Study on Asphaltene Precipitation Using Thin Film Micro-reactor.

By

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Dissertation submitted in partial fulfilment of the requirements for the
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CERTIFICATION OF APPROVAL

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APPROVED BY,

....

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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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MUHAMAD NABIL AZMI BIN MUSA

ABSTRACT

Asphaltenes are present in most petroleum materials and in all heavy oils and bitumen from oil sands. Asphaltenes are the heaviest and most complex molecules in crude oil and are defined by its solubility class as the constituents of oil which are soluble in toluene but insoluble in n-heptane. Asphaltene precipitation in the petroleum industry is detrimental to production operations since oil flow rate is impaired due to asphaltene precipitation or deposition. To study the effect of temperatures on asphaltene precipitation, a thin film micro-reactor is used to carry out the experiment. The effect of bulk temperature on asphaltene precipitation on heat transfer surface is studied and the result is observed and recorded. In this paper, four types of crude oils were tested to see the asphaltene precipitation.

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CHAPTER 1.0:

INTRODUCTION

1.1 Background Studies

Asphaltenes are present in most petroleum materials and in all heavy oils and bitumen from oil sands. Asphaltenes are the heaviest and most complex molecules in crude oil and are defined by its solubility class as the constituents of oil which are soluble in toluene but insoluble in n-heptane (Chandio, M, & Mukhtar). According to (Liao, Zhao, Creux, & Yang, 2009) asphaltenes are a class of complex mixtures in petroleum fluids, which are well-defined as the fraction in petroleum oils soluble in aromatic solvents, such as toluene, whereas insoluble in saturated hydrocarbons, such as n-heptane, and asphaltenes are prepared from crude oils by this simple principle.

Zendehboudi et al., 2014, said that asphaltene precipitation in the petroleum industry is detrimental to production operations since oil flow rate is impaired due to asphaltene precipitation or deposition. Furthermore, one of the major problems that heavy oil and offshore production face is the deposition of heavy organic solids such as asphaltenes in the reservoir and production facilities (Arsalan, Palayangoda, & Nguyen, 2014). A reduction in the well productivity and significant damage to the units and instruments used along the pipelines is caused by asphaltenes precipitation and deposition.

Luo, Wang, & Gu, 2010, found that the yield and properties of asphaltenes precipitated from a crude oil depends on various factors. Thermodynamic models to understand the asphaltene precipitation fall under two molecular thermodynamic frameworks, mirroring two prevalent schools of thought regarding how asphaltenes are stabilized in crude oil (Tavakkoli, Panuganti, Taghikhani, Pishvaie, & Chapman, 2014).

There are various findings in the literature dealing with the characterization of asphaltenes precipitated from products of hydro treating of a wide variety of crude oils, having also a wide range of physical and chemical properties (Leyva, Ancheyta, &

Centeno, 2014). In this report, the author will focus on the effects of certain factors on the asphaltene precipitation.

1.2 Problem Statement

Asphaltene precipitation is considered as an undesired process during oil production via natural depletion and gas injection as it blocks the pore space and reduces the oil flow rate. Moreover, it lessens the efficiency of the gas injection into oil reservoirs (Zendehboudi et al., 2014). Asphaltenes can cause problems at almost any stage of oil production, transportation or even refining (Buckley, 2012).

Asphaltenes deposition can lead to devastating economic consequences due to the exorbitant cost of remediation and subsequent abandonment of the well (Arsalan et al., 2014). Severe asphaltene aggregation problems are faced in both upstream and downstream productions operations (Hassanvand, Shahsavani, & Anooshe, 2012). Thus it is best to understand the parameters governing the precipitation and deposition of asphaltenes in the wellbore in order to anticipate the risks and design accordingly.

1.3 Objectives

The objectives of this study are:

i) To study the effect on temperature on asphaltene precipitation.

ii) To study the asphaltene precipitation on different types of crude oil.

1.4 Scopes of Study

To achieve the objective of this study, the author will use custom designed thin film micro-reactor. The cross sectional view of the micro-reactor is shown in Figure 1. The base of the equipment is made of thick plate of copper with a nicrome heating wires embedded. The top of the micro-reactor is made of a thin sheet of glass with a provision for injecting the crude oil to the interface of the metal plate and the glass. As the metal plate is heated, the crude oil film in the interface also gets heated up (Shetty, Ramasamy, Pendyala, 2014). The equipment will be connected to a microscope and computer to record the data while conducting the experiment.

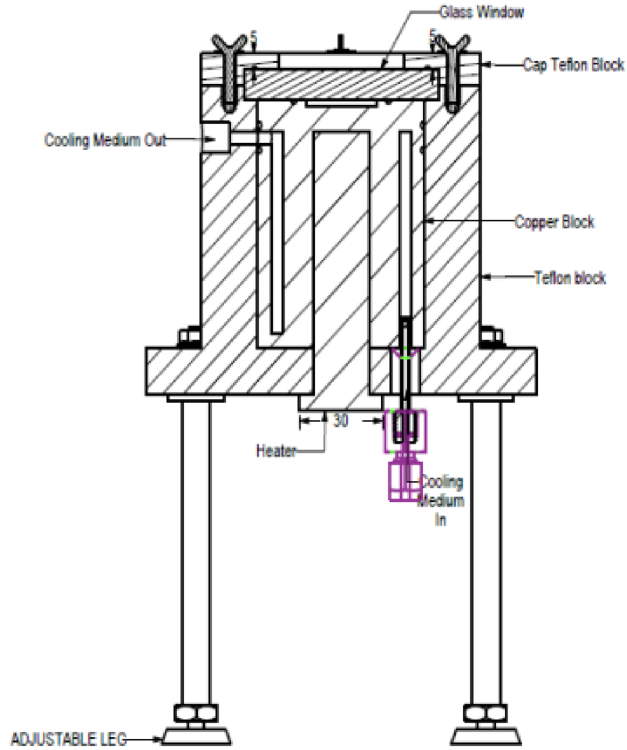


Figure 1: Cross-sectional view of thin film micro-reactor

Figure 2: Thin film micro-reactor

CHAPTER 2.0:

LITERATURE REVIEW

With the depletion of the conventional crude oil resources, the oil industry has begun to explore and exploit abundant unconventional heavy oil reserves (Luo et al., 2010). Luo et al. (2010) also stated that recently, solvent-based heavy oil recovery processes have gained increasing attention because of their cost-effectiveness and environmental friendliness. More than 10 wt. % of asphaltene content is usually presents in heavy oil which has a dramatic effect on the heavy oil physicochemical properties.

2.1 Definition of Asphaltenes

There are many literature that defined asphaltenes. Asphaltene is defined as association of aggregates with 2 to 6 molecules per aggregate (Zendehboudi et al., 2014). Asphaltenes are polydisperse mixture of the heaviest and most polarizable fraction of the crude oil and are defined according to their solubility properties as being soluble in aromatic solvents, but insoluble in light paraffin solvents (Tavakkoli et al., 2014). Meanwhile, Chandio et al. (2014) stated that asphaltene are the heaviest and most complex molecules in crude oil and are defined by its solubility class as the constituents of oil which are soluble in toluene but insoluble in *n*-heptane. Asphaltenes are anticipated as a group of complex compounds, which are highly polydispersed and cannot be absolutely prescribed by this simple principle (Liao et al., 2009). Asphaltenes are the heaviest and most complex fraction in a crude oil sample, which appear as brown or black solid particles precipitated from a crude oil by using low boiling-point alkane such as *n*-pentane or *n*-heptane (Luo et al., 2010). Due to the definition of asphaltene in terms of their insolubility rather than their chemical structures, this definition gives a broad distribution of molecular structures of asphaltenes that are precipitated from different crude oil samples (Luo et al., 2010).

2.2 Problems Caused by Asphaltenes in Petroleum Industry

Occurrence of asphaltene deposition in producing formation constitutes one of the most serious problems currently encountered in the petroleum industry in many areas of the world (Hassanvand et al., 2012). Asphaltenes can cause problems at almost any stage of oil production, transportation, or refining process (Buckley, 2012). Asphaltenes deposits in the reservoir, flow lines, refining equipment and storage tanks can drastically affect petroleum production (Garreto, Mansur, & Lucas, 2013). It is popular that asphaltene precipitation from the heavy oil due to compositional, pressure, and temperature changes can be advantageous and/or problematic in heavy oil production, transportation, and ultimate refining processes. In deep offshore wells, avoiding instability may be impossible, and solutions whether designed into wells and platforms or provided after a problem occurs are likely to be extremely expensive (Buckley, 2012). Deposit formation in crude oil processing can be attributed to various factors but asphaltenes are generally considered as main precursors of deposit formation on heat transfer surfaces in crude oil processing (Chandio et al.).

Asphaltenes are well-known for their tendency to precipitate and deposit during oil production because of changes in pressure, temperature, and composition (Tavakkoli et al., 2014). The presence of asphaltene on heat transfer surfaces further favours fouling of equipment because asphaltenes are more prone to attach to already coked surfaces than a clean metallic alloy surfaces (Chandio et al.). It is inside the asphaltene molecule where most of the metals particularly nickel and vanadium, as well as other impurities such as sulphur and nitrogen is concentrated (Leyva et al., 2014). Thus, any changes in the asphaltenes structure will impact directly on the properties of the upgraded oil, such as reduction of the contents of sulphur, nitrogen and asphaltenes, increase in the API gravity and in the distillate content, and diminution of the viscosity and residue fraction content. The average molecular weight is not necessarily a good parameter to characterize asphaltenes, simply because asphaltene are defined through their solubility in aliphatic hydrocarbons (Liao et al., 2009).

2.3 Characteristics of Asphaltene

Progress in understanding asphaltene phase behavior and in predicting asphaltene problems have been slow, mainly because of the chemical complexity of crude oils' heavy ends (Buckley, 2012). Different analytical and spectroscopic methodologies have been reported to elucidate about the possible relationships among the structural parameters of the asphaltenes and the transformation they suffer during hydro processing of heavy petroleum (Leyva et al., 2014). Flocculation of asphaltenes takes place when the solvent added changes the crude oil's composition, and as a consequence changes the solubility parameter of the petroleum media that disperses the asphaltenes (Garreto et al., 2013). From the macromolecular point of view, the associated asphaltene molecules are assumed to be independent like resins and other oil components (Zendehboudi et al., 2014). It was believed that asphaltenes exist as colloids which are stabilized by the presence of surrounding resins although the evidence of this statement is still mixed (Arsalan et al., 2014). Nonetheless, Zendehboudi et al. (2014) stated that precise prediction and effective mitigation of solid deposition during oil production processes remain challenging due to the complexity of the solids compound.

The solubility parameters of the petroleum and the asphaltene fraction are the properties most widely applied in theoretical work to describe asphaltene precipitation (Garreto et al., 2013). Commonly, asphaltene as polar part of the oil in the form of a complex molecular structure contains aromatics chains within its core and naphthenic part surrounds the core and this complex component usually has a fairly high molecular weight (Zendehboudi et al., 2014). These complex components containing some heteroatoms (Nitrogen, Sulphur, and Oxygen) and trace metals (Nickel, Vanadium, Iron, etc.) (Arsalan et al., 2014). Furthermore, there are fewer large aggregates and narrower distributions once asphaltenes are in infinite-diluted systems of higher temperatures and better solvents; however they still exhibit a real polydispersity. It is popular that models system of asphaltene dispersed in solvents does not satisfactorily reproduce the behavior of asphaltene phases in crude oil.

In order to alleviate problems caused by asphaltenes, a thorough understanding of the process of asphaltene precipitation, flocculation and deposition and the factors affecting them is essential (Chandio et al.). It has been found that the yield and properties of asphaltenes precipitated from a crude oil depends on various factors, such as the precipitant, liquid precipitant-to-oil volume ratio, contact time, temperature and pressure (Luo et al., 2010). Moreover, Luo et al. (2010) also found that, some standard precipitation methods have been developed in laboratory to obtain accurate and consistent asphaltene content. In the event of a change in thermodynamic stability of asphaltenes due to an external factor, asphaltenes start precipitating out of the solution hence begin to flocculate and increase in size and eventually adhere to the surrounding surface such as pipe wall, porous network, asphaltene covered surface (Arsalan et al., 2014). The most efficient parameters contributing to asphaltene precipitation include pressure reduction and the solvent injection during oil production and enhanced oil recovery (EOR) processes (Zendehboudi et al., 2014).

Whether a change in temperature dissolves asphaltenes in oil or it favours precipitation of asphaltenes exist in crude oil in dispersed state forming a colloidal suspension of oil or they exist in dissolved state in oil like true solution, are still debatable (Chandio et al.). Asphaltenes destabilized and start to precipitate when the pressure, temperature and/or composition changes occur during primary production (Hassanvand et al., 2012).

CHAPTER 3.0:

METHODOLOGY

3.1 Research Methodology

The objective of this study is to study the effects of temperature on asphaltene precipitation and deposition in various samples of crude oil. This study is based on experimental to study the change of temperatures and their effect on asphaltene precipitation. The experiment will be manipulated the temperature to observe the effects. Every result will be recorded and compared in order to get the point where asphaltene start to precipitate in the crude oil samples. A telescope will be used to observe the fine particle of the asphaltene that will start to precipitate in the crude oil sample.

3.2 Experimental Procedures

The experiment will be conducted using five (5) different types crude oil that is Bintulu, low sulfur waxy residue (LSWR), Al Shaheen, Pyrenees, Arab Heavy. The properties for each of the crude oils are shown in the table below.

Table 1: Properties of Crude Oils

Properties	Crude Oil				
	Bintulu	LSWR	Al Shaheen	Pyrenees	Arab Heavy
API Gravity @ 60°F (°API)	28.21	25.27	28.3	18.7	27.67
Density @ 15°C (g/ml)	0.789	0.9	0.8868	0.9415	0.887
Kinematic Viscosity (cST) @ 15°C	5.2	446	44	64.81	83.7
Asphaltenes (%)	0.5	0.28	1.7	0.2	7
Pour Point (°C)	-6	45.61	-45	-27	-45
Flash Point (°C)	11.2	156.06	-5	75	6.5

The experiment will be conducted by using the thin film micro-reactor and a PID controller will be used to control the temperature to heat up the micro-reactor. A few drops of crude oil are sufficient enough to form a thin film on the copper plate and covered by the glass plate. The film is slowly heated and brought to the required temperature which is specified at the PID controller. The temperature is increased from time to time until the fouling precursors can be observed via the microscope video recording unit. Once the asphaltene precipitation has been observed, the temperature is kept constant for a period of time. To verify the stability of the formed asphaltene, additional crude oil of the same type was introduced to the existing film. The observation will help the author to know the behavior of asphaltenes.

<u>Operating Procedure</u>

1. The thin film micro-reactor is cleaned with toluene as toluene can dissolve the crude oil to ease the cleaning process.
2. A microscopic recording device is set up to record the observation during experiment.
3. A few drops of crude oil is put to the thin film uniformly for the best result.
4. The PID temperature controller is switched on and set the maximum value to 160°C.
5. Record the any changes in the crude oil particle and observe the experiment for minimum 3 hours.

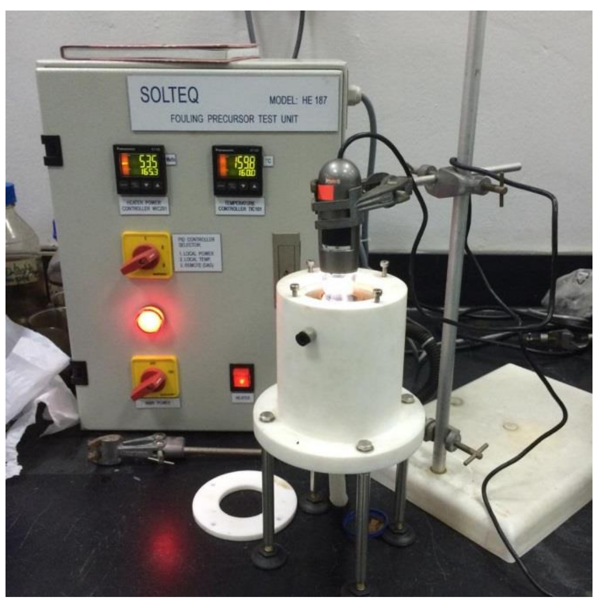


Figure 3: The setup of the experiment

3.3 Gantt chart and Key Milestones

Here is the Gant chart for this Final Year Project I and Final Year Project II. This research is conducted according to the Gant chart provided.

Table 2: Gantt chart and Key Milestones for FYP I

No.	Detail/Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	Selection of Project Topic														
2	Preliminary Research Work														
3	Submission of Extended Proposal														
4	Proposal Defense														
5	Project Work Continues														
6	Submission of Interim Draft Report														
7	Submission of Interim Report														

Table 3: Gantt chart and Key Milestones for FYP II

No.	Detail/Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	Project Work Continues														
2	Submission of Progress Report														
3	Pre-SEDEX														
4	Submission of Draft Final Report														
5	Submission of Dissertation														
6	Submission of Technical Paper														
7	Viva														
8	Submission of Project Dissertation														

CHAPTER 4.0:

RESULTS AND DISCUSSION

4.1 Past Study Result and Discussion

In the past study, two different crude oils were used to study the formation of fouling precursors on the metallic surface of the micro-reactor at 160°C. The properties of the crude oil used in this study are shown in the table.

Properties	Density @ 15°C	API Gravity @ 60°F	Kinematic Viscosity	Pour Point	Flash Point	Sulphur Content
Units	kg/m^3	-	cSt	(°C)	(°C)	-
Crude Oil A	840.0	28.21	5.2	-6	11.2	Low
Crude Oil B	890.0	27.67	83.7	-45	6.5	High

Table 4: Properties of Crude Oil

It is observed during the experiment with crude oil A that the particles grew in size slowly with time and remained constant around 300 minutes. Once the particles reached the maximum size, the surface is cooled by injecting compressed air through the copper block. It is observed that the size of particles slightly increased when the temperature was below 55°C. The micro-photographs of the asphaltene precipitation is shown in the Figure 3.

In the second run two drops of crude oil A was added to the existing film and the surface was brought to 160°C. More particles formed at different parts of the surface and the particles size increased slowly with time and remained constant around 300 minutes. While cooling there was no increase in the number of particles but the particles size slowly increased when the temperature went below 55°C. The same behavior of the fouling precursors was observed when the experiment was continued with repeated additions of crude oil A.

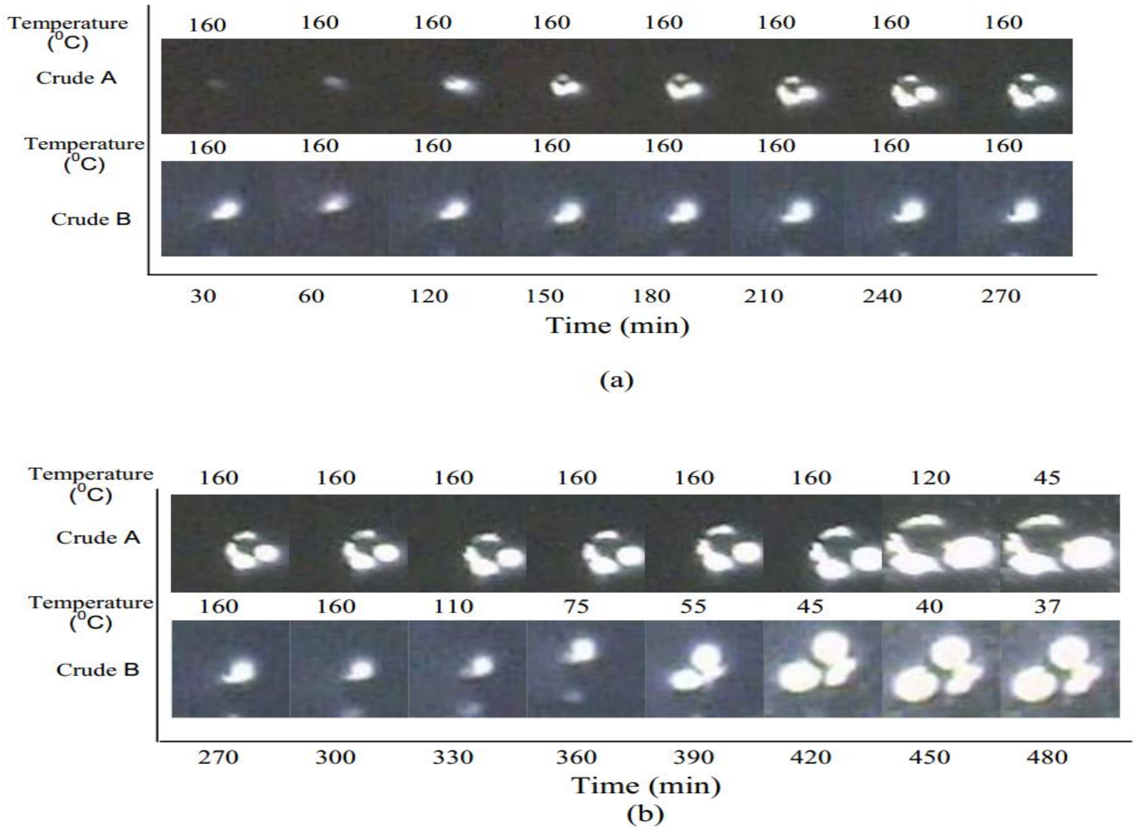


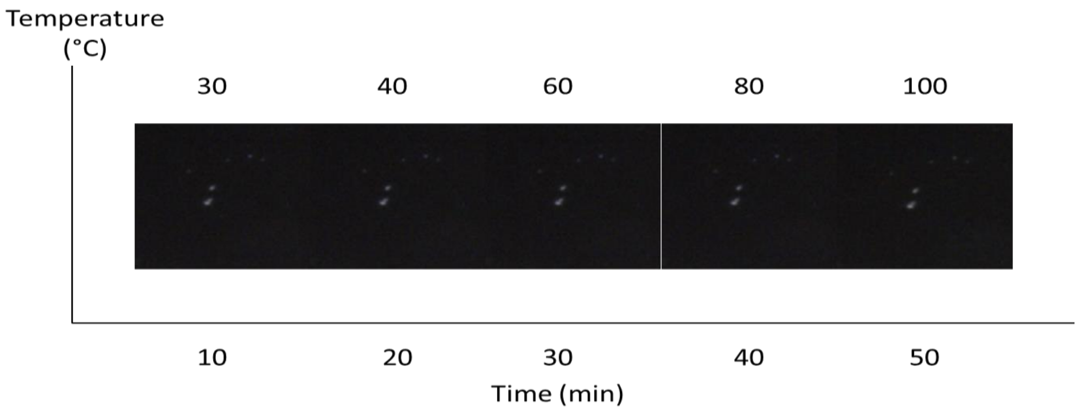
Figure 4: Micro-photographs of the film of the crude oils A and B with time and temperature

Almost similar observations were made when the tests were conducted for crude oil B. In the first run the fouling precursors were observed at 160°C, the particles slowly grew in size with time and remained constant around 300 minutes. While cooling the surface from 160°C to room temperature the particle size remained constant at higher temperatures and the size of the particle increased when the temperature was below 55°C. The particle size tends to increase till the surface temperature reached the room temperature.

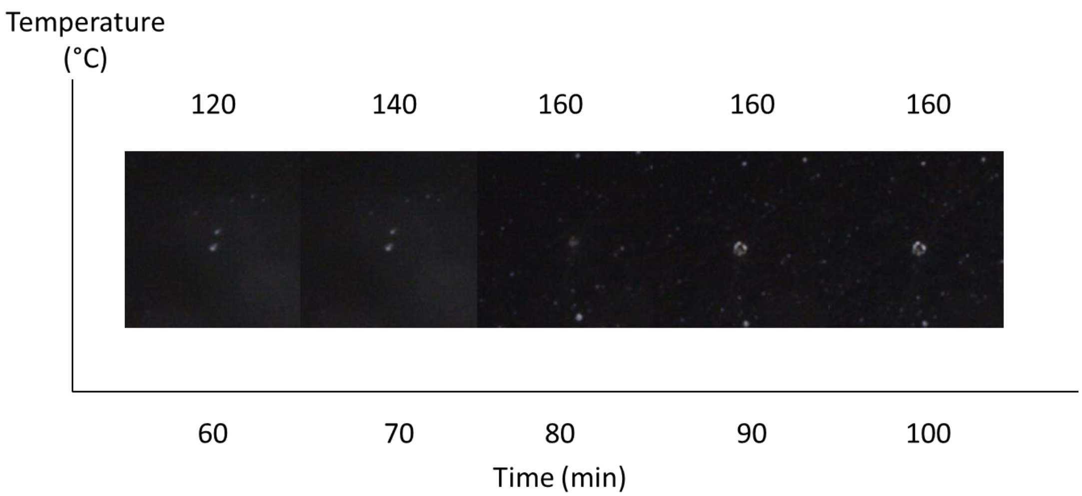
4.2 Experimental Results

Result for Arab Heavy Crude oil.

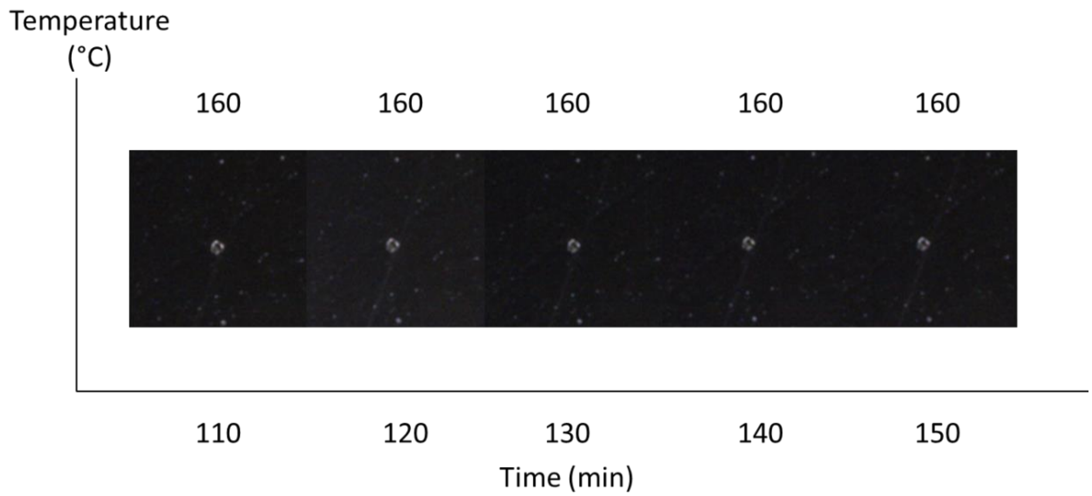
The first crude oil that been tested is the Arab Heavy.



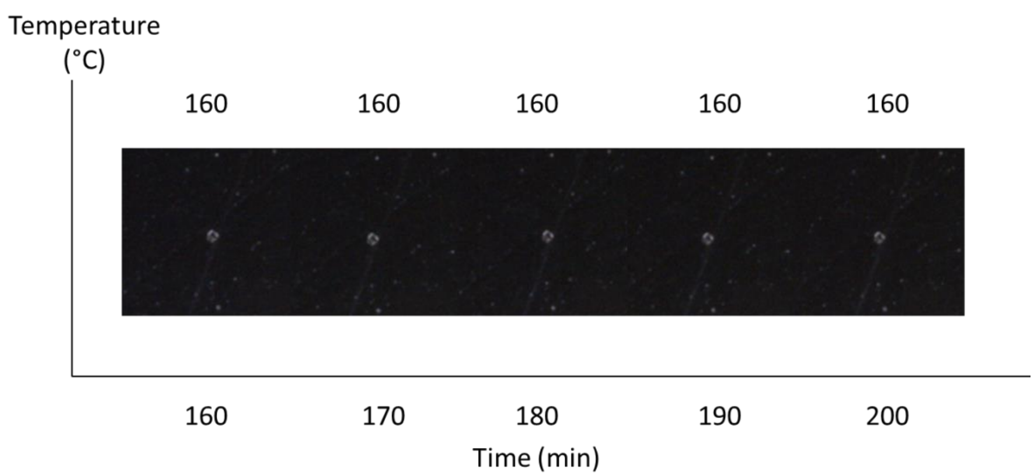
In the first 50 minutes of the experiment, the crude oil was heated from 30°C to 100°C. During this phase of heating, there are no obvious changes that happen in the crude oil.



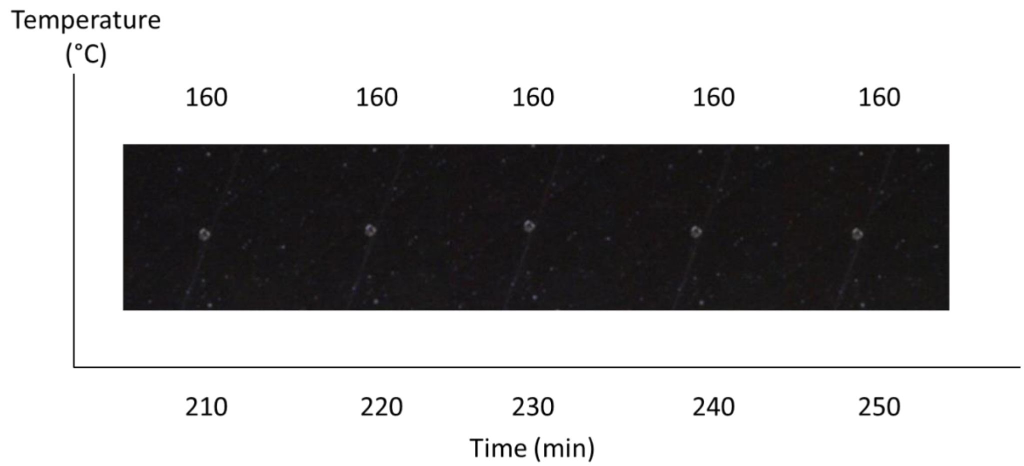
In this graph, the asphaltene particle size decreased when the temperature change from 140°C to 160°C. When the temperature of the crude oil reaches 160°C, it was kept constant for 3 hours and the observation was done every 10 minutes. At the first 10 minutes after reaching 160°C, the asphaltene particles started to increase in size. After 20 minutes the temperature reached 160°C the asphaltene particle remains in size.



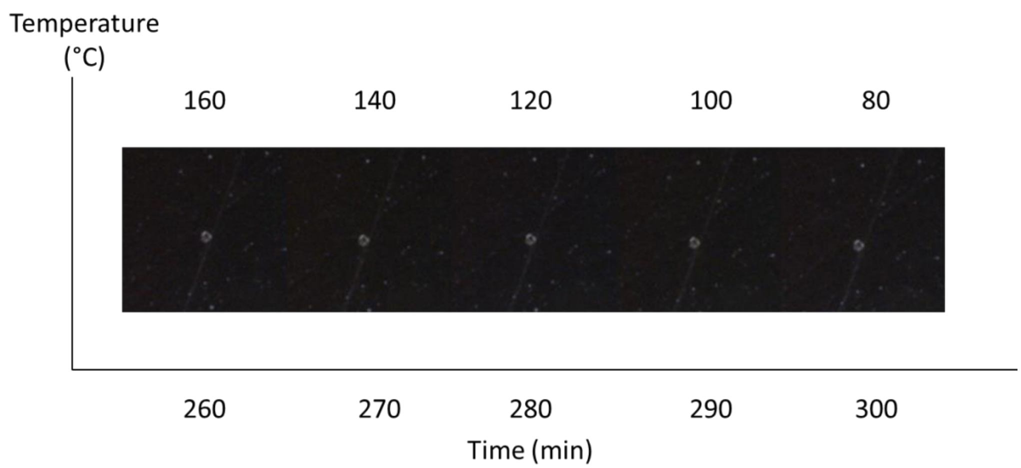
From this graph, we can see that the size of asphaltenes particles remains constant throughout the 50 minutes at 160°C.



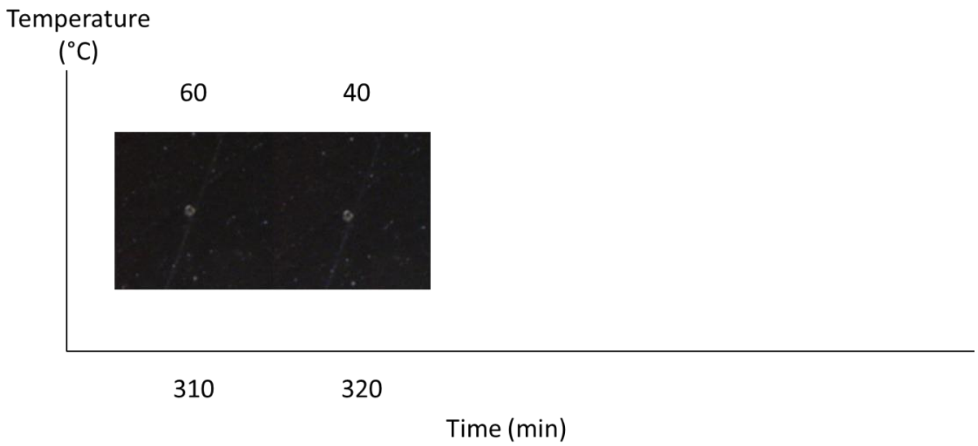
From this graph, we can see that the size of asphaltenes particles remains constant throughout the 50 minutes at 160°C.



In this graph, the asphaltene particle seems to decrease in size, but the change is hardly noticeable.



After three hours of observation at 160°C, the crude oil was let to cool down. The change in asphaltene particle was again observed during the cooling down.



The crude oil was cooled down until 40°C, during this period, there is no notable change happens in the asphaltene particle.

Results for other crude oils

Other experiment was conducted to test the effect of asphaltene precipitation. The other crude oil tested was Bintulu crude oil, Bintulu residue which is Bintulu crude oil after refinery, and also Neat Pyrenes. However, for these types of crude oil, the result is not significant enough to analyze.

4.3 Discussion

From the above result, we can say that different types of crude oil will have different effect on asphaltene precipitation. This is because, each of the crude oils have different properties. Other crude oils that had been tested shows sign of asphaltene precipitation but the change is not obvious to be discussed in this report.

From the Arab Heavy crude oil observation, temperature does have some effect on asphaltene precipitation. When the temperature of crude oil reached 140°C during heating, the size of the asphaltene particles had been decreased and started to increase after 10 minutes when the temperature reached 160°C.

Furthermore, throughout the experiment, the asphaltene particles remain constant in size. The asphaltene particle started to decrease in size a little at 240 minutes.

CHAPTER 5.0:

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

As a conclusion, asphaltene are the heaviest and most complex molecules in crude oil and are defined by its solubility class as the constituents of oil which are soluble in toluene but insoluble in n-heptane. The precipitation of asphaltene can caused problems to oil production, oil transportation in flow lines and in the refining process. There are many parameters that affect asphaltene precipitation such as the pressure, the temperature, the precipitant. Therefore, it is important to study the behavior of these asphaltenes precipitation to provide a suitable counter measure to enhance oil production. To study the effect of temperatures on asphaltene precipitation, a thin film micro-reactor is used. The experiment was conducted using five different types of crude oil with different properties. The crude oil that precipitates most asphaltene is said to have low stability. By understanding the behavior of asphaltene precipitation, oil production efficiency can be increased thus reducing the cost of maintaining the faulty equipment.

5.2 Recommendations

As a recommendations, more experiment need to be done to test the asphaltene precipitation on different types of crude oil. The author encounters some problem with the equipment during the early period of this project. Hence, the amount of time to conduct experiment is limited. More experiment is needed to fully understand the behavior of asphaltene particle at different types of crude oil and different temperature. This is to come with a counter measure to prevent more problem caused by asphaltene in the petroleum industry.

A higher pixel of microscopic device can be used to record clearer picture of asphaltene particles in the crude oil. This is because, by using a higher pixel of microscopic device, we can observe clearly any change that happened in the crude oil.

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