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Effect of Ionic Liquid on Asphaltene Dispersion

by

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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the
Chemical Engineering Programme
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Approved by,

(Dr. Zakaria B. Man)

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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.

SHARIFAH TANTIE SUZIANA BINTI WAN MUSTADAT

ABSTRACT

Study of asphaltenes characteristic and the effect of ionic liquid on asphaltenes dispersion. Ionic liquid is use to treat crude oil in order to reduce amount of asphaltenes in crude oils. Unstable asphaltenes can cause problems in oil production, transportation and processing because depositions of asphaltenes during petroleum production can cause operational problems most of which are total or partial blocking of pipelines and reservoirs, changes in wettability and equipment damages (stick to walls of conduit and reservoirs).

This project is to study the characteristic of asphaltenes and the effect of ionic liquid on dispersion of asphaltenes. Ionic liquid is introduce to treat crude oil originate from Malaysia. Ionic liquid is use to treat Ratawi cude oil in two different temperatures. Three type of ionic liquid were used to treat crude oil within two different temperatures. Amount of asphaltenes before and after treated with ionic liquid is determined and analyze using Elemental Analysis (CHNS analyzer). Determination of asphaltenes is using ASTM D-3279. Ionic liquid were used in this project to determine its ability to reduce asphaltenes in crude oil.

Ionic liquid 1-Butyl-3-Methylimidazolium trifluoromethanesulfonate (Bmim OtF) give the lowest time separation between crude oil and ionic liquid and when crude oil is treated with Bmim Otf amount of asphaltenes is less than the initial in crude oil.

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CHAPTER 1

INTRODUCTION

1.1 BACKGROUND OF STUDY

Organic deposition has become seriously problems that affect petroleum industry around the world which increase the production operational cost. Deposits formation during petroleum during petroleum production causes severe operational problems which are fouling related and affect valves, chokes, filters and tubing [1]. Asphaltenes become unstable as the volume fraction of aliphatic components increase [1]. If the aliphatic fraction of the oil reaches a threshold limit then asphaltenes begin to flocculate and precipitate [1]. Asphaltenes are defined as the fraction separated from crude oil or petroleum products upon addition of hydrocarbon solvents such as n-heptane (Speight, 1999) [2].

Asphaltenes problem occurs in most of production well and production facilities, for example in Petrona Penapisan Melaka (PPM) Sdn. Bhd., Monagas province of Eastern Venezuela and many more. Thus, solutions for this problem have been studied by many researchers in order to overcome the problems.

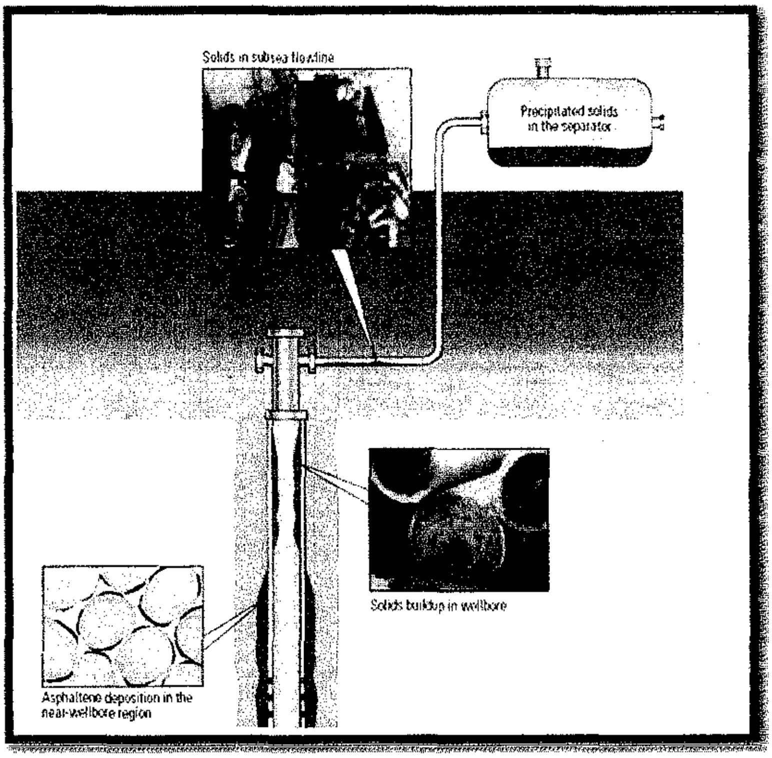

Figure 1.1: Asphaltenes percipitation and depostion occurs.[13]

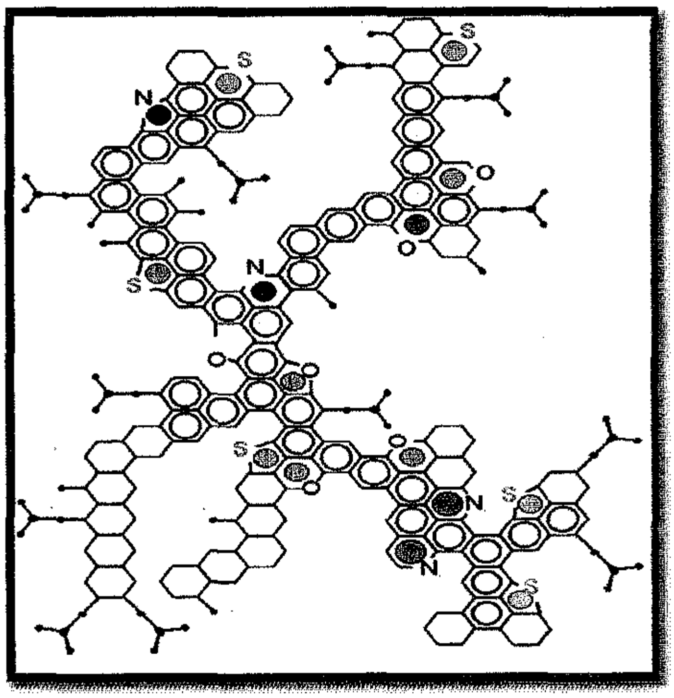

Figure 1.2: Molecule structure of asphaltenes proposed for Maya crude (Mexico) by Altamirano, et al. [IMP Bulletin, 1986]

Previous study has been done by Fan Hong-fu, Li Zhong-bao and Liang Tao in Experimental study on using ionic liquid to upgrade heavy oil in China [3]. They are looking into effect of ionic liquid on viscosity, hydrocarbons composition and average molecular weight of the heavy oil.

Recently, room temperature ionic liquid have come to researcher attention because the application of ionic liquid have a good potential to grow in market because it is environmentally friendly and catalytically active solvent [3]. Ionic liquid were used to treat crude oil to reduce the amount of asphaltenes in crude oils.

In this project, study will be conducted on how to overcome asphaltene issues in crude oil before it fed into refinery process as asphaltene will affect the lifespan of equipments involve in the process. There are several options to overcome this precipitation in crude oil which are asphaltene-removal, stabilize asphaltene and asphaltene-cracking; third option can increase the yield of light product. In order to achieve goal of this project, dispersion of asphaltene will be the option here and three different ionic liquids; a new technology; will be used to study the capability of ionic liquid itself to dispersion of asphaltene from decay the wall of equipments involved and affect its lifespan. Treat crude oil before entering refinery by disperse the asphaltene by using ionic liquid.

1.2 PROBLEM STATEMENT

Unstable asphaltenes can create a serious problem during production such as arterial blockage, blockage in the oil reservoir, in the well, in the pipeline and the in the processing facilities [Mansoori, 1995, 1996] [8]. Asphaltene deposits taken from tubing strings are hard, brittle, dark black, dry solids of similar appearance to coal and other bitumens. These deposits are very difficult to remove from a system because normal thermal methods of hot oil or water are totally ineffective [11].

Figure 1.2: Example of the appearance of asphaltenes from Mars-P crude oil with excess of a) n-pentane and b) n-heptane

1.2.1 Problem identification

To find solution to reduce the amount of asphaltenes in crude oil before entering processing facilities and the best ionic liquid to be use to treat crude oil. Certain temperatures are manipulated to determine which temperature gives the best effect to mixing ionic liquid with crude oil. Ionic liquid are choose based on the hazardous level, polar molecule and insoluble in crude oil.

Types of ionic liquid that will be use in this project are:

NO	IONIC LIQUIDS
1	1-Hexyl-3-Methylimidazolium bis [trifluoromethylsulfonyl] imide (commercial ionic liquid) - Hmim Ntf
2	1-Butyl-3-Methylimidazolium octylsulfate (commercial ionic liquid) – Bmim OcSO4
4	1-Butyl-3-Methylimidazolium trifluoromethanesulfonate (commercial ionic liquid) – Bmim Otf

Table 1.1: Types of Ionic Liquid use in this project

1.2.2 Significant of Project

The significance of this project is that the ionic liquid can be recycled and be used over again. Companies as well as universities would be able to use this research to find other ionic liquid that suitable to be used as the solvent to treat crude oil and reduce the amount of asphaltenes in crude oils.

1.3 OBJECTIVES AND SCOPE OF STUDY

1.3.1 Objectives

Upon completing the project, a few objectives need to be achieved. The objectives of study are as follows:

1. To study the characteristic and physical properties of asphaltenes.
2. To study the dispersion of asphaltene in presence of ionic liquid.
3. To reduce amount of asphaltenes in crude oils.

1.3.2 Scope of Study

The study is divided into 5 major parts as follows:

1. Literature Review

In the literature review stage, existing asphaltenes and ionic liquid are study and identified. Methods and experiments by other researchers are the important highlights to be studied during this stage.

2. Experimental

After identifying the asphaltenes physical properties and characteristic, method of experimental will be selected for laboratory test. The methods of experiments that are suitable will be used in laboratory tests.

3. Laboratory Set Up

Tools and equipment to be used will be identified and familiarized prior to the laboratory tests to avoid any inaccurate result. Accuracy of equipments and chemicals used in the tests also will be checked in order to get accurate results.

4. Laboratory Tests

A series of laboratory tests will be performed in the laboratory that suites the experiment.

5. Analysis of Results

Results obtained from the laboratory tests will be analyzed and interpreted.

CHAPTER 2

LITERATURE REVIEW AND THEORY

2.1 Crude Oil

Crude oil is thick, flammable, yellow-to-black mixture of gaseous, liquid, and solid hydrocarbons that occurs naturally beneath the earth's surface consisting of a complex mixture of hydrocarbons of various molecular weights, plus other organic compound [10]. Crude oil can be separated into fractions including natural gas, gasoline, naphtha, kerosene, fuel and lubricating oils, paraffin wax, and asphalt and is used as raw material for a wide variety of derivative products [10]. The proportion of hydrocarbons in the mixture is highly variable and range from as much as 97% by weight in the lighter oils to as little as 50% in the heavier oils and bitumens [10].

2.2 Asphaltenes

Asphaltenes are aromatic based hydrocarbons of amorphous structure [11]. Asphaltenes consists of condensed polynuclear aromatics and contains small amounts of heteroatoms (S, N and O), and traces of nickel and vanadium [12]. Asphaltenes stability depends on a number of factors, including the composition of surrounding fluid, pressure and temperature [2]. The effect of composition and pressure on asphaltenes precipitation is generally believed to be stronger than the effect of temperature [2].

On heating above 300-400 °C asphaltenes are not melted, but decompose, forming carbon and volatile products [9]. They react with sulfuric acid forming sulfonic acids, as might, as might be expected on the basis of the polyaromatic structure of these components [9].

Asphaltenes constitute an important dispersed phase of crude oils, petroleum residues, bitumens as well as of processed fuels [5]. Asphaltenes are regarded to be polar species, formed by condensed polyaromatic structures, containing alkyl chains, heteroatoms (such as O, S and N) and some metals [5]. The tendency of asphaltenes, to self-aggregate distinguishes them from other oil constituents [5]. Asphaltene aggregation is the cause of complex non-linear effects in such phenomena as adsorption at solid surfaces, precipitation, fluid's rheology, emulsion stability [5].

Current study about asphaltenes is all about the stability and to learn the asphaltenes characteristic. The current treatment is asphaltene dispersion in dilute oil solution. The primary importance is to reveal new details of dispersion formation, especially at initial stages of appearance of the simplest molecular aggregates. The result of this experiment is asphaltenes dispersions in dilute solutions of a crude oil. The problem with this experiment is its can only be test on certain concentration, given less chance to get better result.

2.3 Ionic Liquid

The new technique to study about asphaltenes dispersion is using ionic liquid. Ionic liquid is a liquid that contains essentially only ions. Ionic liquid are salts whose melting point is relatively low (below 100 °C). Salts that are liquid at room temperature are called room-temperature ionic liquids. Ionic liquids have no measureable vapor pressure. Characteristic of ionic liquids are electrically conductive, low combustibility, excellent thermal stability, wide liquid range to be use with, favorable solvating, a catalyst and it also can be solvent and have high selective reactions. It is an ideal way to use ionic liquid to study the asphaltenes because ionic liquid is insoluble with crude oil and it can be recycled and can be use again which will lower the cost. In this project, ionic liquid use has no transition metal and the crude oils will be use is from Malaysia reserves.

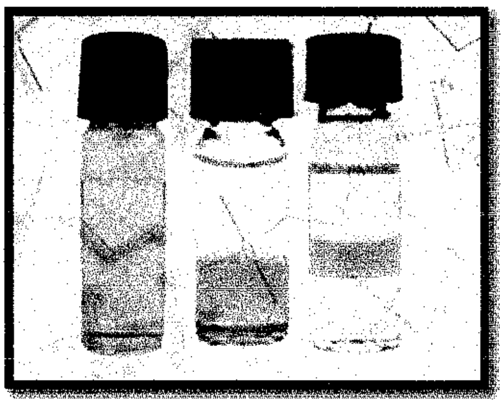

Figure 2.1: Example of Room Temperature Ionic Liquid

There is a study on using ionic liquid to upgrade heavy oil. These experiments investigate the effect of ionic liquids on viscosity, hydrocarbon composition and average molecular weight of the heavy oil [3]. The results indicate that ionic liquids have good viscosity reduction property for the heavy oil [3].

After reaction with ionic liquids, the contents of saturates, aromatics and resins in the heavy oil increase, while the content of asphaltenes decreases [3]. This leads to the decrease of the average molecular weight and the reduction of viscosity of the heavy oil [3]. The results also indicate that metal ion modified ionic liquids have the catalytic effect on upgrading the heavy oil [3].

The experiments are using modified ionic liquid that have transition metal and crude oil from China [3]. The ionic liquid and the organic sulfur in the heavy oil formed a complex formed which weakens the C-S bond and causes the breakage of the C-S bonds [3]. Thus, the bond is break and the average molecular weight is reduced, and the content of saturates and aromatics in the product increase [3].

In this project, three types of ionic liquid is use to treat crude oil (refer Table 1). Ionic liquid use in this project do not contains any transition metal and it is commercial ionic liquid. In the end one of the ionic liquid is choose to be the most suitable to be use to treat crude oil.

CHAPTER 3

METHODOLOGY/PROJECT WORK

3.1 OVERALL

The project is mainly based on researches through past papers (journals, thesis, etc.) to understand more on the dispersion of asphaltene and behaviour of asphaltene and also laboratory experiments to determine asphaltenes and effect of ionic liquid on asphaltene dispersion where temperatures and type of ionic liquid were manipulated. Literature review was done to gain a better understanding before implementing the experiment firsthand.

3.2 EXPERIMENT

3.2.1 Flow of experiment:

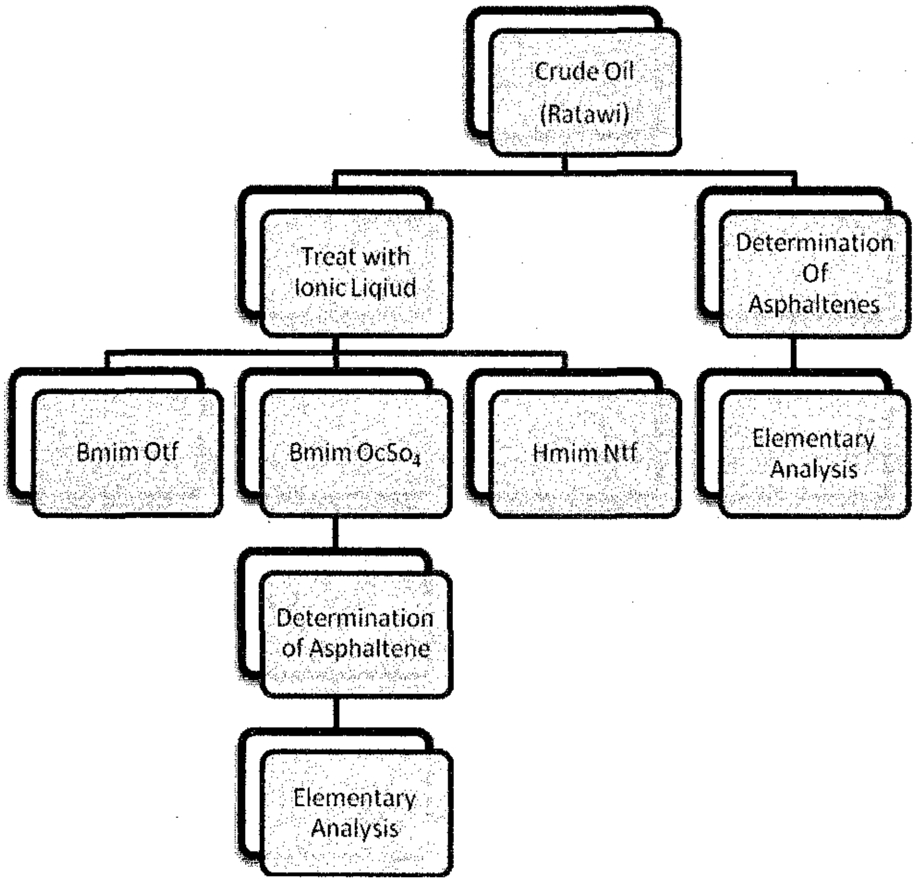

Figure 3.1: Flow of Experiment

3.2.2 Experiment 1 : Determination of asphaltene (n-heptanes insoluble) in crude petroleum [5]

Chemical:

1. Crude Oil. (Ratawi)
2. N-Heptane, 99.0 minimum mol % (Pure Grade)

Apparatus and Material:

1. Erlenmeyer Flask, of 250 mL capacity adapted to an Allihn-type reflux condenser, each with a 35/25 ball joint.
2. Magnetic stirrer and Magnetic-Stirrer Hot Plate, equipped with a voltage regulator.
3. Gooch Crucible, glazed inside and outside with the exception of the outside bottom surface. The approximate dimensions shall be a diameter of 44 mm at the top tapering to 36 mm at the bottom and a depth of 28 mm.
4. Filter pad, glass-fiber 32 mm in diameter.
5. Filter Flask, heavy-wall with side tube, 500-mL capacity.
6. Filter tube, 40 to 42 mm in inside diameter.
7. Rubber Tubbing, or adapter for holding Gooch crucible on the filter tube.

This is a procedure for the determination of the heptanes insoluble asphaltene content of crude petroleum which had been topped to an oil temperature of 98°C.

Procedure:

1. Weight 1g of sample in 250 mL Erlenmeyer flask using 1.0 g of heavy oils.
2. Add n-heptane in ration 100 mL of solvent : 1g of sample.
3. Place the Erlenmeyer flask on the magnetic stirrer hot plate and secure under reflux condenser for 15 to 20 minutes.
4. Place the crucible in an oven at 107°C for 15 minutes, allow to cool in a desiccators.
5. Weight the gooch crucible to the nearest 0.1mg.

6. Set-up the filtering crucible plus filter pad in the suction flask and pre-wet with 5 mL of n-heptane.
7. Let the Erlenmeyer flask to cool down till 38°C to 49°C on the hot plate.
8. Pour its content through the filter using a gentle vacuum.
9. Supernatant liquid id filtered first and insoluble filter last
10. Control the flask while transferring the final precipitate using spatula.
11. Wash the precipitate with three portion of n-heptane about 10 mL each.
12. Place the crucible in 107° for 15 minutes.
13. Cool in desiccators and weight.

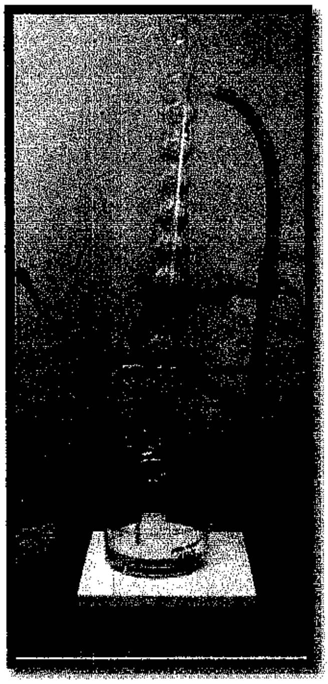

Figure 3.2: Apparatus Setup for n-Heptane Procedure

3.2.3 Experiment 2: Mixing Process of Crude Oil and Ionic Liquids

Reagent and Material

i. Crude Oil, (Ratawi)

ii. Bmim $OcSO_4$

iii. Bmim Otf

iv. Hmim Ntf

Apparatus

i. Syringe,10ml
ii. Rubber tubing as a top cover of the syringe.

Equipment

i. Waterbath
ii. Vortex
iii. Centrifuge

This is a procedure for the mixing of ionic liquid with crude oil.

Procedure:

1. Add 2 ml of ionic liquid in syringe at the bottom and add 2 ml of crude oil on top. Close the top of syringe using stopper and secure with rubber cover. Repeat with another two ionic liquid.

2. 3 syringes contain Bmim $OcSo_4$, Bmim Otf and Hmim Ntf with crude oil into water bath with temperature 70°C for 10 minutes.
3. Put the syringe on vortex and mix the crude oil and ionic liquid by using 2500 rpm for 30 seconds. Repeat with another two syringe.
4. Put the syringes in the water bath with temperature 70°C for 10 minutes.
5. Places the syringes into centrifuge with 2500 rpm for 15 minutes. The result is observed. Repeat another 15 minutes for each syringe have not separated completely.
6. Repeat step 1,2,3,4 and 5 with different temperature (80°C and 90°C).
7. Result are recorded and put into table form.

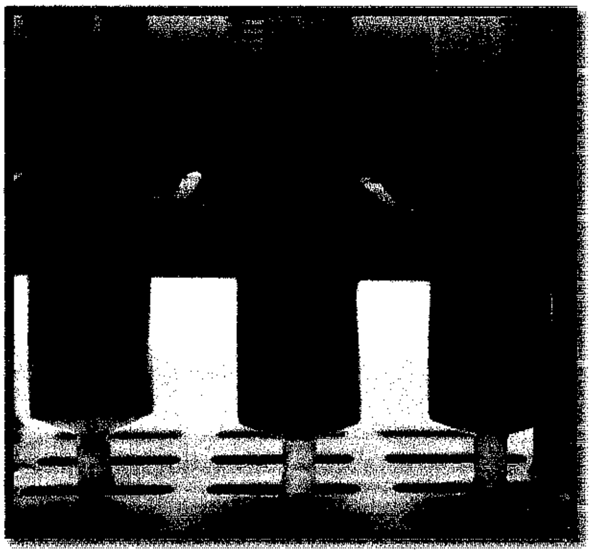

Figure 3.3: Mixing Ionic Liquid with Crude Oil

3.3 GANTT CHART

No	Description	Weeks	1	2	3	4	5	6	7	8	9	10	11	12	13	14
		Dates	19/01/09	26/01/09	2/2/2009	9/2/2009	16/02/09	23/02/09	2/3/2009	9/3/2009	16/03/09	23/03/09	30/03/09	6/4/2009	13/04/09	20/04/09
1	**Set-up laboratory** **-checking equipments** **-checking chemical**															
2	**Start Experiment** **-Determination of Asphaltenes** **-Mixing Ionic Liquid and crude oil** **-Determination of Asphaltenes** **- Elementary Analysis**															
3	**Compile Data and Evaluate result**															
4	**Compile Final Report**															

3.4 TOOLS

This the list of equipment will be using for the experiment conduct in this project:

1. CHNS Analyzer
2. Waterbath
3. Vortex
4. Centrifuge

CHAPTER 4

RESULT AND DISCUSSION

4.1 RESULT

4.1.1 Determination of asphaltene (heptanes insoluble) in crude petroleum

The weight of asphaltene in the sample of crude oil (untreated) is determined in grams.

Subject	Weight (in g)			
	[redacted]			
Crude Oil	1.0023	1.0153	1.0027	1.0027
Crucible	20.213	24.3024	21.0718	24.2957
Crucible with Apshaltene	20.2787	24.3702	21.1446	24.3584
Asphaltene	0.0657	0.0678	0.0728	0.0627
Asphaltene Percentage	6.555%	6.678%	7.260%	6.253%
Mean Asphaltene Percentage	**6.687%**			

Table 4.1 Amount of asphaltenes extracted (untreated)

The weight of asphaltene in the sample of crude oil (treated) is determined in grams.

Subject	Weight (grams)					
	[redacted]					
	OcSo4	Otf	Ntf	OcSo4	Otf	Ntf
Crude Oil	1.0013	1.0036	1.0031	1.096	1.0068	1.0052
Crucible	21.12	21.13	20.2644	21.1185	24.2743	20.2563
Crucible with Asphaltene	21.4044	21.2716	20.6519	21.214	24.3324	20.3188
Asphaltenes (grams)	0.2844	0.1416	0.3875	0.0955	0.0581	0.0625
Asphaltenes Percentage	28.40%	14.11%	38.63%	8.71%	5.77%	6.22%

Table 4.2: Amount of Asphaltenes (Treated)

4.1.2 Mixing Process of Crude Oil and Ionic Liquids

Study on the separation has been conducted in order to determine the capability of the ionic liquids to separate and also to determine the ideal separation temperature for the mixture. As per stated in the methodology part, three types of ionic liquids will be used at six different temperatures.

Tables below showed the result of this study:

Time Taken for phase separation			
Ionic Liquid			
Temperature (°C)			
40	30	90	X
50	30	60	X
60	30	60	X
70	60	60	60
80	15	X	X
90	30	15	75

Table 4.3: Phase Separation Study

4.1.4 Elementary Analysis

Elemental Analysis (wt%)	Untreated Crude Oil		
	Crude Oil Ratawi	Maltene	Asphaltene
Carbon	80.4	83.57	71.38
Hydrogen	12.5	12.78	8.523
Nitrogen	0.493	0.276	1.153
Sulfur	4.66	4.46	7.125

Table 4.4: Elementary Analysis Untreated Crude Oil

Elemental Analysis (wt%)	Treated Crude oil					
	[redacted]			Asphaltenes (90°C)		
	Otf	OcSO4	Ntf	Otf	OcSO4	Ntf
Carbon	72	65.26	63.31	69.25	73.24	67.26
Hydrogen	8.729	6.905	4.931	7.991	6.548	6.27
Nitrogen	5.118	2.512	2.16	1.44	1.433	4.922
Sulfur	10.01	9.147	8.425	8.211	8.885	12.41

Table 4.5: Elementary Analysis Treated Crude Oil for Asphaltene

4.2 DSICUSSION

4.2.1 Determination of asphaltene (heptanes insoluble) in crude petroleum (Untreated and Treated)

This experiment the ratio of solvent to crude oil is 100 : 1 in order to determined asphaltenes. First four experiment we use 1 gram of crude oil and 100 mL of solvent (n-heptane), average asphaltene that we can extract from the crude oil is approximately 0.06 gram. From four experiments of 1 grams of crude oil the mean of asphaltene percentage have been extract from crude oil is 6.687%.

Amount of asphaltenes in crude oil after treated at 70°C and 90°C with ionic liquid is determined. Asphaltenes extracted from 80°C cannot determined because of two of the sample cannot be separated. This may be because of the different tube used during the separation. From the table 4.2 we can see that crude oil which is treated with ionic liquid at 70°C have increase in asphaltene amount compare with initial asphaltenes(untreated) for all three types of ionic liquid. This maybe result from the component in ionic liquid maybe attracted to asphaltenes which make the asphaltene structure even larger. At 70°C, it is not a suitable temperature to mixed ionic liquid with crude oil because some of the ionic liquid did not totally separate from crude oil.

Amount of asphaltenes seem to decrease when the temperature is higher. At 90°C, amount of asphaltenes is less than amount of asphaltenes at 70°C. This means that at high temperature crude oil and ionic liquid is totally mixed and ionic liquid is totally separed from crude oil. Bmim Otf give the lowest amount of asphaltenes, 5.77% compared to initial amount of asphaltenes(untreated), 6.687%. Thus Bmim Otf meets the objective to reduce the amount of asphaltenes in crude oil.

4.2.2 Mixing Process of Crude Oil and Ionic Liquids

From the results above, it is clearly shown that separation at 90°C give the best separation of ionic liquid with crude oil by using centrifuge. Three selected ionic liquids are known to have hydrophobic criteria, however as shown above, Hmim Ntf did not separate from crude oil by using centrifugal separation method. Hence, another separation method should be studied to separate the mixture. Nonetheless, the Hmim Ntf mixture will be extracted to obtain its asphaltenes and will be analyzed in order to study Hmim Ntf dispersion effect on asphaltenes. By acquiring results above, it will give hints in designing the most ideal separation method after mixing the ideal ionic liquid for dispersion of asphaltene in crude oil. Using different tube for the experiment may affect the result of the separation because of the shape of the tube must be constant and makes the separation easy. This explain why two of the sample at 80°C cannot be separated.

Obtained asphaltenes then will be measured by using CHNS analyzer. The ideal temperature for mixing is novel in this project in order to identify the most efficient dispersion of asphaltene and separation process.

4.2.3 Elementary Analysis

From table 4.5 and table 4.6, we can see that the amount of carbon and hydrogen is higher than amount of hydrogen. Carbons are bond with each other forming a ring and have a hydrogen attach to them, thus show why the amount of carbons are higher than hydrogen. This shows that asphaltenes contain aromatic compound. The amount of sulfur in table 4.6 is higher from the amount of sulfur for asphaltenes in table 4.5. This might indicate there are some of sulfur from ionic liquid itself is left in asphaltenes. Asphaltenes is a highly- polar molecule while our ionic liquid also a polar molecule, thus result ionic liquid might make bond with asphaltenes structure that have a lot of branches, refer to Figure 1.1. Ionic liquid will be filling the free space area (branches area). Thus will result to more sulfur in asphaltenes treated with ionic liquid.

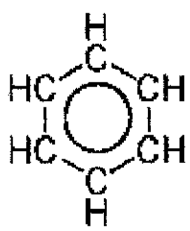

Figure 4.1: Example Structure of Aromatic Compound

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 CONCLUSION

The conclusion of this experiment is the more amount of crude oil is use the more amount of asphaltene can be extract from the crude oil. The best temperature to mixed ionic liquid with crude oil is 90°C. The most easy to separate ionic liquid and reduce the amount of asphaltenes in crude oil is 1-Butyl-3-Methylimidazolium trifluoromethanesulfonate (commercial ionic liquid) – Bmim Otf. Data from elementary analysis shows that there are aromatics compound in asphaltenes by looking at the ratio of C/H in table 4.5 and table 4.6. Amount of sulfur is increase after crude oil is treated with ionic liquid, which indicates some of sulfur compound bond with asphaltenes in crude oil.

5.2 RECOMMENDATION

Recommendation for experiments determination of asphaltenes using n-heptane is to determine the right rpm for each of increase amount of crude oil and solvent use. The rotation also can give effect to the formation of asphaltene in crude oil. The suitable equipment and apparatus should be used.

Experiments for mixing ionic liquid with crude oil should use the suitable test tube for mixing because different shape of test tube will give effect for mixing and separation between ionic liquid and crude oil. The proper amount of ionic liquid and crude oil should be determined because different amount of both ionic liquid and crude oil will also affect time in waterbath and vortex.

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