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Synthesis of Pour Point Depressant from Black Soldier Fly Maggot Oil

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Abstract. This Final Project research focuses on the synthesis of pour point depressant (PPD) from Black Soldier Fly (BSF) Maggot Oil to reduce the pour point of crude oil samples. Crude oil itself is a non-renewable energy source that is still in high demand in Indonesia, especially as a vehicle fuel. Crude oil contains paraffin wax, which can crystallize and hinder the transportation and production processes of crude oil. Therefore, this study aims to produce PPD from BSF maggots as an additive for crude oil to ensure its smooth flow. Maggots were chosen due to their high fatty acid content, ranging from 29% to 32%, making them a potential source for PPD. The method used for PPD synthesis involves esterification using a reflux process. Pour point testing will be conducted using the ASTM D-5853 method. The synthesized PPD could lower the pour point of crude oil by up to 24°C from the original 39°C. Characterization using FTIR testing successfully revealed C-O vibrations (ester groups) at the wave number range of 1158 – 1155 cm^{-1} while eliminating O-H vibrations (carboxylic acid groups). GC-MS characterization was also conducted to identify the formed ester compounds. The GC-MS results showed that PPD 1 had the highest ester content at 87.71%.

Introduction

Crude oil is a non-renewable energy source. The need for crude oil in Indonesia is still very high. One of the most widely used crude oil processed products is as a vehicle fuel to fulfill daily life activities. It is noted that the need for crude oil in Indonesia reaches 1.4 million barrels per day (bpd) [**1**]. Meanwhile, according to the Special Task Force for Upstream Oil and Gas Business Activities (SKK Migas) in 2022, production of crude oil will only reach 616.6 thousand barrels per day (bpd) [**2**]. This is certainly a concern for the government to be able to increase the amount of production to meet current crude oil needs. Seeing the need for crude oil, the crude oil that has been produced will be transported and stored in tankers to be transported to oil refineries for further processing. As is known, crude oil contains many hydrocarbon chain compounds, one of which is paraffin wax. Paraffin wax is a saturated hydrocarbon compound that occurs naturally in nature, comes from minerals, has long, linear, branched chains, and has an empirical formula, namely CnH_{2n+2}. Paraffin wax generally has 18 to 36 carbon atoms (C18-C36) [**3**]. Paraffin wax can inhibit and affect the flow of crude oil in pipes when transported. This is because paraffin wax is stable at room temperature which makes it solidify when it is at room or low temperature [**4,5**]. Thus, paraffin wax is considered an obstacle in the process of transporting crude oil.

To handle this, an additive is needed, namely using a Pour Point Depressant (PPD) which functions to inhibit the formation of larger solids from paraffin wax so that crude oil can still flow at room or low temperature [**6**]. Generally, PPD is made from polymer materials with a mixture of organic solvents [**7**]. PPD is considered efficient and can be used sustainably without reducing the amount of crude oil production because there is no need to shut in wells [**3**]. Apart from that, PPD is also needed to reduce the pour point in crude oil samples. The commercial PPD that is widely used today is

Ethylene Vinyl Acetate (EVA) [**8**]. However, EVA has the disadvantage that it is not very effective in reducing the pour point value. Even though EVA is considered safe to use, if it is not handled properly or the waste is disposed of carelessly, it can cause pollution to the environment (Nur et.al. 2021) [**9,10**].

To replace the use of EVA compounds as PPD, one compound that has the potential to be used as PPD is the FAME (Fatty Acid Methyl Ester) compound. This is because the structure of FAME has a framework similar to paraffin (hydrocarbon). To obtain FAME, you can use fatty acids that can be obtained from maggots which are then esterified with an acid catalyst and reacted with an alcohol source. Maggots have protein levels of around 40-50% and fatty acids of 29-32% [**11,12**]. The fatty acid content in maggots includes oleic acid, palmitic acid, lauric acid, linoleic acid, linolenic acid, and other types of acids [**13,14**]. The highest content was found in lauric acid, namely 49.18% [**15**]. Looking at the high fatty acid content in maggots, it would be a shame if maggots were only used as animal feed. Therefore, maggots can be used as raw material for making PPD which is more environmentally friendly.

Material and Methods

Materials. The materials used are oil from Black Soldier Fly maggot (BSF), methanol, ethanol, 2-propanol and Phenolphthalein indicator were obtained from Merck, H_2SO_4 were obtain from Aldrich; and crude oil samples from Petroleum Engineering. All reagent and chemical used were of analytical grade. Meanwhile, the instruments used for analysis were Fourier-Transform Infrared Spectroscopy from Thermo-Fisher Scientific Nicolet Type iS5-iD1 and Gas Chromatography-Mass Spectrometry by Thermo-Scientific Trace 1310-MS Thermo-Scientific ISQ 9000.

Methods

Determination of Free Fatty Acid Levels (%FFA)

3 grams of maggot oil was weighed and then put into an Erlenmeyer flask. In a separate place, 30 mL of ethanol was heated for ±15 minutes at 60ºC using a hotplate. Then, the Erlenmeyer flask containing maggot oil was added with heated ethanol and then homogenized. After that, 3 drops of PP indicator were added to the mixture. 0.05 N NaOH was put into the burette as a titrant. The mixture was titrated using 0.05 N NaOH until the color changed to light pink. This is done in duplicate. The volume of NaOH was recorded and the %FFA was calculated. The formula for calculating %FFA is as follows.

$$\%\ \mathrm{FFA} = \frac{\text{mL NaOH x N NaOH x MM Fatty Acid}}{\text{weigh sample x 1000}}\ \text{x } 100\% \qquad \text{[Equation 1] [18]}$$

Synthesis of Pour Point Depressant (PPD)

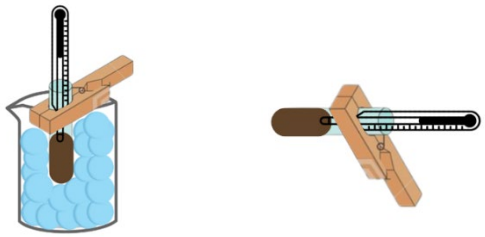
The first step in the synthesis process is esterification using the reflux method. 50 grams of maggot oil was weighed and put into a three-neck flask. Next, the maggot oil is heated while stirring using a magnetic stirrer for 30 minutes at a temperature of 60ºC. In a different container, methanol is prepared (the ratio of maggot oil and alcohol used is 1:6). Then, 1% H_2SO_4 is added to methanol. Then, the mixture of methanol and H_2SO_4 was heated for 15 minutes at 60ºC. After heating, the mixture was put into maggot oil, and the esterification process was carried out for 2 hours at a temperature of 60ºC with a speed of 400 rpm. After 2 hours, the results of the esterification process were put into a separating funnel for washing using warm distilled water. Washing is done 2-5x. The washing results are left overnight until the organic phase and water phase are separated. If 2 phases have been formed, the water phase can be discarded, and the organic phase is taken for the next process, namely evaporation using a rotary evaporator. After evaporation, the results are weighed. This result is what is called Pour Point Depressant (PPD). The resulting PPD is calculated by the %yield value using the following equation:

$$\%\mathrm{yield} = \frac{\text{Actual weigh of PPD}}{\text{Weight of Oil}}\ \text{x } 100\% \qquad \text{[Equation 2] [17]}$$

Do the same for other variations of alcohol.

Pour Point Testing

Testing of the pour point value of crude oil samples in this study was carried out using the ASTM D-5853 standard method. The first is to carry out pre-treatment by heating the crude oil sample until it melts at a temperature of ±60ºC. The liquid crude oil sample was put into a test tube which was then injected with PPD from maggot oil which had been esterified with injection doses of 8, 10, 12, 14, and 16%. The temperature was observed every 3ºC interval by tilting it to 90º for 5 seconds. If the crude oil sample is no longer flowing, it will be the observation temperature, while for reporting temperature the pour point is the observation temperature added with 3ºC.



(a) (b)

Fig. 1. (a) Pour Point Testing Equipment Circuit, (b) Pour Point Test Observation Position

Determination of Viscosity and Density Values of Pour Point Depressant (PPD) and Crude Oil.

Determination of viscosity for crude oil samples was carried out using a digital viscometer (Viscometer NDJ-8S). The first thing to do is heat the crude oil so that it is stable at a temperature of 50oC. There are 4 rotors (rotor 1, rotor 2, rotor 3, and rotor 4). Viscosity measurements started by using the smallest rotor, namely rotor 4, at four different speeds (6, 12, 30, and 60 rpm). This is done on all rotors using the same speed. The viscosity value that appears on the screen is recorded, and the value with a percent above 10% is selected. Meanwhile, density determination is carried out using a pycnometer. and the density value can be determined based on the following equation:

$$\rho = \frac{\text{Weight}_{\text{pikno+sample}} - \text{Weight pycnometer (empty)}}{\text{volume pycnometer}} \quad \text{[Equation 3] [20]}$$

Characterization of Pour Point Depressant (PPD)

Data analysis is carried out quantitatively and scientifically in the laboratory to answer the research problem formulation and test hypotheses. Analysis of pour point depressant (PPD) synthesis data was carried out by characterization of Fourier-Transform Infrared Spectroscopy (FTIR) using plat KBr and Gas Chromatography-Mass Spectrometry (GC-MS).

Results And Discussions

Synthesis of Pour Point Depressant

One of the raw materials in the synthesis of Pour Point Depressant (PPD) is fatty acids. The source of fatty acids used in this research is maggot oil. To determine the fatty acid content in maggot oil, the Free Fatty Acid (%FFA) level was tested. FFA testing is needed to control the quality and performance of PPD. According to Om & Achugasim (2023), the higher the %FFA level, the lower the stability of the oil, so its effectiveness as a PPD to reduce the pour point of crude oil will also be lower [16]. In this study, FFA testing was carried out using the acid-base titration method. The average %FFA content of maggot oil is 1.26%. Usually, the ideal %FFA level for PPD is around 0.1-0.5%. However, FFA levels can still be tolerated and varied through esterification reactions. Therefore, an esterification process was carried out on maggot oil. The esterification process for maggot oil uses the reflux method with a ratio of maggot oil and alcohol of 1:6. The alcohols used include methanol, ethanol, and 2-propanol. Before the esterification process is carried out, the alcohol

and catalyst, namely 1% H_2SO_4, are mixed and heated for 15 minutes at 60°C. In this process, alcohol is not a solvent, but a reactant with the catalyst. The results of synthesis using the reflux method produce three products, namely PPD 1 (75.565%), PPD 2 (89.592%), and PPD 3 (94.527%).
After the %yield value was found, the PPD product was then tested for %FFA levels again to check whether the esterification process was successful in reducing the %FFA content of maggot oil which was previously 1.26%. for PPD 1 and PPD 2, the %FFA level decreased, namely to 0.63% and 1.08%. The decrease in the %FFA levels of PPD 1 and PPD 2 indicates that it is true that the esterification process can reduce the %FFA levels. On the other hand, for PPD 3 the calculation results for the %FFA level increased to 1.45%. The %FFA value of PPD 3 is the highest, which means that in PPD 3 the amount of free fatty acids is the highest. Another cause is that the results of PPD 3 are mostly re-hydrolyzed in the esterification process when the synthesis is carried out.

Table 1. Summary of % Yield and FFA Levels

No	Sample	Name of Alcohol	%Yield	%Average FFA
1	PPD 1	Methanol	75,565%	0,63
2	PPD 2	Ethanol	89,592%	1,08
3	PPD 3	2-Propanol	94,527%	1,45

The FFA values is lower than the reference by Afifah, 2020.

FTIR Characterization of Pour Point Depressant (PPD) and BSF Maggot Oil

The process was identifying functional groups and wave numbers for PPD and maggot oil samples was characterized using Fourier-Transform Infrared Spectroscopy Attenuated Total Reflection (FTIR-ATR). Below in Figure 2 below is the spectrum of the synthesized maggot oil and PPD samples.

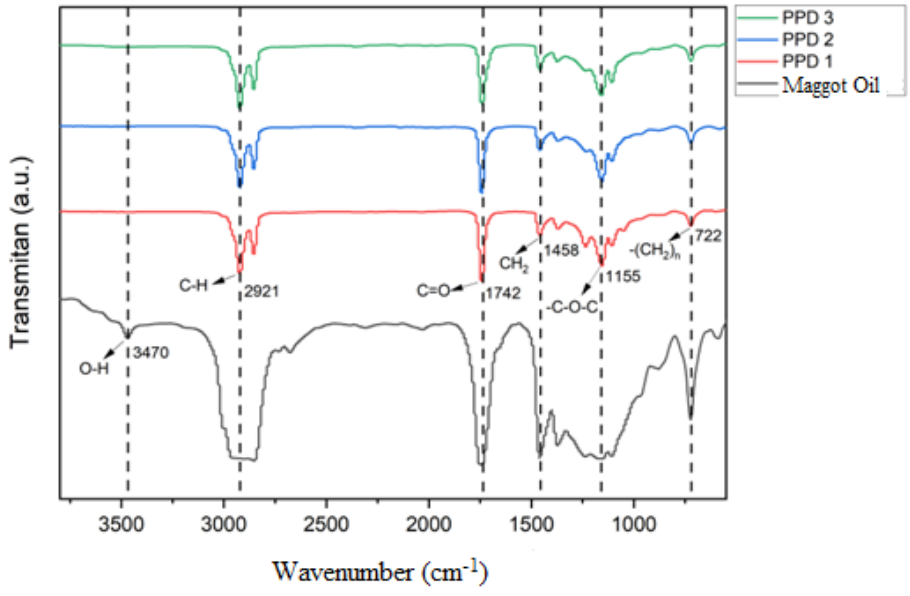


Fig.2. FTIR spectrum of Maggot Oil, PPD 1, PPD 2, and PPD 3

Table 2. FTIR spectrum analysis of Maggot oil, PPD 1, PPD 2, and PPD 3

Vibration	Functional Groups	Wavenumber (cm^{-1})				Literature	
		Maggot Oil	**PPD 1**	**PPD 2**	**PPD 3**	**Kathumbi, et al. (2022), [17]**	**Kamari, et al. (2020) [18]**
O-H- bending	Carboxylate Acid / Alcohol	3470	-	-	-	3480 cm^{-1}	-
C-H aliphatic (Stretching)	Aliphatic ($-(CH_2)_n$)	2850	2921	2921	2921	2926 cm^{-1}	2920 dan 2851 cm^{-1}
C=O (Stretching)	Ester	1750	1742	1742	1740	1741 cm^{-1}	1741 cm^{-1}
C-O (Stretching)	Ester	-	1155	1155	1158	1458 cm^{-1}	1160-1110cm^{-1}

When compared with reference data, there is a shift where the O-H vibration is at a wave number of 3470 cm^{-1}. The O-H vibrations in maggot oil are small. The shift is also indicated by asymmetric C-H stretching vibrations at a wave number of 2850 cm^{-1}. The peak at wave number 2850 cm^{-1} is also not sharp, so the presence of O-H and C-H stretching vibrations indicates that little fatty acids contained in maggot oil are used. This is also supported by the previously determined %FFA of maggot oil, namely 1.26%. The C=O stretching vibrations in maggot oil are at a wave number of 1750 cm^{-1}.

The wave number shift occurs because the components in maggot oil and PPD products are different. Where maggot oil contains triglycerides and free fatty acids. Initially, in maggot oil, the C-H vibrations were in the triglyceride framework, namely in the alkyl part. Then, there was a change in PPD products containing ester compounds. If concluded, the C-H vibration comes from triglycerides and free fatty acids contained in maggot oil, while in PPD products the C-H vibration comes from the alkyl part of the ester. This causes a shift in the wave number. Then, for C=O vibrations, the wave number shift is not too far because the C=O vibrational character of maggot oil and PPD is not different, both originate from the ester group, so the wave number shift does not change significantly. The most significant difference between maggot oil and PPD samples is the O-H and C-O vibrations. The maggot oil sample has O-H vibrations, whereas in the PPD sample, these vibrations are lost. Likewise, in maggot oil, there are no C-O vibrations, while C-O stretching vibrations appear in the three PPD products, namely at wave numbers 1155 and 1158 cm^{-1}. As it is known that the –C-O-C bond is part of the ester, the disappearance of O-H vibrations and the appearance of C-O vibrations in the three PPD products that have been synthesized indicates that the esterification reaction of maggot oil has been carried out successfully. All the vibrations have been comparing with the reference by Kathumbi, et al 2022 and Kamari, et al 2020.

GC-MS Characterization of Pour Point Depressant (PPD)

The synthetic PPD product was subjected to GC-MS characterization to determine the content of ester compounds in it. The following is Figure 3 which shows the chromatogram of PPD 1-PPD 3.

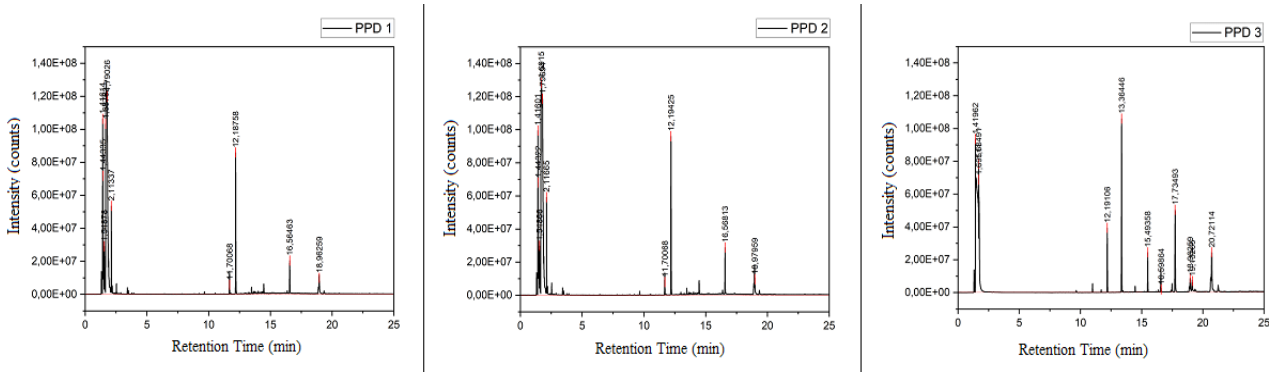


Fig. 3. GC-MS chromatogram of PPD 1-3

For PPD 1, based on the chromatogram data above, 78 peaks appear with retention times between 2.24-20.57 minutes. To find out which ester compounds from the PPD synthesis result are dominant, %Area calculations are carried out. If seen from the %Area, Hexadecenoic acid, methyl ester is the most dominant compound, namely 31.35% at a retention time of 16.56 minutes. If you look at the %Area comparison above, there are still fatty acid compounds in the PPD 1 product, namely 5.88%. Then, the total % area of the alkyl ester compound, namely methyl ester, was obtained at 87.71%.
For PPD 2, based on the chromatogram data above, 67 peaks appear with retention times between 2.24–23.03 minutes. Based on GC-MS analysis, it shows that 1.42% of Undecanoic acid, ethyl ester was formed at a retention time of 12.99 minutes, while the remaining fatty acid, namely Thioacetal acid, was identified at a retention time of 2.24 minutes with a %Area of 0.48%.
For PPD 3, based on the chromatogram data above, 38 peaks appear with retention times between 9.66–26.95 minutes. The dominant propyl ester compound in PPD 3 is Hexadecenoic acid, propyl ester, or propyl palmitate at a retention time of 17.73 minutes with the highest %Area, namely 19.13%. Apart from that, PPD 3 still contains fatty acids with a total %Area of 4.75%. Meanwhile, the total propyl ester formed was 42.90%.
A comparison between the % area of alkyl esters and fatty acids contained in each PPD product that has been synthesized is summarized in Table 3.

Table 3. % Area of Alkyl Ester and Fatty Acids PPD 1, PPD 2, PPD 3

Type Of PPD	% Area	
	Alkyl Esters	Fatty Acids
PPD 1	87,71	5,88
PPD 2	1,42	0,48
PPD 3	42,90	4,75

From the results in Table 3, the highest % area of alkyl esters is PPD 1 > PPD 3 > PPD 2. The % area value for PPD 1 reaches 87.71% which indicates that many esters produced from the esterification process are formed even though they are not perfect as proven by There are still fatty acid compounds contained in it, although it is relatively small, namely only 5.88%. Meanwhile, the %Area PPD 2 obtained was the smallest, indicating that ester formation was not optimal.
The reaction used is esterification, the basic principle of esterification itself is a substitution reaction. The main factor in substitution reactions is the aspect of nucleophilicity. Nucleophilicity in this reaction is represented by the presence of alcohol. If you look at the functional groups involved, they are all alcohols, so the electron source aspects are the same. However, what differentiates %Area is from the alkyl attached to the alcohol. The presence of alkyl can interfere with the nucleophilicity of OH. Disturbances can be seen from the presence of steric hindrance. The one with the strongest steric hindrance is ethanol, so that when ethanol wants to enter the C atom in the carboxylic acid it will be

blocked and cause the ester yield for PPD 2 to be small. Furthermore, 2-propanol has many carbons but is branched, so it is still better than ethanol. And if you look at the reaction mechanism, the nucleophilic character of methanol is strong. Compared to the other two alcohols, methanol has a lower steric hindrance and tends to be lighter because it only has one C atom, so it is easier to attack the C atom in the positively charged carboxylic acid. The ease with which methanol attacks causes PPD 1 to produce more esters which are indicated by the size of the %Area.

Pour Point Testing

Pour point testing was carried out to determine the effect of adding additives, namely synthesized PPD. The method used in testing is ASTM D-5853 [**19,20**] with a minimum pour point type which focuses more on the delay in the formation of paraffin wax. The results of the crude oil pour point test can be seen in Table 4 below.

Table 4. Crude Oil Pour Point Test Results

%PPD	Injection Dosage (mL)		Pour Point (°C)				
	PPD	Crude Oil	Blank	Maggot Oil	PPD 1	PPD 2	PPD 3
0	0	5	39	-	-	-	-
8	0,4	4,6	-	39	39	33	33
10	0,5	4,5	-	36	39	33	33
12	0,6	4,4	-	39	21	36	33
14	0,7	4,3	-	39	21	33	33
16	0,8	4,2	-	39	15	33	30

In crude oil injected with PPD 1, the decrease was quite drastic, namely decreasing by 24°C to 15°C at an injection dose of 16%. PPD 2 can only reduce the pour point of crude oil by 6°C, even when the dose is increased to 16% the pour point value remains 33°C and no further decrease occurs. As stated by Afdahl (2022), the dosage of PPD for each type has an optimum point for the pour point value. So, even if the dose is increased, if it has reached the optimum point, the pour point temperature will not decrease [**15**]. The final test was to use PPD 3 which only slightly lowered the crude oil pour point by 9°C. So, the PPD that is effective for lowering the pour point of crude oil is PPD 1 which uses methanol alcohol. This can occur due to the strong van der Waals interaction between PPD and paraffin wax when PPD is added to crude oil, resulting in inhibition of the formation of paraffin wax so that crude oil can still flow at low temperatures. Maggot oil which contains the fatty acid lauric acid reacts with methyl ester to produce a longer chain (Figure 4), so it can interact with paraffin wax. This is because PPD has two sides, polar and nonpolar. The polar side containing oxygen atoms will produce steric interference when PPD is added to crude oil. After the interaction between crude oil and paraffin wax is broken, the paraffin wax will interact with the nonpolar side of PPD [**20**].

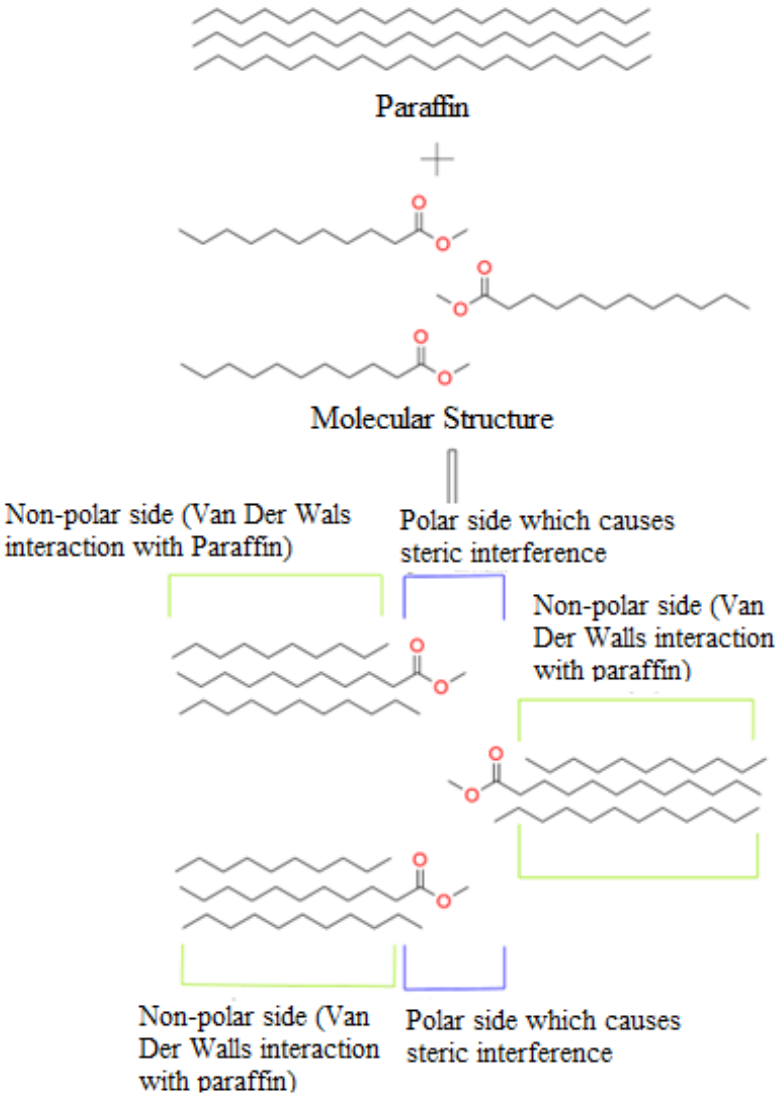


Fig.4. Mechanism of Paraffin Wax Inhibition by PPD 1 [**20**]

Viscosity and Density Test Results

Pour point reduction is also influenced by viscosity and density. The following are the results of the viscosity and density tests which can be seen in Table 5 below.

Table 5. Results of Determining Viscosity and Density Values

Sample	Viscosity (cSt)	Density (kg/m^3)
Crude Oil	27,8	751,1
PPD 1	18,32	833,6
PPD 2	20,37	834,3
PPD 3	18,15	831,6

From the results obtained, the viscosity and density values of PPD 1 are quite low, although not as low as those of PPD 3. Viscosity means viscosity, where the lower the viscosity value, the more soluble it will be, and helps to lower the pour point value. This also applies to density where the lower the PPD density value, the more effective it will be in reducing the pour point value of crude oil. Then, if we look at the ester recovery produced based on GC-MS characterization, PPD 1 will be more effective than PPD 3. This is because PPD 1 has a higher amount of ester, namely 87.71%, whereas for PPD 3 the ester recovery only reaches 42.90%. So, it can be concluded that the most effective PPD for reducing the pour point value of crude oil is PPD 1.

Conclusions

The PPD synthesis was successfully carried out with the highest %yield values is for PPD 3 (94.527%). The addition of synthetic PPD can reduce the pour point value of crude oil, PPD 1 reduced 24ºC with 16% (v/v), PPD 2 reduced it by 6ºC with 8% (v/v), and PPD 3 reduced it by 9ºC with 16% (v/v). The viscosity value of PPD is in the range 18.15 – 20.37 c St. Then, the PPD density value is in the range of 831.6 – 834.3 kg/m^3. The FTIR characterization obtained is that the synthetic PPD contains an ester functional group (C-O) at a wave number of 1158 – 1155 cm^{-1} and there is no carboxylic acid (O-H) group which was originally at a wave number of 3470 cm^{-1}. And GC-MS analysis succeeded in validating the ester compounds formed and the fatty acids. The characterization results show that the amount of ester for PPD 1 is 87.71%, for PPD 2 it is 1.42%, and for PPD 3 the amount of ester is 42.90%.

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