

Rheological Characterization of Thixotropic Waxy Crude

by

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Dissertation submitted in partial fulfilment of

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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the
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Approved by,



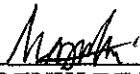
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UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK

May 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 or persons.



MUHAMMAD MARZUKI BIN MUHAMMAD KAMAL

ABSTRACT

This report basically discusses the research done of the chosen topic, which is **Rheological Characterization of Thixotropic Waxy Crude**. The objective of the project is to characterize the Thixotropic Waxy Crude with the effect of pre-shear and waiting time and understand the viscosity behaviour rheologically. The challenge in this project is dealing with the Waxy Crude itself, since it is a thixotropic Non-Newtonian substance and understanding the behavior is another thing. Lab testing was done on the waxy crude sample using Rheometer. The data obtained from the rheometer was analyzed and fitted to a suitable rheological model.

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CHAPTER 1

INTRODUCTION

1.1 Background of the Project

The rheological behaviour of waxy crude oils is a crucial parameter in the design of pipelines for transportation of crude oil and down-stream processing equipment. Most of these types of crude oils exhibit a combination of time-independent and time-dependent rheological properties. Hence, the measurement and modeling of the flow properties of these crude oils has been proven difficult (D. Tiwary, A. K. Mehrotra, 2002). The process of gel formation is a function of many variables, of which the most important are thermal history, shear rate and composition.

1.2 Problem Statement

In the oil and gas industry, transportation of crude oils containing large proportions of high molecular weight compounds presents a significant complexity and could cause many difficulties such as clogged pipelines especially during shut-down (Vinay et. al., 2005. Hoffman and Amundsen. 2010). Such crude oils are known as waxy crude oil. These crude oils are light or intermediate crude containing between 2 to 10% of paraffin wax (Vinay et. Al., 2005. Tinsley et. al., 2009). The waxy crude has high Wax Appearance Temperature (WAT) and Pour point and are thixotropic. i.e. the rheological behavior of the crude is shear dependent, apart from it being temperature dependent. The shear-dependence property is an indicator of the gelation behavior of the waxy crude. i.e. increasing yield stress and viscosity with time with decreasing shear. The formation of the paraffin crystals, or wax, is mainly dependent

on the temperature of the crude (Vinay et. al., 2005). Above the WAT, crude oil behaves mainly like a Newtonian liquid below which non-Newtonian behavior is increasingly significant (Vinay et. Al., 2005).

1.3 Objective and Scope of Study

The objectives of this project are to:

- To perform a rheological characterization of a waxy crude by experimental measurements using a rheometer and analyzing the rheological data by fitting them to a suitable rheological model.

The scope of this project will be simplified as follows:

- The waxy crude sample that will be used for the purpose of this study is taken from the Dulang Field
- Data obtained from rheometer will be analyzed and fitted to a suitable rheological model (e.g. Power Law)

CHAPTER 2

LITERATURE REVIEW

2.1 Waxy Crude

Most crude oils at higher temperatures, generally above 30-40°C, behave as simple Newtonian liquids. Below a certain temperature (known as wax appearance temperature or WAT), which is unique for different crude oil, wax crystals start to form and become suspended in the Newtonian base liquid in this case is the crude oil (D. Tiwary, A. K. Mehrotra, 2002). As the further reducing in temperature, wax crystals grow in size and precipitate from the solution. The precipitated wax may adhere to cold surfaces, which may give rise to problems during storage, transportation, handling and processing (D. Tiwary, A. K. Mehrotra, 2002). As more wax precipitates with a further lowering of temperature, the rheological behavior becomes distinctly non-Newtonian (Ronningsen, H.P, 1992). The non-Newtonian behavior exhibited by these waxy crude oils ranges from time-independent characteristics such as Bingham plasticity and pseudoplasticity to more complex time-dependent characteristics such as thixotropy (Mewis, J., 1979, Barnes, H.A., 1997). Ultimately, the interaction of wax crystals gives rise to an interlocking structural network, resembling polymer gels, that possesses a flow limit or yield stress (Billington, E.W., 1960).

2.1.1 Thixotropy

Thixotropy behaviour is when a non-Newtonian fluid decrease in viscosity (i.e. alteration/reduction of structure mechanical strength) with time under a uniform shear force, and the fluid is called a thixotropic fluid (Karan, K., Ratulowski, J. and German, P., 2000). Thixotropy of a non-Newtonian liquid plays an important role in the measurement of its flow properties (R. P. Chhabra and J. F. Richardson, 2008)

2.1.2 Wax Appearance Temperature (WAT)

Wax Appearance Temperature is the lowest temperature before the fluid start to exhibit the waxy characteristic. When the temperature is decreased, wax crystals started to grow and will precipitate from the fluid. As the fluid is cooled further toward the pour-point temperature (i.e. the lowest temperature at which it will pour), the individual wax crystals give rise to an interlocking structural network, which ultimately turns the crude oil into a gelled, solidlike state. It is pointed out that the wax deposit is not entirely solid; i.e., it consists of liquid and solid phase. (Nitin V. Bhat and Anil K. Mehrotra, 2004)

Thermodynamically, WAT is the true solid-liquid phase boundary temperature, i.e. it is the maximum temperature at which the solid and liquid phases exist in equilibrium (phase equilibrium) at a fixed pressure. However, the experimental WAT represents the temperature at which the first crystals are detected and, therefore, depends on the type and sensitivity of the measurement technique and the equipment used i.e. there seemed to be different WAT values obtained from different experimental techniques for the same crude (Ratulowski, 2000).

2.1.3 Time Effect

When crude oil was cooled to a specific temperature, wax crystals separated out from liquid hydrocarbon, forming a concentrated suspension with time effects and the thixotropic behavior is hence observed. Under constant shear rate, the shear stress gradually decreased with time until a dynamic equilibrium with constant shear stress is reached. (Dafan Yan and Zheming Luo, 1987) When the shear rate is changed to lower shearing or the shearing process stopped, the thixotropic system restore its original structure slowly with time, showing increased resistance to flow (Govier, G.W. and Aziz, K., 1972).

In the case of Daqing crude oil, under the constant shear rate applied to the crude oil, the time needed to exhibit thixotropy is in the range of 20 to 130 minutes with an average of 60 minutes. The viscosity will decrease with time until the equilibrium constant shear. (Dafan Yan and Zheming Luo, 1987) In this period, breakdown and aggregation of waxy structural units take place in such a way that equilibrium with flow resistance is essentially reached. After equilibrium is reached at a given shear rate, the fluid is kept at rest by stopping the shearing process for a period of time and again subjected to the same shear rate. The recovery of shear stress illustrates the restoration of the rheological structure (Bao, C. and Yan, D., 1983, Devlinkamof, V.V., Habibullin, Z.A., and Kabirol, M.M., 1975, Mewis, J., 1979)

2.2 Rheology

Rheology is the study of the flow of matter: primarily in the liquid state, but also as 'soft solids' or solids under conditions in which they respond with plastic flow rather than deforming elastically in response to an applied force (W. R. Schowalter, 1978). It applies to substances which have a complex molecular structure, such as muds, sludges, suspensions, polymers and other glass formers (e.g. silicates), as well as many foods and additives, bodily fluids (e.g. blood) and other biological materials.

The flow of these substances cannot be characterized by a single value of viscosity (at a fixed temperature). While the viscosity of Newtonian liquids normally varies with temperature, it is variations with other factors which are studied in within the subject of rheology. For example, ketchup can have its viscosity reduced by shaking (or other forms of mechanical agitation) but water cannot. Since Sir Isaac Newton originated the concept of viscosity, the study of variable viscosity liquids is also often called Non-Newtonian fluid mechanics (W. R. Schowalter, 1978).

2.3 Rheometer

A rheometer is a laboratory equipment used to measure the way in which a liquid, suspension or slurry flows in response to applied forces. It is used for those fluids which cannot be defined by a single value of viscosity and therefore require more parameters to be set and measured than is the case for a viscometer. It measures the rheology of the fluid.

There are two distinctively different types of rheometers. Rheometers that control the applied shear stress or shear strain are called rotational or shear rheometers, whereas rheometers that apply extensional stress or extensional strain are extensional rheometers (K. Walters, 1975). Rotational or shear type rheometers are usually designed as either a native strain-controlled instrument (control and apply a user-defined shear strain which can then measure the resulting shear stress) or a native stress-controlled instrument (control and apply a user-defined shear stress and measure the resulting shear strain).

As for extensional rheometers, the development has proceeded more slowly than shear rheometers, due to the challenges associated with generating a homogeneous extensional flow. Firstly, interactions of the test fluid or melt with solid interfaces will result in a component of shear flow, which will compromise the results. Secondly, the strain history of all the material elements must be controlled and known. Thirdly, the strain rates and strain levels must be high enough to stretch the polymeric chains beyond their normal radius of gyration, requiring instrumentation with a large range of deformation rates and a large travel distance

CHAPTER 3

METHODOLOGY/PROJECT WORK

3.1 Project Flow

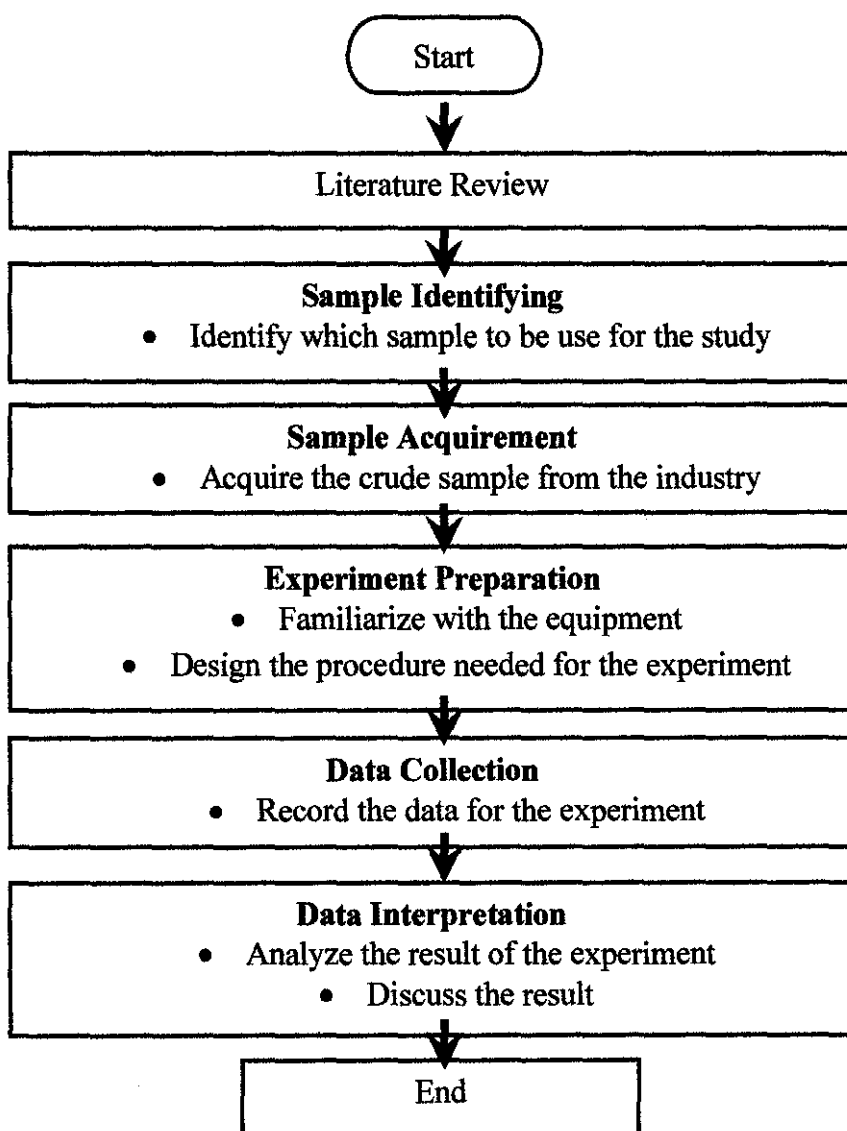


Table 1: Gantt Chart FYP1

| No | Task | 1 | 2 | 3 | 4 | 5 | 6 | MID-SEMESTER BREAK | | | | | | | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 |
|----|--|---|---|---|---|---|---|--------------------|--|--|--|--|---|---|---|---|---|----|----|----|----|----|
| 1 | Selection of Project Topic | █ | █ | | | | | | | | | | | | | | | | | | | |
| 2 | Literature Review & Research Work | | █ | █ | █ | █ | █ | | | | | | █ | █ | █ | █ | █ | █ | █ | █ | █ | █ |
| 3 | Submission of Preliminary Report | | | | ☺ | | | | | | | | | | | | | | | | | |
| 4 | Sample Procurement | | | █ | █ | █ | █ | | | | | | | | | | | | | | | |
| 5 | Submission of Progress Report | | | | | | | | | | | | | ☺ | | | | | | | | |
| 6 | Seminar | | | | | | | | | | | | | ☺ | | | | | | | | |
| 7 | Sample Procurement continue | | | | | | | | | | | | █ | █ | █ | █ | █ | █ | █ | █ | █ | █ |
| 8 | Rheometer Lab Familiarization | | | | | | | | | | | | | | | █ | █ | █ | █ | | | |
| 9 | Submission of Interim Report Final Draft | | | | | | | | | | | | | | | | | | | | | ☺ |
| 10 | Oral Presentation | | | | | | | | | | | | | | | | | | | | | |

☺ Suggested Milestone

█ Process

During Study Week

Table 2: Gantt Chart FYP2

| No | Task | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 |
|----|---|---|---|---|---|---|---|---|---|---|----|----|----|----|----|
| 1 | Experiment Preparation | █ | █ | █ | | | | | | | | | | | |
| 2 | Data Collection | | | | █ | █ | █ | | | | | | | | |
| 3 | Submission of Progress Report | | | | | | | ☺ | | | | | | | |
| 4 | Data Analysis | | | | | | | █ | █ | █ | | | | | |
| 5 | Pre-EDX | | | | | | | | | | ☺ | | | | |
| 6 | Submission of draft report | | | | | | | | | | | ☺ | | | |
| 7 | Submission of dissertation (soft bounded) | | | | | | | | | | | | ☺ | | |
| 8 | Submission of technical paper | | | | | | | | | | | | ☺ | | |
| 9 | Oral Presentation | | | | | | | | | | | | | ☺ | |
| 10 | Submission of project Dissertation (Hard bound) | | | | | | | | | | | | | | ☺ |

MID-SEMESTER BREAK

☺ Suggested Milestone

█ Process

3.3 Experimental Section

3.3.1 Sample Material

The waxy crude sample used in the experiment is from Dulang Oil Field which is acquired from PETRONAS Research Sdn Bhd (PRSB).

The sample of the crude oil has the following properties:

Table 3: Dulang Crude Oil Properties

| Properties | Value |
|------------------|-------------|
| Density @ 15°C | 0.8388 kg/l |
| API (degree) | 37.2 |
| Pour Point | +36°C |
| Sulphur | 0.0522 wt% |
| Wax Content | 3.0 wt% |
| Nitrogen Content | 124 ppm |
| Sodium (Na) | 6 ppm |

| Properties | Value |
|---------------|---------|
| Potassium (K) | 1 ppm |
| Copper (Cu) | 1 ppm |
| Lead (Pb) | < 1 ppm |
| Iron (Fe) | 2 ppm |
| Nickel (Ni) | 1 ppm |
| Vanadium | <1 ppm |
| Arsenic (As) | <1 ppm |

CHAPTER 4

RESULT & DISCUSSION

4.1 Data Gathering and Analysis

4.1.1 Viscosity Measurement with Water

The result for the water viscosity measurement as stated in the table below:

Table 4: Water Viscosity Result

| Temp °C | S. Stress (Pa) | Viscosity (cP) | RP M | S. Rate (s ⁻¹) | Log[Vi scosity] | Log[S. Rate] |
|---------|----------------|----------------|---------|----------------------------|--------------------|-----------------|
| 28 | 0 | 0 | 0 | 0 | - | - |
| 28.1 | 0.2 | 280 | 1 | 0.85 | 2.4471 | -0.0705 |
| 28.1 | 0.2 | 287.6 | 1 | 0.9 | 2.4587 | -0.0457 |
| 28.1 | 0.2 | 268.6 | 1 | 0.85 | 2.4291 | -0.0705 |
| 28.1 | 0.2 | 277.5 | 1 | 0.85 | 2.4432 | -0.0705 |
| 28.2 | 0.2 | 287.6 | 1 | 0.85 | 2.4587 | -0.0705 |
| 28.2 | 0.2 | 56.3 | 5 | 4.252 | 1.7505 | 0.6285 |
| 28.2 | 0.2 | 56.6 | 5 | 4.3 | 1.7528 | 0.6334 |
| 28.2 | 0.2 | 56.3 | 5 | 4.252 | 1.7505 | 0.6285 |
| 28.2 | 0.2 | 29.9 | 10 | 8.5 | 1.4771 | 0.9295 |
| 28.2 | 0.3 | 29 | 10 | 8.503 | 1.4756 | 0.9294 |
| 28.2 | 0.3 | 14.8 | 20 | 17.006 | 1.4623 | 0.9295 |
| 28.2 | 0.2 | 14.4 | 17 | 17 | 1.1702 | 1.2306 |
| 28.2 | 0.3 | 14.9 | 20 | 17.006 | 1.1583 | 1.2304 |
| 28.2 | 0.2 | 14.5 | 20 | 17.006 | 1.1731 | 1.2306 |
| 28.2 | 0.3 | 7.9 | 40 | 34.012 | 1.1613 | 1.2306 |
| 28.2 | 0.3 | 8.1 | 40 | 34 | 0.8976 | 1.5316 |
| 28.3 | 0.3 | 8 | 40 | 34.012 | 0.9084 | 1.5314 |
| 28.3 | 0.3 | 4.3 | 80 | 68.024 | 0.9030 | 1.5316 |
| 28.3 | 0.3 | 4.4 | 80 | 68.024 | 0.6334 | 1.8326 |
| 28.3 | 0.4 | 2.9 | 160 | 136.048 | 0.4623 | 2.1336 |
| 28.3 | 0.4 | 2.9 | 320 | 272.096 | 0.3617 | 2.4347 |
| 28.4 | 0.6 | 2.3 | 320 | 272.096 | 0.3617 | 2.4347 |
| 28.5 | 0.6 | 2.1 | 320 | 272.096 | 0.3617 | 2.4347 |
| 28.5 | 1.3 | 2.6 | 600 | 510.18 | 0.3617 | 2.7077 |

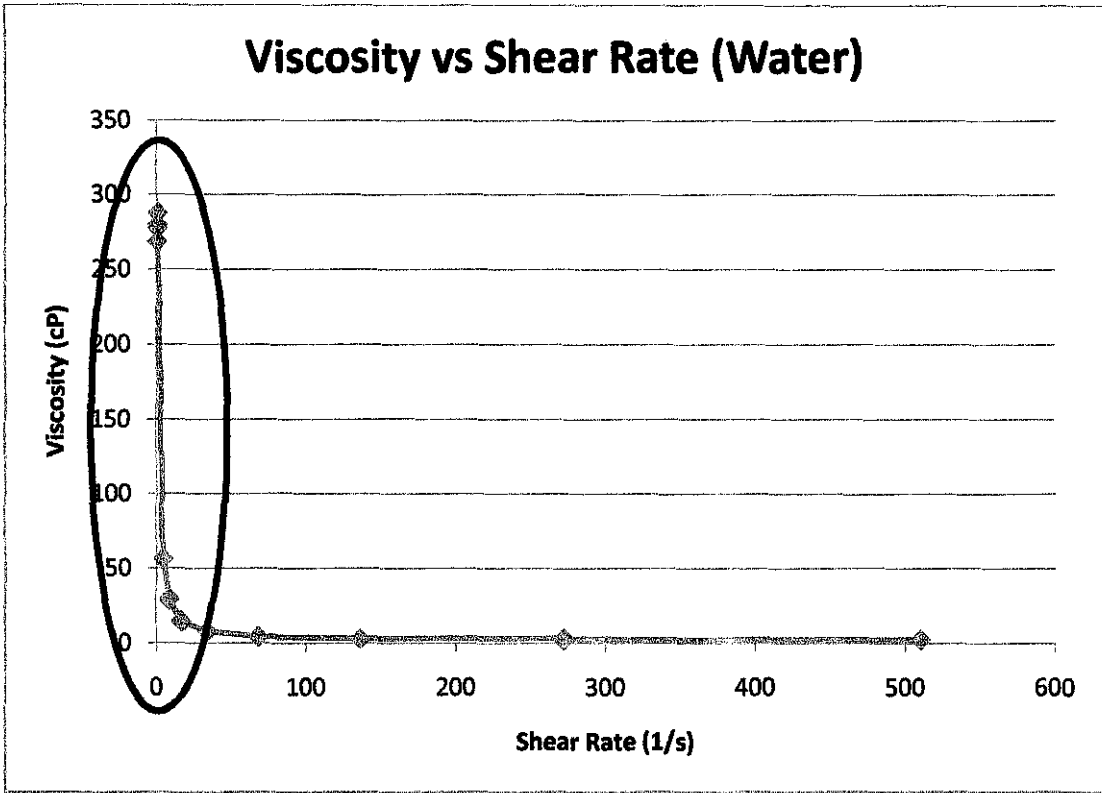


Figure 2: The Relationship of Viscosity and Shear Rate of Water

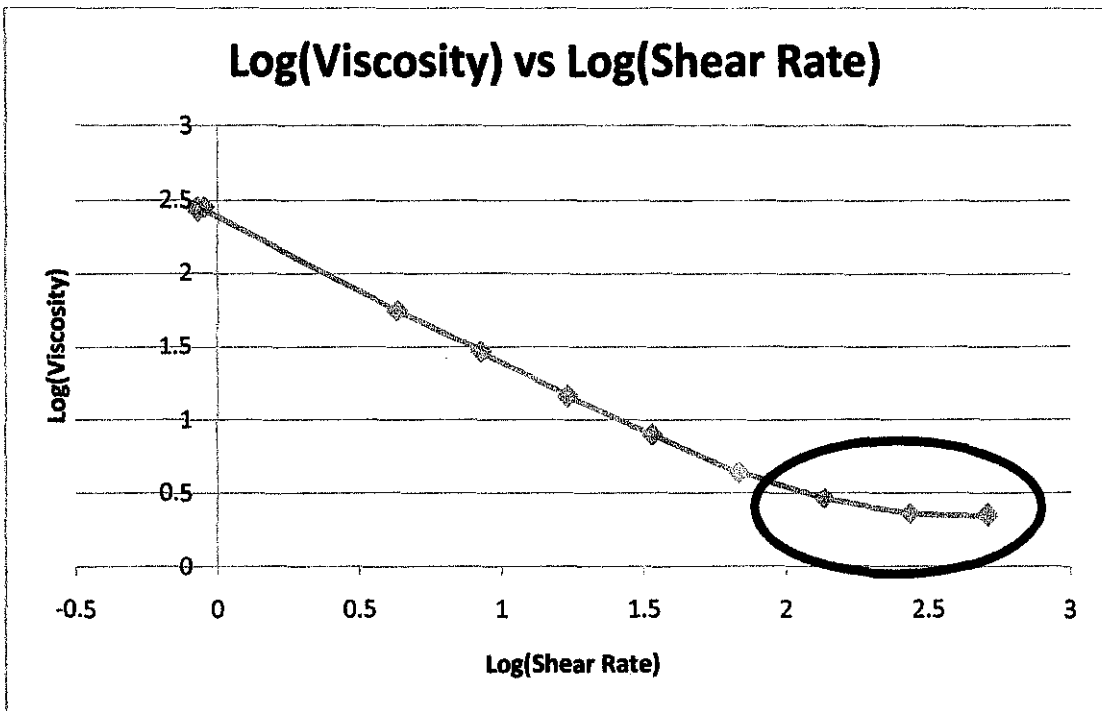


Figure 3: Log(Viscosity) vs Log(Shear Rate) of Water

The plot shows that the viscosity of water is a linear characteristic where it will remain constant till the end regardless of higher shear rate introduced and the stress in liquid will falls to zero immediately after the shearing is stopped. The first part of the plot in Figure 2 (ranges from 0-100 1/s) shows some high viscosity value of water. This is due to the first rotation of the spindle or in other word, the inertia of the spindle viscometer caused the reading to have high value.

The $\log(\text{Viscosity})$ vs $\log(\text{Shear rate})$ shows exactly the same explanation. The first part of the plot (ranges from 0-1.5) indicates the inertia of the viscometer take place. After that (in the highlighted region), the plots show constant viscosity indicates that it's the Newtonian fluid.

The experiment was done at ambient temperature (i.e. 28.0-28.6 °C) not at 25 °C because cooling the fluid may induce other variable in the experiment. The viscosity of water at 25 °C is around 0.890 cP. But in this experiment at ambient temperature (28.0-28.6 °C), the water viscosity is higher compared to the viscosity at 25 °C. This may due to the improper calibration of the viscometer. Although that is the case, the result still clearly justify that viscosity of water is dependant only on temperature but not on shear rate and time. This shows the behaviour of Newtonian liquids.

4.1.2 Viscosity Measurement with Crude Oil

The experiment with crude oil was done by varying the pre-shear rate and waiting time (time after pre-shear). The results show in the Table belows:

Result of Viscosity at Zero-Minute Waiting Time (0 minute)

Table 5: The Viscosity Result Pre-Shear at 1 RPM

| Temperature (°C) | Shear Stress (Pa) | Viscosity (cP) | RPM | Shear Rate (1/s) |
|------------------|-------------------|----------------|-----|------------------|
| 28.7 | 12.1 | 23.7 | 600 | 510.2 |
| 28.7 | 12.1 | 23.7 | 500 | 425.15 |
| 28.7 | 8.5 | 25 | 400 | 340.1 |
| 28.7 | 8.5 | 25 | 300 | 255.09 |
| 28.7 | 5.2 | 30.5 | 200 | 170.1 |
| 28.7 | 5.2 | 37.5 | 100 | 85.03 |

Table 6: The Viscosity Result Pre-Shear at 5 RPM

| Temperature (°C) | Shear Stress (Pa) | Viscosity (cP) | RPM | Shear Rate (1/s) |
|------------------|-------------------|----------------|-----|------------------|
| 28.7 | 11.6 | 22.7 | 600 | 510.18 |
| 28.7 | 9.8 | 23.1 | 500 | 425.1 |
| 28.7 | 8.2 | 24 | 400 | 340.12 |
| 28.7 | 6.4 | 25 | 300 | 255.1 |
| 28.7 | 4.8 | 28.3 | 200 | 170.06 |
| 28.7 | 3 | 35.7 | 100 | 85.03 |

Table 7: The Viscosity Result Pre-Shear at 10 RPM

| Temperature (°C) | Shear Stress (Pa) | Viscosity (cP) | RPM | Shear Rate (1/s) |
|------------------|-------------------|----------------|-----|------------------|
| 28.7 | 11.3 | 22.1 | 600 | 510.18 |
| 28.7 | 9.6 | 22.6 | 500 | 510.2 |
| 28.7 | 7.9 | 23.2 | 400 | 340.12 |
| 28.8 | 6.2 | 24.4 | 300 | 255.1 |
| 28.8 | 4.7 | 27.5 | 200 | 170.06 |
| 28.8 | 2.9 | 33.8 | 100 | 85 |

Result of Viscosity at 1-minute Waiting Time (1 minute)

Table 8: The Viscosity Result Pre-Shear at 1 RPM

| Temperature (°C) | Shear Stress (Pa) | Viscosity (cP) | RPM | Shear Rate (1/s) |
|------------------|-------------------|----------------|-----|------------------|
| 28.8 | 10.8 | 25 | 600 | 510.2 |
| 28.8 | 10.8 | 25.5 | 500 | 425.15 |
| 28.8 | 9 | 26.5 | 400 | 340.1 |
| 28.8 | 7 | 27.3 | 300 | 255.09 |
| 28.8 | 5.2 | 30.7 | 200 | 170.1 |
| 28.9 | 3.3 | 38.3 | 100 | 85.03 |

Table 9: The Viscosity Result Pre-Shear at 5 RPM

| Temperature (°C) | Shear Stress (Pa) | Viscosity (cP) | RPM | Shear Rate (1/s) |
|------------------|-------------------|----------------|-----|------------------|
| 28.9 | 12.2 | 23.8 | 600 | 510.2 |
| 28.9 | 10.2 | 24.1 | 500 | 425.15 |
| 28.9 | 8.2 | 24.2 | 400 | 340.1 |
| 28.9 | 6.6 | 25.7 | 300 | 255.09 |
| 28.9 | 4.9 | 29 | 200 | 170.1 |
| 28.9 | 3.1 | 36.5 | 100 | 85.03 |

Table 10: The Viscosity Result Pre-Shear at 10 RPM

| Temperature (°C) | Shear Stress (Pa) | Viscosity (cP) | RPM | Shear Rate (1/s) |
|------------------|-------------------|----------------|-----|------------------|
| 29 | 11.1 | 21.7 | 600 | 510.2 |
| 29 | 9.5 | 22.4 | 500 | 425.15 |
| 29 | 7.7 | 22.7 | 400 | 340.1 |
| 29 | 6.2 | 24.2 | 300 | 255.09 |
| 29 | 4.5 | 26.6 | 200 | 170.1 |
| 29 | 2.9 | 34.2 | 100 | 85.03 |

Result of Viscosity at 2-minute Waiting Time (2 minute)

Table 11: The Viscosity Result Pre-Shear at 1 RPM

| Temperature (°C) | Shear Stress (Pa) | Viscosity (cP) | RPM | Shear Rate (1/s) |
|------------------|-------------------|----------------|-----|------------------|
| 29.1 | 10.5 | 20.5 | 600 | 510.2 |
| 29.1 | 8.9 | 20.9 | 500 | 425.15 |
| 29.1 | 7.3 | 21.5 | 400 | 340.1 |
| 29.2 | 5.8 | 22.8 | 300 | 255.09 |
| 29.1 | 4.3 | 25.5 | 200 | 170.1 |
| 29.1 | 2.6 | 31.1 | 100 | 85.03 |

Table 12: The Viscosity Result Pre-Shear at 5 RPM

| Temperature (°C) | Shear Stress (Pa) | Viscosity (cP) | RPM | Shear Rate (1/s) |
|------------------|-------------------|----------------|-----|------------------|
| 29.2 | 10 | 19.6 | 600 | 510.2 |
| 29.2 | 8.5 | 19.9 | 500 | 425.15 |
| 29.3 | 7 | 20.5 | 400 | 340.1 |
| 29.3 | 5.6 | 22 | 300 | 255.09 |
| 29.3 | 4.1 | 24.1 | 200 | 170.1 |
| 29.3 | 2.5 | 29.8 | 100 | 85.03 |

Table 13: The Viscosity Result Pre-Shear at 10 RPM

| Temperature (°C) | Shear Stress (Pa) | Viscosity (cP) | RPM | Shear Rate (1/s) |
|------------------|-------------------|----------------|-----|------------------|
| 29.4 | 9.5 | 18.6 | 600 | 510.2 |
| 29.3 | 8.1 | 19 | 500 | 425.15 |
| 29.4 | 6.7 | 19.6 | 400 | 340.1 |
| 29.4 | 5.4 | 21.1 | 300 | 255.09 |
| 29.4 | 3.9 | 23.1 | 200 | 170.1 |
| 29.4 | 2.4 | 28.6 | 100 | 85.03 |

Result of Viscosity at 5-minute Waiting Time (5 minute)

Table 14: Viscosity Result Pre-Shear at 1 RPM

| Temperature (°C) | Shear Stress (Pa) | Viscosity (cP) | RPM | Shear Rate (1/s) |
|------------------|-------------------|----------------|-----|------------------|
| 29.5 | 9.1 | 17.8 | 600 | 510.2 |
| 29.5 | 7.7 | 18.2 | 500 | 425.15 |
| 29.5 | 6.4 | 18.9 | 400 | 340.1 |
| 29.5 | 5.2 | 20.3 | 300 | 255.09 |
| 29.5 | 3.7 | 22 | 200 | 170.1 |
| 29.5 | 2.4 | 27.8 | 100 | 85.03 |

Table 15: Viscosity Result Pre-Shear at 5 RPM

| Temperature (°C) | Shear Stress (Pa) | Viscosity (cP) | RPM | Shear Rate (1/s) |
|------------------|-------------------|----------------|-----|------------------|
| 29.6 | 8.7 | 17.1 | 600 | 510.2 |
| 29.6 | 7.4 | 17.5 | 500 | 425.15 |
| 29.5 | 6.2 | 18.3 | 400 | 340.1 |
| 29.5 | 5 | 19.7 | 300 | 255.09 |
| 29.6 | 3.6 | 21.3 | 200 | 170.1 |
| 29.6 | 2.3 | 26.9 | 100 | 85.03 |

Table 16: Viscosity Result Pre-Shear at 10 RPM

| Temperature (°C) | Shear Stress (Pa) | Viscosity (cP) | RPM | Shear Rate (1/s) |
|------------------|-------------------|----------------|-----|------------------|
| 29.6 | 8.5 | 16.6 | 600 | 510.18 |
| 29.6 | 7.2 | 17 | 500 | 425.1 |
| 29.6 | 6.1 | 17.8 | 400 | 340.12 |
| 29.6 | 4.9 | 19.1 | 300 | 255.1 |
| 29.6 | 3.5 | 20.8 | 200 | 170.06 |
| 29.6 | 2.3 | 26.5 | 100 | 85.03 |

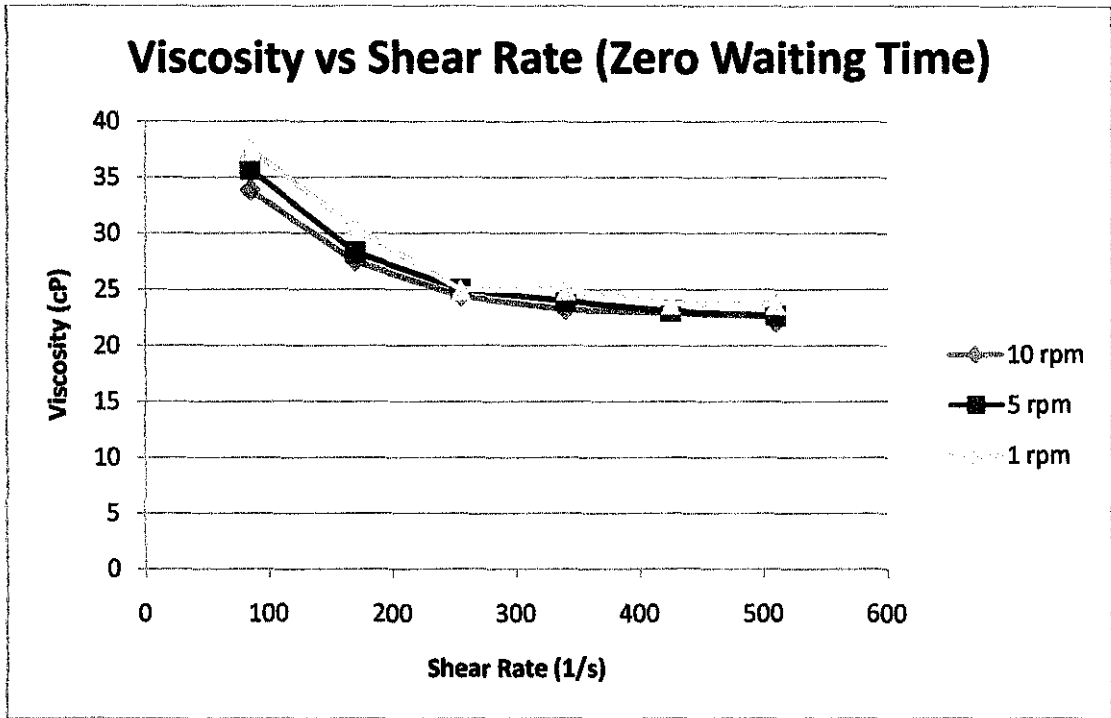


Figure 4: The Relationship of Viscosity and Shear Rate of Crude Oil with Zero Waiting Time

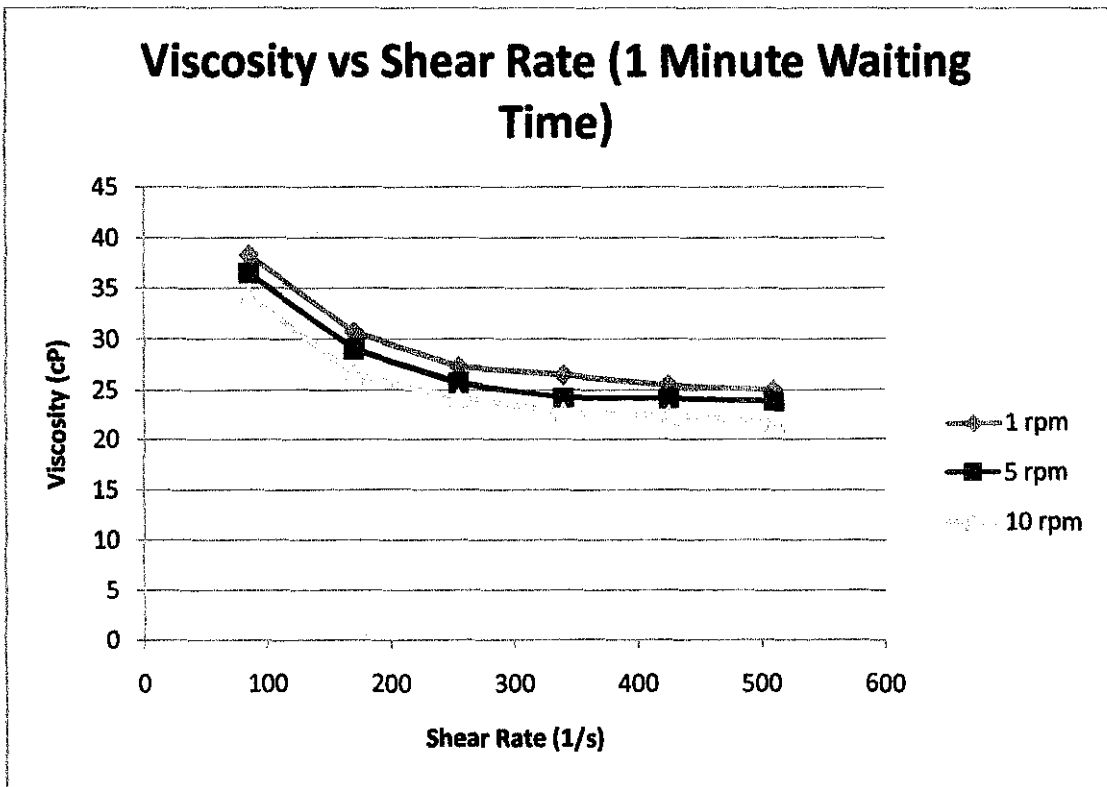


Figure 5: Relationship of Viscosity and Shear Rate of Crude Oil with 1 Minute Waiting Time

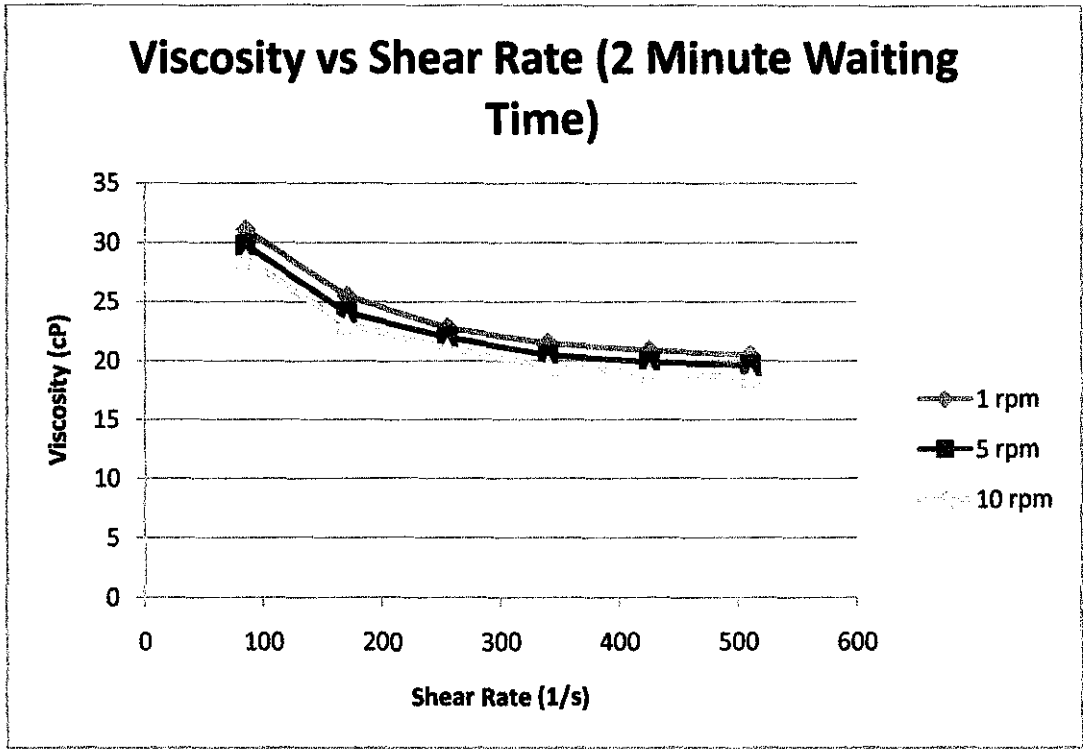


Figure 6: Relationship of Viscosity and Shear Rate of Crude Oil with 2 Minute Waiting Time

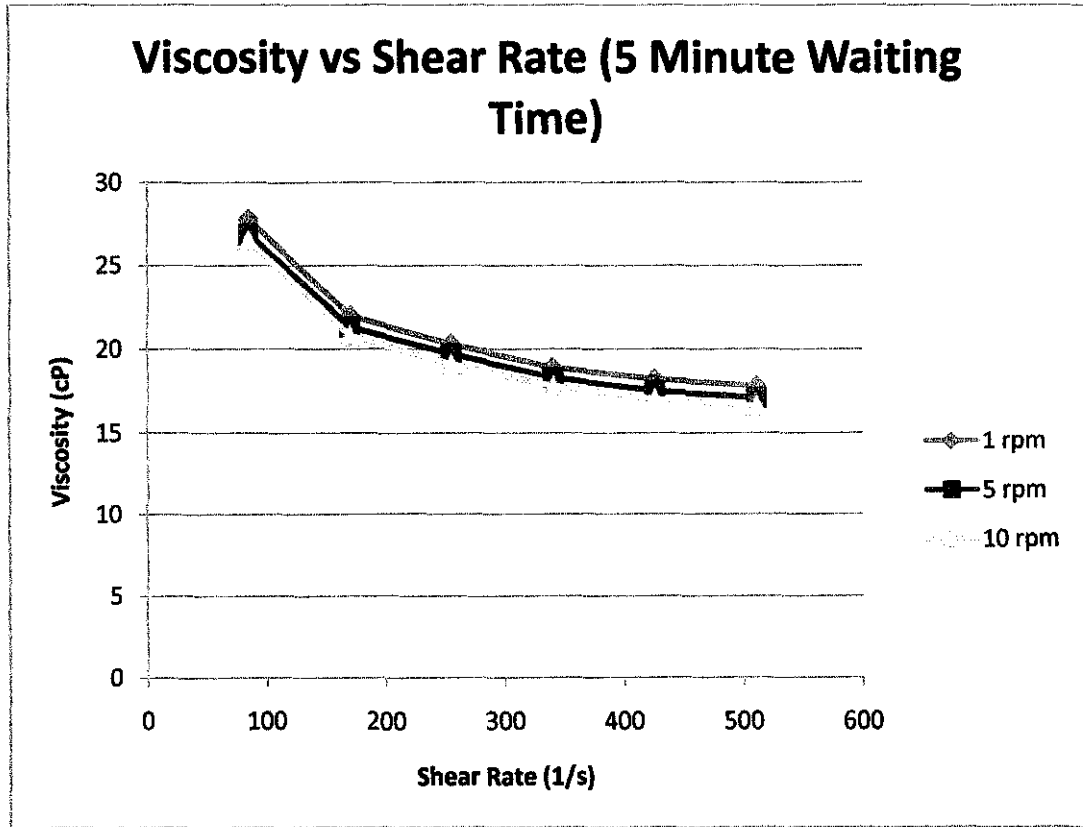


Figure 7: Relationship of Viscosity and Shear Rate of Crude Oil with 5 Minute Waiting Time

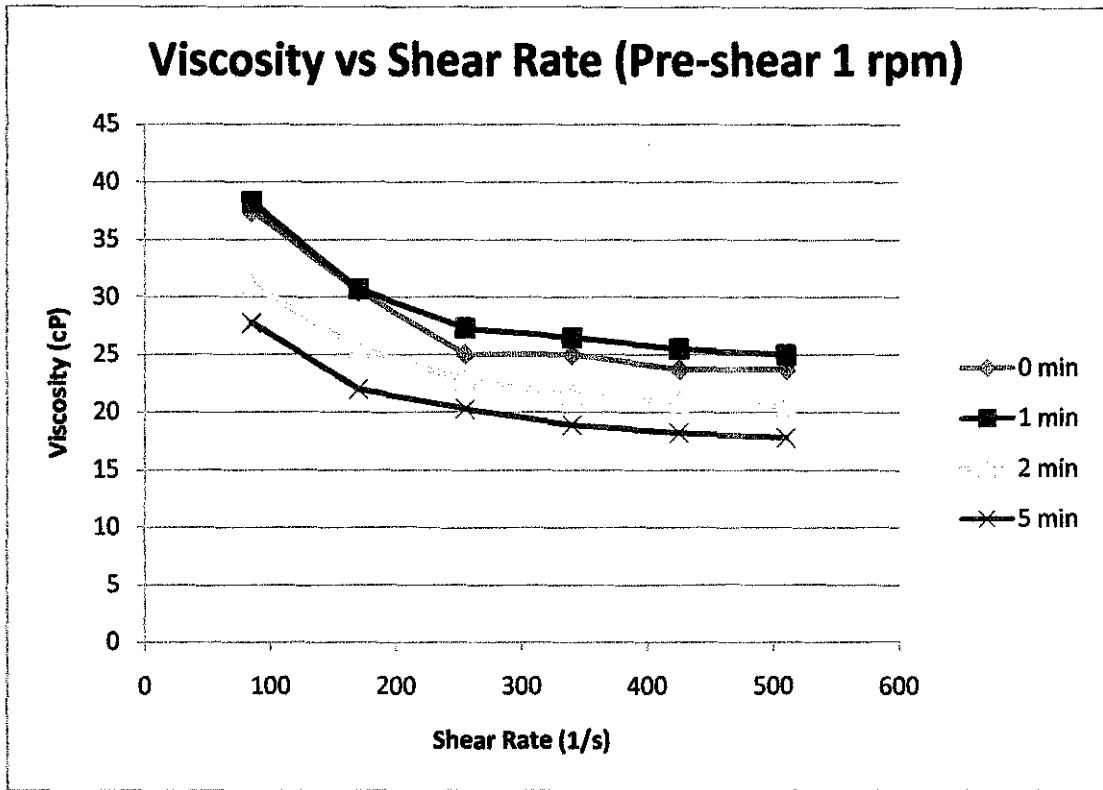


Figure 8: Relationship of Viscosity and Shear Rate of Crude Oil with 1 RPM Pre-Shear

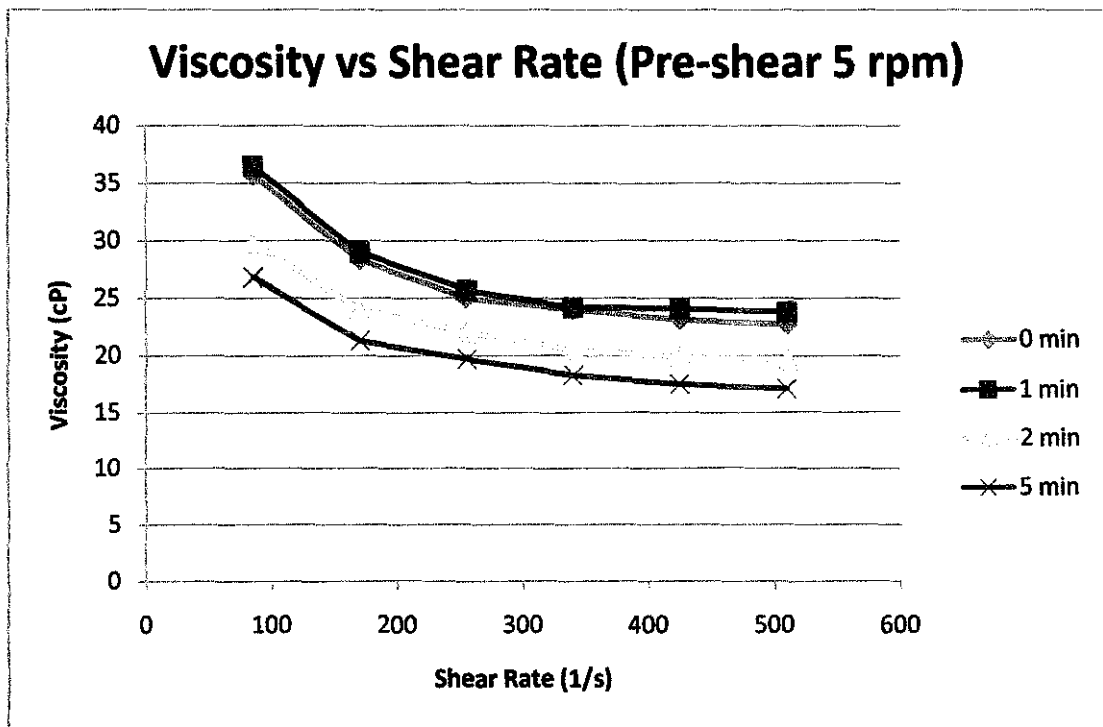


Figure 9: Relationship of Viscosity and Shear Rate of Crude Oil with 5 RPM Pre-Shear

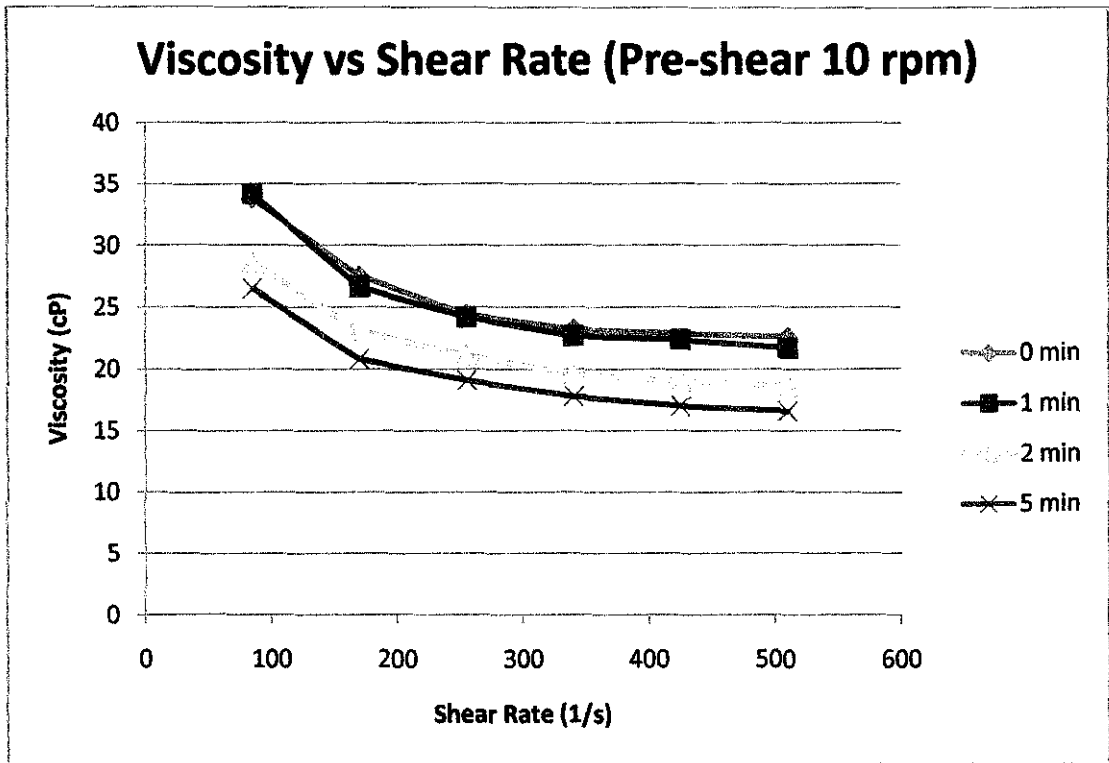


Figure 10: Relationship of Viscosity and Shear Rate of Crude Oil with 10 RPM Pre-Shear

4.2 Discussion

4.2.1 Effect of Pre-Shear

Figure 4, 5, 6 and 7 shows the effect of pre-shear on the viscosity of crude oil with respect to shear rate. The effect of 3 different pre-shear values 1, 5 and 10 rpm were studied while keeping the composition and shear rate the same. As can be seen from the plot, a lower pre-shear value (1 rpm) results in higher viscosity trend compared to the other pre-shear value (i.e. 5 rpm and 10 rpm). This illustrates that pre-shear before measurement will vary the viscosity of the crude oil. That is because the fluid structure (i.e. waxy structure) of the crude oil is broken down and because of that the viscosity of the crude oil will decrease.

This effect of pre-shear also can be illustrates from a bottle containing ketchup sauce. When you want to force out the ketchup from the bottle, you shake the bottle putting up some inertia to the bottle. That is pre-shear in this case. Because of that pre-shear, the structure of the ketchup is broken down and at the same time decreasing the viscosity so that it can flow out of the bottle. This shows that ketchup sauce is a Non-Newtonian fluid like crude oil.

Another interesting point from the plots is that, at higher shear rate, the viscosity is not as sensitive to a change in shear rate as it is at lower shear rates. This shows from all the plots above. The low sensitivity changes of viscosity in the results maybe because of the fluid reaches their equilibrium-viscosity where as more shear rate is introduced, the changes will be small.

4.2.2 Effect of Waiting Time

The effect of waiting on the viscosity is shown in the plots. From Figure 5, the plot shows that between all the pre-shear rate values, there's a wide gap between the viscosity trend showing that the 10 rpm pre-shear value at the lowest viscosity and vice versa. Moving on to Figure 6 and Figure 7, the gap between the viscosity trends becomes narrower. This clearly shows the effect of waiting time on the viscosity of crude oils.

As the crude oil is pre-sheared, the structure of the fluid (i.e wax structure) is broken down and results in decreasing of viscosity. Crude oil is a time-dependent fluid. When the waiting time variables are introduced, this gives time to the structure to recover to the original wax structure. This clearly shows on Figure 7, where the viscosity trend between all the pre-shear values is close together. The 5-minutes waiting time gives the crude oil to recover from its pre-shearing that break their wax structure.

The result of varying the waiting time and pre-shear value shows that the crude oils are time-dependant and thixotropic. As with increasing in shear rate, the apparent viscosity decreased. Although there's no direct plots or data that shows that the crude oils decrease in viscosity over a period of time under constant shear rate, from all the plots above we can still see that the crude oil exhibits a shear-thinning characteristic with respect to time and shear rate.

4.2.3 Effect of Temperature

The effect of temperature was not studied directly in this experiment. Experiments were done at ambient temperature. As the experiments continue, the temperature increase with respect to time and shear rate. The data from Table 5-16, shows that the temperature increases from 28.7 -29.6 °C. The viscosity from the first experiment till the last experiment shows decreasing in viscosity with respect to temperature since the experiments were done continuously.

CHAPTER 4

CONCLUSION AND RECOMMENDATION

4.1 Conclusion

Rheological characterization of waxy crude oils is necessary to be able to understand and predict their behavior to avoid many problems during production, transportation and processing, such as clogging of pipelines. In this work, the characteristic of Dulang waxy crude was investigated.

The result of viscosity at different pre-shear value shows that giving the pre-shearing to the waxy crude affect the viscosity of the crude oil. At higher pre-shearing value, the viscosity is lower and vice versa. Furthermore, the effect of waiting time (rest time after pre-shear) to the viscosity shows that crude oil is a thixotropic fluid which is time-dependent and this affects the result of the crude oil viscosity that is pre-sheared.

As a conclusion, this researches and study regarding rheology, viscometer and rheometert have greatly improved understanding and knowledge related to the non – Newtonian fluid concept. The understanding regarding the rheological behavior of crude oils can be applied to solve various problems as stated in objective.

4.2 Recommendation

1. The rheometer should be calibrated first to prevent errors during measurement.
2. The experiment should be done with new sample and part by part for each type of experiment due to the shear history. If running each experiment with new sample, the shear history won't be an issue and the result might become more accurate.
3. The temperature when running the experiment should be kept constant because as well known, crude oil is a temperature-dependent fluid. So, the slight changes of temperature might alter the result to be useless.
4. The experiment to study the thixotropy of the crude oil should be done separately, so that the effect can be clearly shown (i.e. measure the viscosity under constant shear rate over period of time).
5. The experiment can be done with varying other variable (such as pressure) to see the effect on the viscosity.

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APPENDICES

Preparing for a test

1. Fill the sample cup with the proper amount of fluid based on the bob you are using. Refer to the chart below.

Table 17: Proper Amount of Fluid

| Bob Type Confirm | B1 | B2 | B3 | B4 | B5 | NB1 | NB2 | NB5 |
|-------------------------------------|----|----|----|-----|----|-----|-----|-----|
| Sample Amount in mL (R1 Sample Cup) | 42 | 78 | 96 | 104 | 52 | 32 | 73 | 44 |

2. Hold the sample cup by hand and position the bob in the center. Push the sample cup up past the o-ring. Hold the sample cup in place and screw the sample cup nut into place.
3. Position the heat bath under the sample cup. Then raise it using the “Heater Lift / Lower” switch.
4. To pressurize the sample, gradually rotate the regulator knob clockwise. To reduce pressure, gradually rotate the regulator knob counter-clockwise at a rate no greater than 60 PSI per minute.

Pressure is only necessary for tests temperatures above 200°F (95°C). The heaters will be deactivated if the sample is not pressurized enough to prevent boiling. The ORCADA™ software will indicate this with a yellow alarm light. The alarm light will turn green when the appropriate pressure is applied to the sample. Refer to the chart below for the minimum pressure requirements.

5. Once the pressure is set and the heater is in place, the Model 1100 is ready to run a test.

Table 18: Minimum Pressure Requirement

| Temperature | Pressure |
|------------------------------------|---------------------|
| Ambient - 200°F (Ambient - 93.3°C) | 0 PSI (0 kPa) |
| 201° - 295°F (93.9° - 146.1°C) | 100 PSI (690 kPa) |
| 296° - 355°F (146.7° - 179.6°C) | 200 PSI (1,380 kPa) |
| 356° - 395°F (180.1° - 201.8°C) | 300 PSI (2,070 kPa) |
| 396° - 445°F (202.4° - 229.6°C) | 500 PSI (3,450 kPa) |
| 446° - 500°F (230.2° - 260.2°C) | 800 PSI (5,520 kPa) |