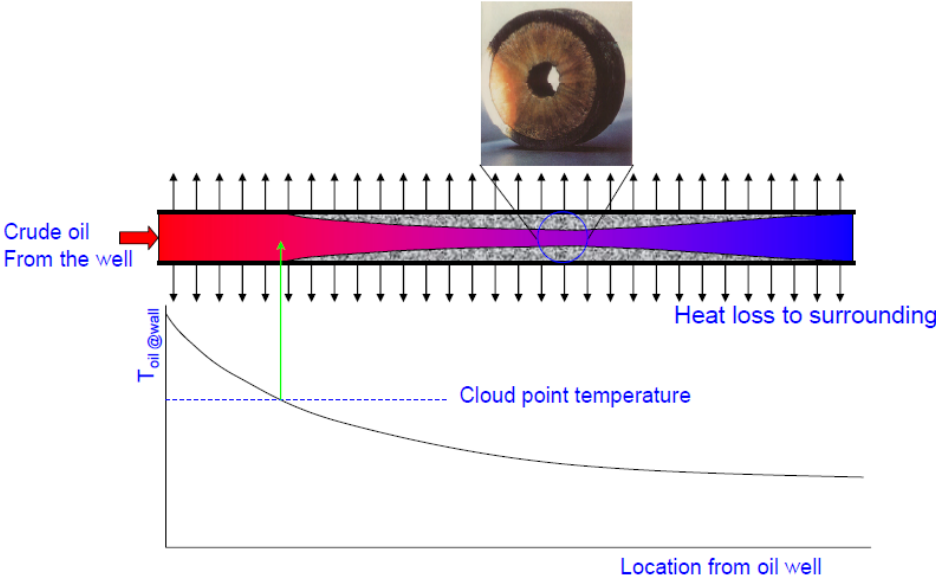
At higher temperatures, generally above 30-40°C, most crude oils behave as simple Newtonian liquids <sup>[4]</sup>. As it started to go down until it reaches Wax Appearance Temperature (WAT), wax crystals or gelled crude starts to form. As the temperature goes down, void formation inside of the crude oil will be taken place.

Aside of that, because of the presence of the gelled crude, waxy crude oils display complex rheology and at least eight parameters affecting their flow behavior have been identified <sup>[5]</sup>. Out of eight parameters, there are three variables that are important variables to this study which are thermal history, shear history and composition.

The restart of the gelled oil may also result from the breakdown of the gel structure itself (cohesive failure) or it may occur because of the breakage at the pipe-gel interface (adhesive failure) depending on the cooling rate and wax content <sup>[6]</sup>. This shows that the gelled oil itself could be compressed and will eventually decrease the restart pressure being used.

In addition, the restart pressure required after plant shutdown is significantly high to ensure the flow ability of the gelled crude which eventually will increase the energy usage and cost. Therefore, research in proving the compressibility of gas voids would cause a great deal to oil and gas industry.

On the other hand, another phenomenon occurs in pipeline is wax precipitation. Wax precipitation during crude oil flow causes wax deposition and flow restriction. Wax deposition can only occur when the inner pipe wall temperature is below the cloud point temperature. The precipitated wax molecules near the pipe wall start to form an incipient gel at the cold surface. The incipient gel formed at the pipe wall is a 3-D network structure of wax crystals and contains a significant amount of oil trapped in it<sup>[7]</sup>. The incipient gel grows as time progresses while there are radial thermal and mass transfer gradients as a result of heat losses to the surroundings as shown in Figure 1:



: Wax deposition occurs when the inner wall temperature is below the cloud point temperature.

Figure 1: Wax Deposition

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 proven that there were gas voids produced by cooling process of the crude oil in the flow line<sup>[8]</sup>. This proves that gas voids formation may affect the compressibility of the gelled crude inside the pipeline thus

could reduce the restart pressure of the operation. Figure below shows the illustration of formation of gas void as the temperature reduced to the seabed temperature.

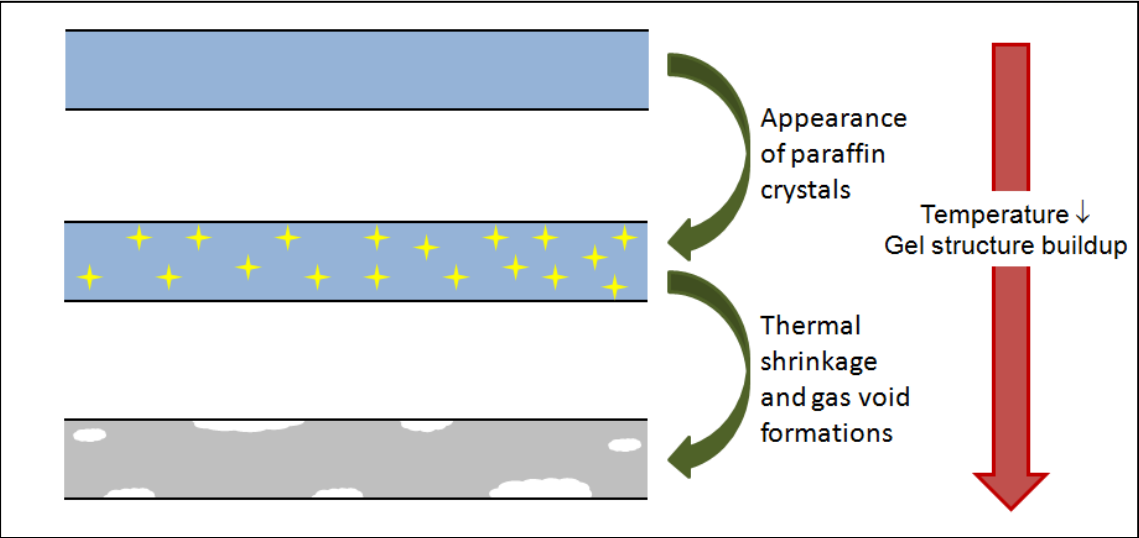


Figure 2: Wax formation stage

Therefore, this study will employ certain considerations such as the size of the gas voids form at constant and variable cooling rate; the pressure of the gas voids itself as well as the composition of gas void formed. This study will be mainly focused on the void formation of crude oil as it undergoes thermal shrinkage inside the pipeline.

**CHAPTER 3**  
**METHODOLOGY**

**3.1 Research Methodology**

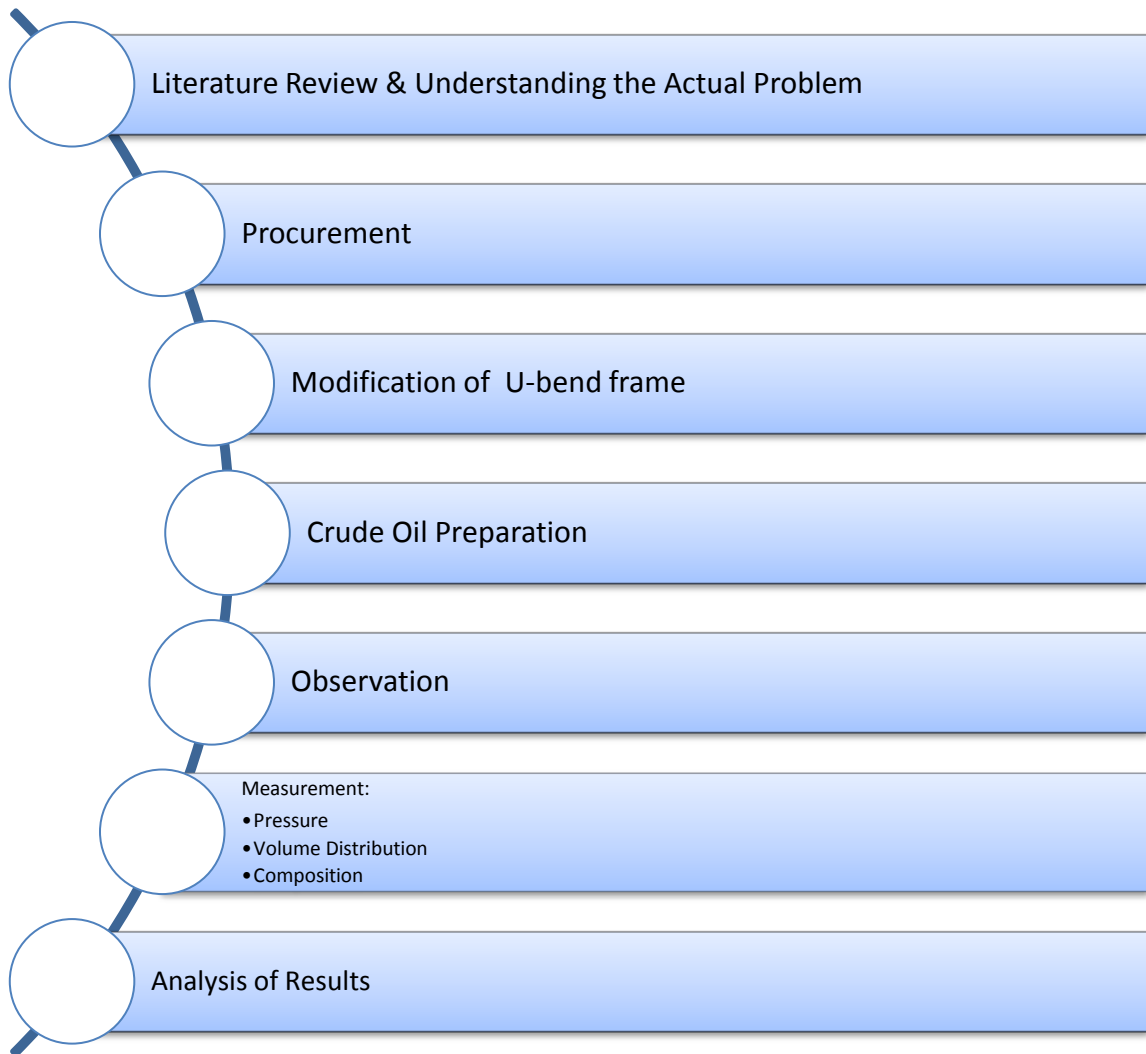


Figure 3: Project Research Methodology

### 3.2 Experiment Apparatus

#### 3.2.1 Bubble line flow rig

- It consists of:

- Polyurethane tube with internal diameter of 8mm
- PVC stopcock valve
- U-bend frame made of clear color acrylic sheet

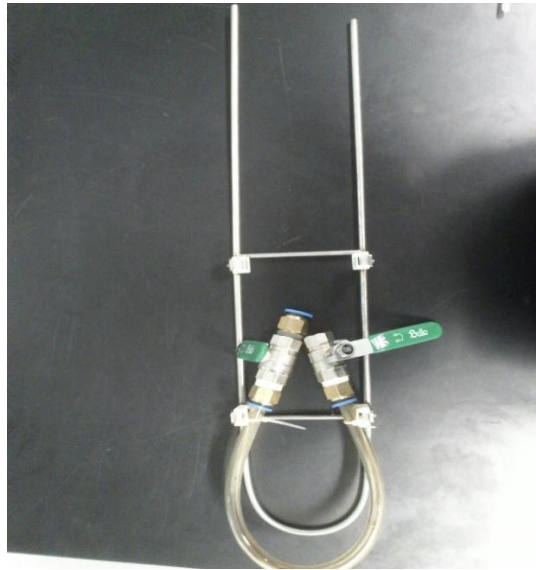


Figure 4: Bubble line flow rig.

#### 3.2.2 Vernier caliper

- To measure the gas voids volume formed within the tube.

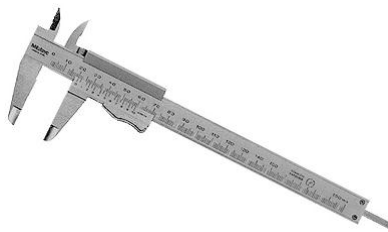


Figure 5: Vernier caliper.



### 3.2.3 Pressure transmitter

- To measure the gas voids pressure.

-Unit in mmHg



Figure 6: Pressure transmitter.

### 3.2.4 Vivo RT2 water bath

- It is used for heating and cooling of crude oil.

-Have fixed cooling rate



Figure 7: Vivo RT2 water bath.

### 3.2.5 Syringe

-To be tapped at the tube twice; one for pressure measurement and another one for gas composition.



Figure 8: Syringe.

### 3.2.6 Gas chromatography machine

- To determine the gas voids composition.

-Need assistant from technician in handling the machine



Figure 9: Gas chromatography machine.

### 3.3 Project Activities

The detailed activities of the project can be described below:

#### 3.3.1 Modification of U-bend Frame

Problem:

After the first trial of experiment has been done, it is found out that the length of the U-bend frame is too long.

Modification:

- i. By using abrasive cutter machine, the frame is cut to make the length from the top until the other end is 15 cm.
- ii. Gas tungsten arc welding machine (GTAW) is used to position of tube's support to the top.



Figure 10: Smoothing of the frame's surface.

Purpose:

- i. The frame is fully immersed inside the water bath.
- ii. The frame is positioned as inverted-U rather than U-shape for the voids to form at the apex.

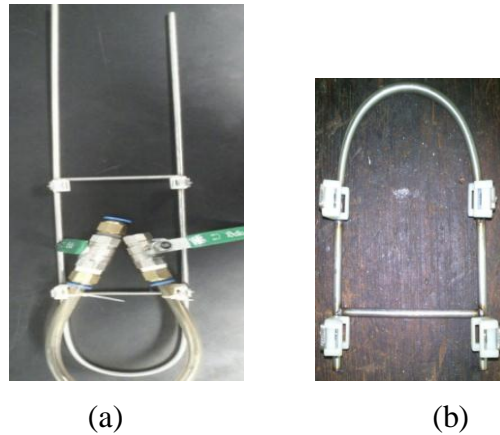


Figure 11: Modification of U-bend frame (a) before (b) after cut.

### 3.3.2 Determining the Best Experimental Protocol

After modification of the frame, there are several trial experiments done in order to get the best standard experimental protocol so that the gas voids appear at the apex.

Problem:

- i. The voids formed at the side at the tube rather than at the apex,
- ii. Water seeps through the connection between valve and tube, Figure 11.



Figure 12: Water present inside the tube.

Modifications:

- i. Original length of tube is reduced from 30 cm to 20 cm. The hypothesis is shorter tube's length will provide enough time for the voids to migrate to the apex as crude oil become more solidified at low temperature.
- ii. Secondly, the usage of cling film around the connection of tube and valves able to avoid water from seeping through.

After implementing the suggested steps, the voids formed are at the apex and continuous. After the best protocol has been determined, the real experiments are started.



Figure 13: Voids formed at apex.

### 3.3.3 Preparation of Crude Oil

Problem:

- i. As the crude oil is being stored in a long period, it tends to separate to heavy part and light part due to density difference.
- ii. Thermal Shock occurs when the hot crude oil is filled inside a non-heated tube due to temperature difference.

Solutions:

- i. Before doing the experiment, the crude is heated up to  $60^{\circ}\text{C}$  for two hours of agitation process to allow normal mixing process.
- ii. Before filling crude oil inside tube, the tube is heated to the same temperature of crude oil temperature which is  $60^{\circ}\text{C}$ .

Reasons:

- i. This is to make sure that the crude becomes homogeneous and at  $60^{\circ}\text{C}$ , the crude will behave as Newtonian fluid.
- ii. To avoid thermal shock and avoid the crude from solidified as it enters the tube.

### 3.4 Process Flow of Methodology

In doing this study of thermal shrinkage of the waxy crude oil, it involves the following process:

- 1) Firstly, the U-tube with crude oil inside it will be immersed in the water bath for 30 minutes at 60 °C and left to be cooled to 20°C right after 30 minutes of heating.
- 2) The gas void formation is then observed. Every 5°C interval, the condition of the crude oil inside the U-tube is recorded and picture of it is taken for analysis as shown in figure xx:

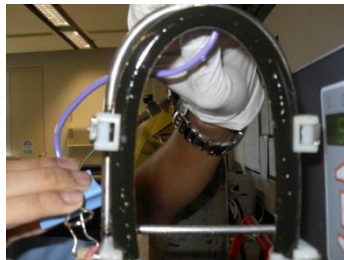


Figure 14: Observation of the tube every 5°C decrement

- 3) Then measurement of gas void is done at the apex of U-tube with respect of three parameters which are volume, pressure and gas composition. The details of procedure of the measurement taken are discussed below.
- 4) Then Step 1 to Step 3 is repeated using different parameters which are final temperature of 15°C and 10°C for the experiment involving the variation the final temperature.
- 5) On the other hand, for the experiment determining the effect of aging process, Step 1 to Step 3 is repeated using final temperature of 20°C with 30 minutes aging period.
- 6) In order to get average results so that it would be accurate, each experiment is repeated three times.
- 7) All the results are recorded and analyze.

### 3.4.1 Measurement of Gas Voids Volume

The measurement phase begins with the measurement of gas voids. Percentage of gas voids volume over total crude oil volume is calculated in order to examine the distribution of gas voids. The procedure is described as below:

- i. The diameter and length of the gas voids are measured directly at the tube by using vernier caliper.
- ii. Gas void volume over total crude volume is calculated.

### 3.4.2 Measurement of Gas Voids Pressure

The measurement of gas voids pressure is done using pressure transmitter. The procedure is described as below:

- i. A syringe is connected to a pressure transmitter to record the pressure.
- ii. Then the syringe is tapped at the apex of the tube where the gas voids are located due to pressure difference.
- iii. The reading is taken three times to calculate the average value

### 3.4.3 Measurement of Gas Voids Composition

Gas voids composition is determined by using gas chromatography. The additional procedure as below:

- i. A syringe is tapped at the apex of the tube.
- ii. The syringe is sealed.



Figure 15: Sealed Syringe before tested in Gas Chromatography

- iii. Finally, the gas inside the syringe will be transferred to the gas chromatography machine.
- iv. The result of the gas composition will be produced and further analysis will be done.

Note: pressure measurement and gas chromatography are done separately to avoid contamination of gas voids.

### **3.5 Key Milestones**

Several targets have been set for the FYP I and FYP II. Table 1 shows the project activities and key milestones for FYP I and FYP II respectively.



| <b>Project Title: THERMAL SHRINKAGE AND GAS VOID FORMATIONS STUDY OF WAXY CRUDE</b> |                |            |            |            |            |            |            |            |             |            |            |
|---|----------------|------------|------------|------------|------------|------------|------------|------------|-------------|------------|------------|
| <b>Project Tasks</b>  | <b>Project</b> |            |            |            |            |            |            |            |             |            |            |
|   | <b>2012</b>    |            |            |            |            |            |            |            |             |            |            |
|   | <i>Jan</i>     | <i>Feb</i> | <i>Mar</i> | <i>Apr</i> | <i>May</i> | <i>Jun</i> | <i>Jul</i> | <i>Aug</i> | <i>Sept</i> | <i>Oct</i> | <i>Nov</i> |
| Project Title Selection   |                |            |            |            |            |            |            |            |             |            |            |
| <b>Planning &amp; research phase</b>  |                |            |            |            |            |            |            |            |             |            |            |
| Literature review<br>Rheology<br>Basics of rheology                                 |                |            |            |            |            |            |            |            |             |            |            |
| <b>Design, testing and analysis phase</b>   |                |            |            |            |            |            |            |            |             |            |            |
| Acquiring samples   |                |            |            |            |            |            |            |            |             |            |            |
| <b>Verification of thermal shrinkage</b>  |                |            |            |            |            |            |            |            |             |            |            |
| Design of flow line bubble rig  |                |            |            |            |            |            |            |            |             |            |            |
| Fabrication of flow line bubble Rig   |                |            |            |            |            |            |            |            |             |            |            |
| Testing and analysis  |                |            |            |            |            |            |            |            |             |            |            |
| <b>Presentation phase</b>   |                |            |            |            |            |            |            |            |             |            |            |
| Submission of Progress Report   |                |            |            |            |            |            |            |            |             |            |            |
| Pre-EDX   |                |            |            |            |            |            |            |            |             |            |            |
| Submission of Draft Report  |                |            |            |            |            |            |            |            |             |            |            |
| Submission of Dissertation  |                |            |            |            |            |            |            |            |             |            |            |
| Submission of Technical Paper   |                |            |            |            |            |            |            |            |             |            |            |
| Oral Presentation   |                |            |            |            |            |            |            |            |             |            |            |
| Submission of Project Dissertation  |                |            |            |            |            |            |            |            |             |            |            |

## CHAPTER 4

### RESULT AND DISCUSSION

#### 4.1 Sepat Crude Properties

From the crude properties table, it is shown that the Sepat wax Appearance Temperature is 35 degree Celsius and the pour point is 35 degree Celsius. This shows that Sepat crude is heavy oil or black oil which is very thick and has a high viscosity. Since the pour point is 35 degree Celsius which is quite low, the cooling process will take longer time for crude to form wax. Base on this criterion, the cooling temperature of the crude will be 20 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 Total crude volume

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.

$$\text{Total crude volume} = \pi(r)^2L$$

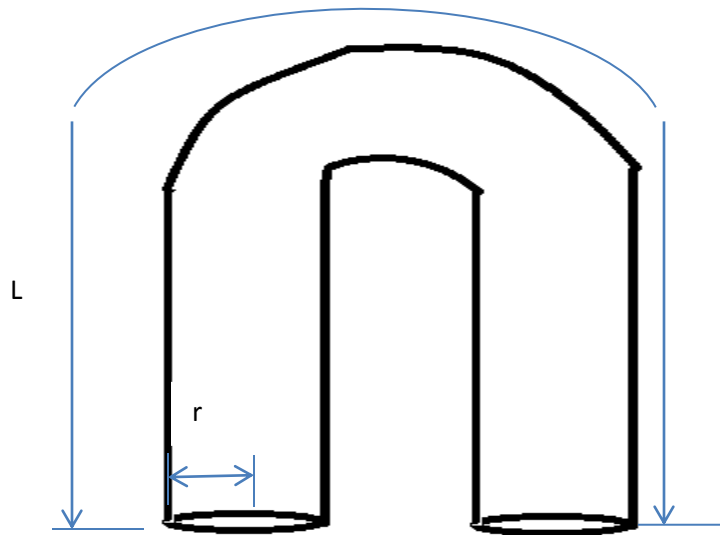


Figure 16 :2D diagram of crude tube

Where  $r = \text{Tube Diameter} / 2$

$$D = 0.8 \text{ cm} \quad L = 20 \text{ cm}$$

$$r = \frac{0.8}{2}$$

$$r = 0.4 \text{ cm}$$

$$\text{Total crude volume} = \pi (0.4)^2 (20)$$

$$\text{Total crude volume} = 10.05 \text{ cm}^3$$

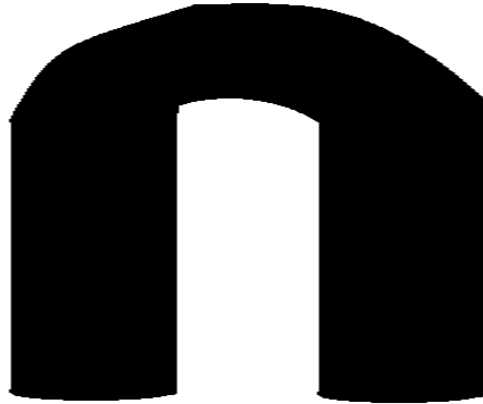


Figure 17: Tube with Sepat Crude Oil before Thermal Shrinkage

#### 4.3 Total Gas Voids Volume

Throughout the experiment, the condition of the crude oil inside tube is observed and each data is taken for further analysis. Below is the data gathered from the experiments:

##### i. Variation of Final Temperature Experiment

Objective:

- To determine the effect of final temperature on the gas void formation.

##### Experiment 1




Condition:




Final Temperature: 20<sup>0</sup>C




Length of tube: 20cm

Inner diameter of tube: 0.8cm

Results:

| Temperature | Time   | Condition   |
|-------------|--------|---|
| 60°C        | 2.46pm | No gas void detected<br>   |
| 55°C        | 2.56pm | No gas void detected<br>  |
| 50°C        | 3.06pm | No gas void detected<br> |

|                   |        |   |
|-------------------|--------|---|
| 45 <sup>0</sup> C | 3.18pm | No gas void detected<br>   |
| 40 <sup>0</sup> C | 3.29pm | No gas void detected<br>  |
| 35 <sup>0</sup> C | 3.41pm | No gas void detected<br> |

|      |        |   |
|------|--------|---|
| 30°C | 3.53pm | Gas Void detected<br>                  |
| 25°C | 4.07pm | Gas void grow<br>                     |
| 20°C | 4.20pm | Gas void detected along the tube<br> |

## Experiment 2



Condition:

Final Temperature: 15°C




Length of tube: 20cm


Inner diameter of tube: 0.8cm

Results:

| Temperature | Time   | Condition   |
|-------------|--------|---|
| 60°C        | 3.12pm | No gas void detected<br> |
| 30°C        | 4.15pm | Gas Void detected<br>   |



|                        |               |   |
|------------------------|---------------|---|
| <p>25<sup>0</sup>C</p> | <p>4.27pm</p> | <p>Gas void grow</p>    |
| <p>20<sup>0</sup>C</p> | <p>4.39pm</p> | <p>Gas void detected along the tube</p>  <p>Tube at the front</p>  <p>Tube at the back</p> |

|                   |        |   |
|-------------------|--------|---|
| 15 <sup>0</sup> C | 4.52pm | Gas Void detected at the front and the back of the tube<br> |
|-------------------|--------|---|

### Experiment 3



Condition:




Final Temperature: 10<sup>0</sup>C


Length of tube: 20cm

Inner diameter of tube: 0.8cm

Results:

| Temperature       | Time    | Condition   |
|-------------------|---------|---|
| 60 <sup>0</sup> C | 11.20am | No gas void detected<br> |
| 30 <sup>0</sup> C | 12.20pm | Gas Void detected<br>   |

|                        |                |   |
|------------------------|----------------|---|
| <p>25<sup>0</sup>C</p> | <p>12.32pm</p> | <p>Gas void grow</p>    |
| <p>20<sup>0</sup>C</p> | <p>12.43pm</p> | <p>Gas void detected along the tube</p>                          |
| <p>15<sup>0</sup>C</p> | <p>12.55pm</p> | <p>Gas Void detected at the front and the back of the tube</p>  |

|                   |        |  |
|-------------------|--------|--|
| 10 <sup>0</sup> C | 1.08pm | Gas Void detected at the front and the back of the tube grow<br> |
|-------------------|--------|--|

ii. **Variation Aging Period Experiment**

Objective:

- To assess the evolution of the gas voids when the gel is aging

**Experiment 1**

Condition:



Final Temperature: 20°C

Aging Period: 0 Minutes

Length of tube: 20cm

Inner diameter of tube: 0.8cm

Results:


| Temperature | Time    | Condition   |
|-------------|---------|---|
| 60°C        | 12.18am | No gas void detected<br> |
| 55°C        | 12.28am | No gas void detected<br> |

|      |         |  |
|------|---------|--|
| 50°C | 12.38am | No gas void detected<br>   |
| 45°C | 12.48am | No gas void detected<br>  |
| 40°C | 12.59am | No gas void detected<br> |



|      |        |  |
|------|--------|--|
| 35°C | 1.12pm | No gas void detected<br> |
| 30°C | 1.24pm | Gas Void detected<br>   |
| 25°C | 1.37pm | Gas void grow<br>      |



|      |        |  |
|------|--------|--|
| 20°C | 1.50pm | Gas void detected along the tube<br> |
|------|--------|--|

## Experiment 2

Condition:


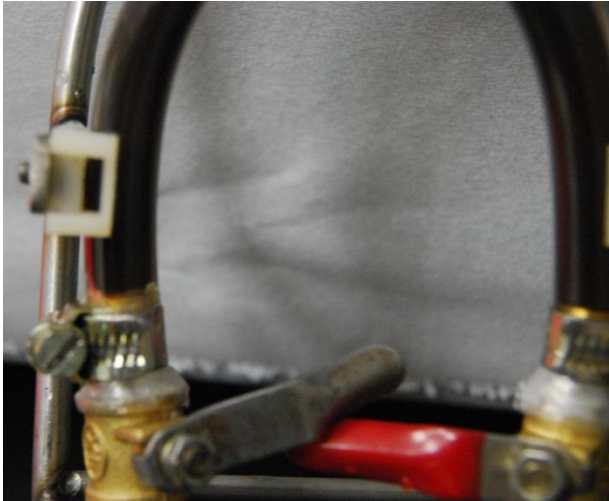
Final Temperature: 20°C




Aging Period: 30 Minutes

Length of tube: 20cm

Inner diameter of tube: 0.8cm

Results:

| Temperature | Time   | Condition   |
|-------------|--------|---|
| 60°C        | 5.25pm | No gas void detected<br> |
| 30°C        | 6.31pm | Gas Void detected<br>   |

|                                      |               |   |
|--------------------------------------|---------------|---|
| <p>25°C</p>                          | <p>6.43pm</p> | <p>Gas void grow</p>                      |
| <p>20°C</p>                          | <p>6.57pm</p> | <p>Gas void detected along the tube</p>  |
| <p>20°C<br/>After 30<br/>minutes</p> | <p>7.27pm</p> |   |

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. Gas voids inside the tube form along the tube. As the final temperature decreased the gas void volume is increased.

The geometry of the gas voids that form inside the tube is in cylinder shaped at temperature 20<sup>0</sup>C and in the form of hollow shaped at temperature 15<sup>0</sup>C and 10<sup>0</sup>C. So the gas voids volume is calculated with the reference of the diameter of gas void that formed inside the tube that is measured using a vernier caliper.

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.

i. **Variation of Final Temperature Experiment**

**Experiment 1 – Final temperature equals to 20<sup>0</sup>C**

Assumption: gas voids volume is in the shape of cylinder

Calculation for Gas voids Volume:

$$\begin{aligned}\pi j^2L &= \pi (0.2/2)^2(20) \\ &= 0.628 \text{ cm}^3\end{aligned}$$

**Experiment 2-Final temperature equals to 15<sup>0</sup>C**

Assumption: gas voids volume is in the shape of hollow

Gas voids Volume:

$$\begin{aligned}\pi j^2_{\text{large}}L - \pi j^2_{\text{small}}L &= \pi (0.4)^2(20) - \pi (0.38)^2(20) \\ &= 0.98 \text{ cm}^3\end{aligned}$$

### Experiment 3-Final Temperature equals to 10°C

Assumption: gas voids volume is in the shape of hollow

Gas voids Volume:

$$\pi j^2_{\text{large}}L - \pi j^2_{\text{small}}L = \pi (0.4)^2(20) - \pi (0.335)^2(20)$$
$$= 3.00 \text{ cm}^3$$

| Final Temperature<br>20 C |             | Final Temperature<br>15 C |        | Final Temperature<br>10 C |        |
|---------------------------|-------------|---------------------------|--------|---------------------------|--------|
| Exp                       | Volume(cm3) | Exp                       | Volume | Exp                       | Volume |
| 1st Exp                   | 0.628       | 1st Exp                   | 2.00   | 1st Exp                   | 3.00   |
| 2nd Exp                   | 0.628       | 2nd Exp                   | 0.88   | 2nd Exp                   | 3.17   |
| 3rd Exp                   | 0.628       | 3rd Exp                   | 0.98   | 3rd Exp                   | 3.17   |

Table 1: Gas Voids volume at 20°C, 15°C and 10°C

#### ii. Variation Aging Period Experiment

##### Experiment 1 – Final temperature equals to 20°C and aging period equals to 0 min

Gas voids Volume:

$$\pi j^2L = \pi (0.139)^2(20)$$
$$= 0.388 \text{ cm}^3$$

##### Experiment 2-Final temperature equals to 20°C and aging period equals to 30 min

Gas voids Volume:

$$\pi j^2L = \pi (0.139)^2(20)$$
$$= 0.388 \text{ cm}^3$$

| Aging Period : 0 minutes  |             | Aging Period : 30 minutes |             |
|---------------------------|-------------|---------------------------|-------------|
| Final Temperature<br>20 C |             | Final Temperature<br>20 C |             |
| Exp                       | Volume(cm3) | Exp                       | Volume(cm3) |
| 1st Exp                   | 0.353       | 1st Exp                   | 0.353       |
| 2nd Exp                   | 0.459       | 2nd Exp                   | 0.465       |
| 3rd Exp                   | 0.353       | 3rd Exp                   | 0.353       |

Table 2: Gas voids volume at 20°C at different aging period.

#### 4.4.1. Percent of Gas Voids Volume Using Water Bath

##### i. Variation of Final Temperature Experiment

For the experiment, the cooling at 20 degree Celsius had been done by cooling the crude using the water bath.

The gas shape is assumed to be in cylinder shaped at 20°C and hollow shaped at 15°C and 10°C for easier calculation.

##### **Experiment 1 – Final temperature equals to 20°C**

Calculation for Gas voids Volume: 0.628 cm<sup>3</sup>

$$\text{Gas voids percentage} = \frac{0.628}{10.05} * 100$$

Gas voids percentage = **6.2%**

##### **Experiment 2-Final temperature equals to 15°C**

Gas voids Volume: 0.98 cm<sup>3</sup>

$$\text{Gas voids percentage} = \frac{0.98}{10.05} * 100$$

Gas voids percentage = **9.8%**

**Experiment 3-Final Temperature equals to 10°C**

Gas voids Volume: 3.00 cm<sup>3</sup>

$$\text{Gas voids percentage} = \frac{3.00}{10.05} * 100$$

Gas voids percentage = **29.9%**

**ii. Variation Aging Period Experiment**

The gas shape is assumed to be in cylinder shaped at 20°C for easier calculation.

**Experiment 1 – Final temperature equals to 20°C and aging period equals to 0 min**

Gas voids Volume: 0.388 cm<sup>3</sup>

$$\text{Gas voids percentage} = \frac{0.388}{10.05} * 100$$

Gas voids percentage = **3.9%**

**Experiment 2-Final temperature equals to 20°C and aging period equals to 30 min**

Gas voids Volume: 0.388 cm<sup>3</sup>

$$\text{Gas voids percentage} = \frac{0.388}{10.05} * 100$$

Gas voids percentage = **3.9%**

#### 4.4 Cooling Rate

Cooling rate for the water bath is fixed for all experiments. Therefore, by making experiment 1 from the variation of final temperature experiment as the reference point, the cooling rate of water bath is as follows:

Experiment 1 – Final temperature equals to 20<sup>0</sup>C

The cooling process is start at 60 degree Celsius to 20 degree Celsius

| Initial               | After Cooling Down     | Time required to cool down from 60 <sup>0</sup> C to 20 <sup>0</sup> C |
|-----------------------|------------------------|--|
| 3.55pm<br>Heat Up     | Pressure: -0.5 mmHg    | 94 min   |
| 4.32pm<br>Cooled Down | Volume= refer to above |  |

Table 3: Experiment 1 results.

$$\text{Cooling rate} = \frac{60^{\circ}\text{C} - 20^{\circ}\text{C}}{94 \text{ minutes}}$$

$$\text{Cooling rate} = 0.426 \text{ degree Celsius /minute}$$



#### 4.5 Gas Voids Pressure

##### i. Variation of Final Temperature Experiment

| Final Temperature<br>20 C |                | Final Temperature<br>15 C |                | Final Temperature<br>10 C |                |
|---------------------------|----------------|---------------------------|----------------|---------------------------|----------------|
| Exp                       | Pressure (kPa) | Exp                       | Pressure (kPa) | Exp                       | Pressure (kPa) |
| 1st Exp                   | -0.0399        | 1st Exp                   | -0.0399        | 1st Exp                   | -0.0533        |
| 2nd Exp                   | -0.0533        | 2nd Exp                   | -0.0533        | 2nd Exp                   | -0.0666        |
| 3rd Exp                   | -0.0533        | 3rd Exp                   | -0.0533        | 3rd Exp                   | -0.0666        |

Table 4: Gas Voids pressure at 20°C, 15°C and 10°C

##### i. Variation Aging Period Experiment

| Aging Period : 0 minutes  |                | Aging Period : 30 minutes |                |
|---------------------------|----------------|---------------------------|----------------|
| Final Temperature<br>20 C |                | Final Temperature<br>20 C |                |
| Exp                       | Pressure (kPa) | Exp                       | Pressure (kPa) |
| 1st Exp                   | -0.0399        | 1st Exp                   | -0.0399        |
| 2nd Exp                   | -0.0533        | 2nd Exp                   | -0.0399        |
| 3rd Exp                   | -0.0533        | 3rd Exp                   | -0.0399        |

Table 5: Gas Voids pressure at 20°C at different aging period

## 4.6 Gas chromatography

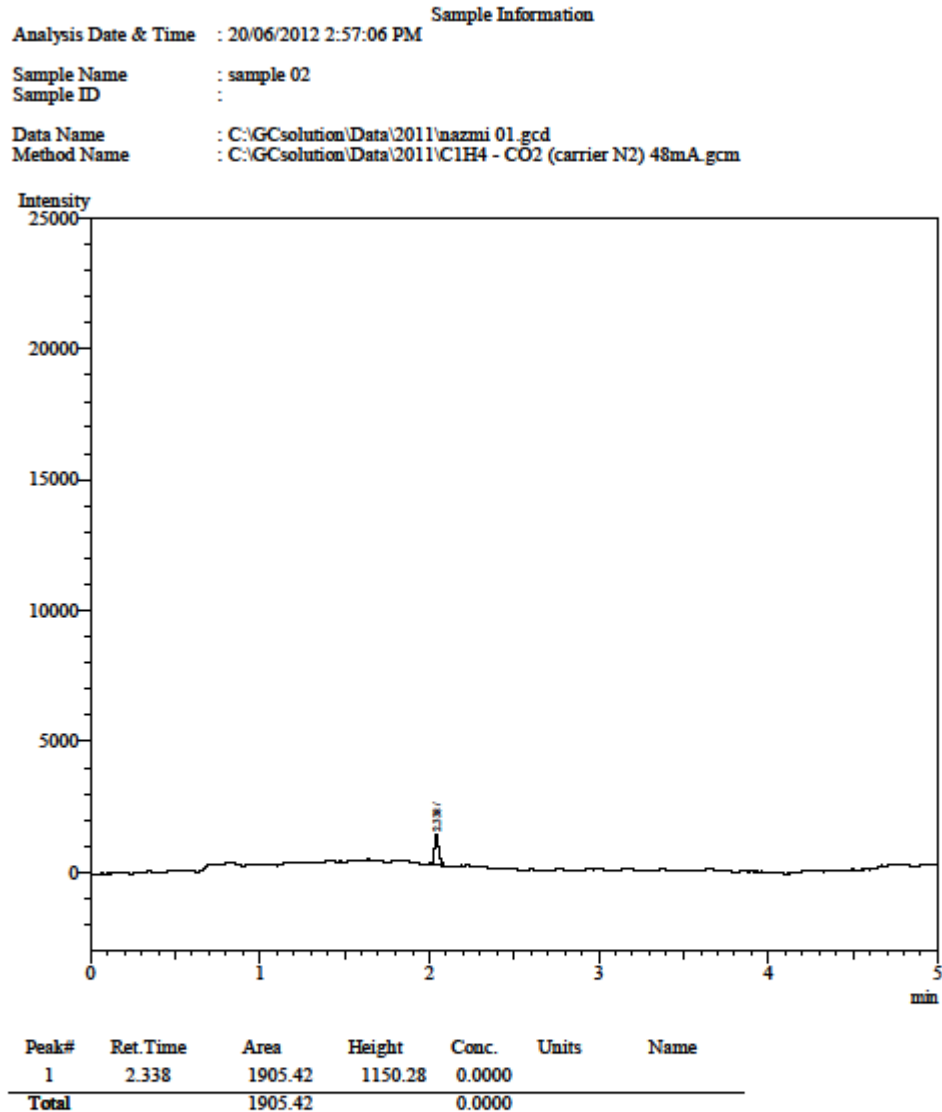


Figure 18: Gas Chromatography result

Gas Chromatography is a graph of intensity vs time. It use flame ionization detector to detect the composition of fluids. Based on the Gas Chromatography result, it shows that at retention time of 2.338s, there is an existence of air inside the gas voids with area of 1905.42.

The area represents the concentration of particular elements in certain fluids. It has no unit as it is in the form of a ratio. Normally, a fluid that contains several elements will result of several peaks in the graph to show the concentration of each element. However, based on the

graph above, a single peak is produced. Thus proved that, there is an existence of air in the gas void properties.

## DISCUSSIONS

Based on the results obtained, graphs are being constructed as follow:

### i. Variation of Final Temperature Experiment

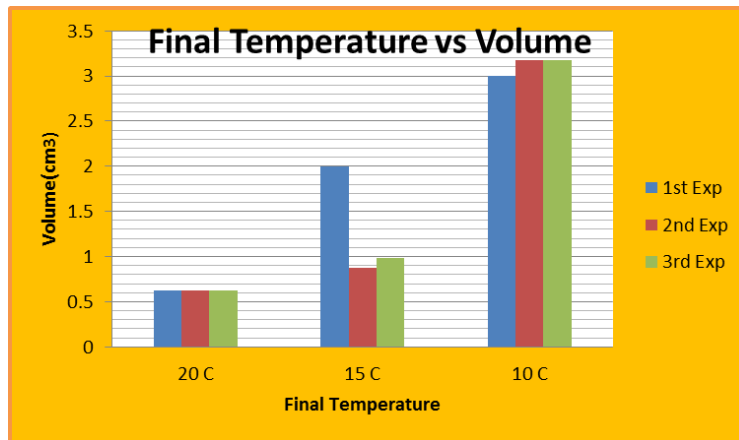


Figure 19: Final Temperature Vs Volume Graph

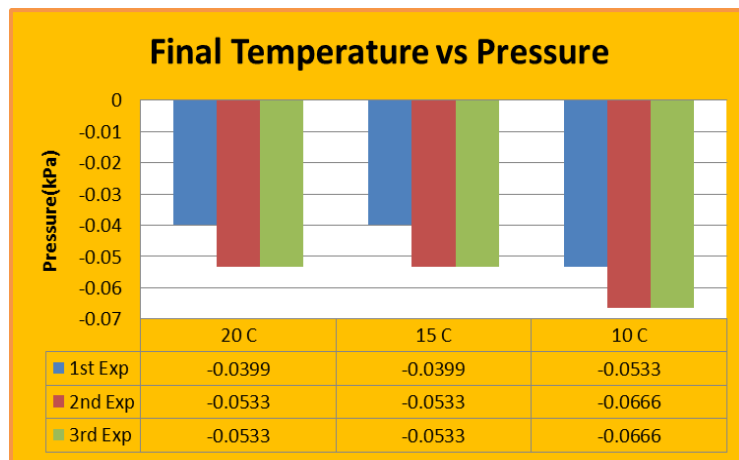


Figure 20: Final Temperature Vs Pressure Graph

For the variation of final temperature the results show that at 20<sup>0</sup>C, the volume of gas voids formed is 0.628 cm<sup>3</sup> with pressure between -0.04 to -0.05 kPa which is below ambient pressure. As the final temperature decrease, the volumes increased while the pressure being within the range.

ii. Variation Aging Period Experiment

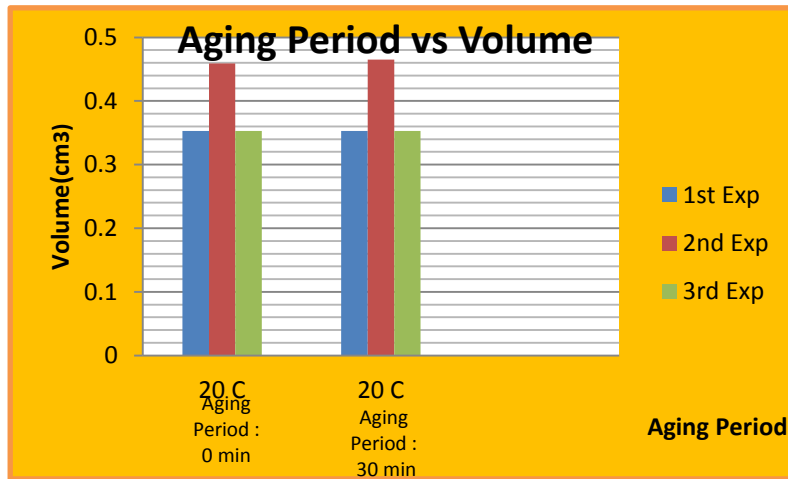


Figure 21: Aging Period Vs Volume Graph

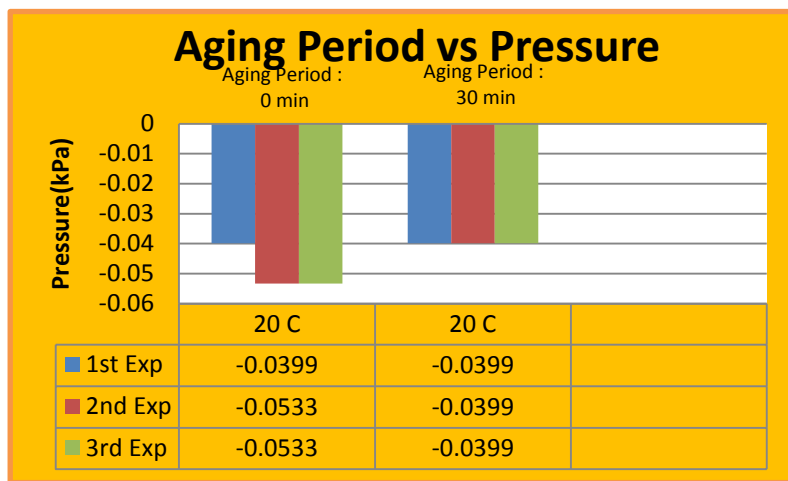


Figure 22: Aging Period Vs Pressure Graph

For the effect of aging process, based on the result, at condition of 20<sup>0</sup>C for 0 min aging period and 20 <sup>0</sup>C for 30 min aging, the results show no significant difference. The pressure obtained is between the ranges of -0.04kPa to -0.07kPa.

## CHAPTER 5

### CONCLUSION

#### 5.1 Conclusion

In a conclusion, this project is a research about gas voids that form after the thermal shrinkage of the waxy crude oil. Through the experiment it is clearly proven that gas voids will eventually appear below the pour point. The gas voids pressure is lesser than the ambient pressure and it contains air properties. By varying the final temperature of the cooling process, the gas voids volume differs. The lower the final temperature, the higher the volume of the gas voids appeared. At 20<sup>0</sup>C the gas voids form taking the shape of cylinder while at the temperature of 15<sup>0</sup>C and 10<sup>0</sup>C, the gas voids is in hollow shaped. By having shrinkage at top and bottom of pipe will enhance pipe-gel interface breakage.

On the other hand, for the second type of experiment which is done by manipulating the aging period of crude oil; it is found out that there is no effect of aging process to the volume and pressure of gas voids. The graphs obtained show no difference between aging period of 0 min and 30 min for the temperature at 20<sup>0</sup>C.

Besides that, the gas voids pressure is lesser than the ambient pressure and it contains air properties. Lastly, 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 Recommendations

Throughout this project, there are several improvements that could be done for future work:

1. Ways to get the pressure correctly to avoid contamination as the pressure is below ambient pressure and the quantity of gas voids that should be assess in GC machine.
2. Compressive study on the light part of crude oil that consists of carbon dioxide, nitrogen, methane, ethane and propane which may be the composition of the gas voids instead of air. It is said that from a research and experiments conduct by my fellow friends in Petroleum department, as they manipulating the pressure of crude oil, the light part of crude oil will bubble out forming gas inside tube. Therefore, I assume that, the gas voids that form due to the decreasing of temperature may consist of the gases from light part of crude oil.
3. Further study using different types of Crude Oil in Malaysia which is waxier or less waxy crude such as Penara and parameters such as shear stress and strain will strengthen the facts of high potential of gas voids compressibility.
4. Using tube with bigger Inner Diameter to see whether the similar shrinkage pattern is observed.
5. Conduct similar experiment but cooling under dynamic condition such as using flow loop.
6. To vary the cooling rate. Different cooling rate will have different effect on the formation of gas void
7. To analyze the void volume by using CATIA drawing, by which the calculations by pixel of the void formed can be made, rather than assuming it as a cylinder
8. Use different type of crude with lighter part. This will give higher chance of getting more than one component during gas chromatography
9. To implement a more accurate method of obtaining the gas sample. This can be done by having two syringes instead of one where one syringe will be filled with a known composition inert gas. Then, both syringes are tabbed into the tube simultaneously. The syringe with inert gas will be pushed to displace the gas inside the tube so that the other empty syringe can suck it

10. To determine the thermal conductivity of the polyurethane tube and also to consider using an insulation
11. To investigate the structure of the wax formed using microscope
12. To perform heat transfer analysis in order to predict the cooling rate
13. To check the pressure obtained whether it satisfies the Ideal Gas Equation or not

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