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Interesterification Process of Palm Oil Using Base Catalyst: The Effect of Stirring Speed and Type of Catalyst on Kinetic Energy and Dipole Moment

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Abstract— Making biodiesel which has so far been carried out, is a transesterification process with glycerol by-products. Glycerol has a low economic value and is usually only disposed of as waste. An alternative process for producing biodiesel with more valuable by-products is interesterification. The by-product of the interesterification reaction is triacetin which is widely used in the chemical, food, and pharmaceutical industries. The operating conditions of the interesterification reaction were the reaction temperature of 60°C, the molar ratio of palm oil: methyl acetate = 1: 6, the reaction time of 1 hour, catalyst type (KOH, NaOH), stirring speed (200, 300, 400, 500, 600 rpm) and catalyst mass (0.25, 0.5, 0.75% wt. oil). From the analysis and calculation, the highest FAME yield was 57.30% at reaction temperature 60°C, the molar ratio of palm oil: methyl acetate = 1: 6, reaction time 1 hour, KOH catalyst, stirring speed of 300 rpm, and catalyst mass 0.75% wt oil. From the calculation of ChemDraw software for the *triglyceride + methyl acetate + KOH* system had a kinetic energy of 3,670 kJ/kmol and a dipole moment of 20,330 debyes, whereas the *triglyceride + methyl acetate + NaOH* system had a kinetic energy of 2,977 kJ/kmol and a dipole moment of 11,457 debyes so that the KOH catalyst was superior in terms of reactivity and solubility. Biodiesel produced had an acid value of 0.3927 mg KOH/gr and met ASTM D664 for a maximum acid value of 0.5 mg KOH/gr.

Keywords— Biodiesel; interesterification; triacetin; kinetic energy; dipole moment.

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I. INTRODUCTION

Petroleum supplies are running low along with the increasing rate of the world economy. Biodiesel as a renewable energy source is needed to replace diesel fuel. Biodiesel is more environmentally friendly, the availability of raw materials is guaranteed, and biodegradable [1]. The resulting emissions are also small to reduce the formation of CO₂ gas [2]. Biodiesel is also an alternative fuel that can be used directly or mixed with diesel in diesel engines. As the most prospective biodiesel raw material is palm oil because the price is the cheapest, there are many supplies, and the oil components are safe if used. Palm oil is safer to use than castor oil because castor oil contains *curcin* and *phorbol esters* which are very poisonous [3]. Palm oil is also better used as a raw material than waste cooking oil. Waste cooking oil requires some pretreatment before it is ready to be used as raw material. Waste cooking oil needs a neutralization

process to reduce FFA levels where for FFA biodiesel feedstock must be below 3% because FFA will react with base catalysts that will reduce biodiesel yield [4]. Waste cooking oil also needs a process to eliminate high water content, namely the evaporation process, where the requirements for biodiesel feedstock must be ≤ 6000 ppm [4].

The process of making biodiesel that is commonly done is the transesterification process with glycerol by-products. The process of separating glycerol is relatively difficult because of its high viscosity and glycerol is usually only disposed of as waste [5]. An alternative process for making biodiesel that is more promising is the process of interesterification with its by-products is triacetin, where triacetin is more valuable than glycerol [5]. Triacetin can be used as a plasticizer, polymer and as a *bioadditive* fuel [6].

Several studies of interesterification processes using heterogeneous and homogeneous catalysts have been carried out. In the interesterification of rapeseed oil with *SnO catalyst*,

obtained a yield of 90% at a reaction time of 4 hours, a reaction temperature of 483K, and a molar ratio of methyl acetate: oil = 40 [2]. Interesterification of soybean oil with *CaO* catalyst, obtained 62.3wt% FAME at reaction temperature 325°C, a molar ratio of oil: methyl acetate = 1:40, and reaction time 2 hours [7].

Homogeneous catalysts have the advantage of a faster reaction rate [8]. Casas et al. [5] interesterification of sunflower oil with *CH₃OK* catalyst and obtained a mass fraction of FAME 20% at a reaction time of 15 minutes. Kusumaningtyas et al. [9] obtained a yield of 9% at interesterification of castor oil at 60°C, the molar ratio of oil: ethyl acetate = 1:6, and reaction time of 8 hours. Chuepeng and Komintarachat [10] obtained a yield of 50% on interesterification of used cooking oil with a *CH₃COOH* catalyst at a reaction temperature of 80°C, a molar ratio of oil: ethyl acetate = 1:30, and a reaction time of 3 hours.

The interesterification process is also carried out using a biocatalyst and solvent. In the insect fat interesterification with *Novozym* 435 catalyst, the yield was 96.97% at the reaction time of 12 hours, the molar ratio of fat: methyl acetate = 1:14.64, and the reaction temperature was 39.5°C [11]. In the interesterification of ultrasonic *Crambe* oil with lipase enzyme *Novozym* 435, the FAME yield was 98.25% at a reaction temperature of 60°C, the reaction time of 6 hours, the mole ratio of methyl acetate: oil = 12, catalyst 20% by mass oil and solvent n-heptane [12]. *Crambe* oil interesterification at 350°C, the mass ratio of oil: methyl acetate = 1:4, reaction time 45 minutes to get 25% yield [13]. Interesterification with co-solvent *methyl palmitate*, yield 83% methyl oleate at a reaction temperature of 120°C, a mole ratio of methyl acetate: oil = 20, iron sulfate catalyst 7.5%- and 24-hours reaction time [14]. Interesterification with γ -alumina catalyst 5% obtained a yield of 52.49% at a reaction time of 1 hour, a reaction temperature of 250°C, and a mole ratio of methyl acetate: macaw oil = 30 [15].

The research done has never discussed the stirring speed variable in the interesterification reaction and the role of the catalyst as a reactant solvent, namely triglycerides and alkyl acetate. Stirring is essential in the interesterification reaction because it will magnify the collision fraction between the reacting components. Biodiesel production yields 42% at a stirring speed of 200 rpm in the transesterification process and a reaction time of 123 minutes [8]. The research objective was to find the optimum conditions of stirring speed and reaction time in palm oil's interesterification process into methyl esters and triacetin. Besides, this research aims to compare NaOH and KOH catalysts' performance in the interesterification process in terms of its role as a solvent in accelerating the reaction.

II. MATERIALS AND METHOD

A. Materials

The ingredients used were palm oil, *aquadest*, *oxalic acid*, *KOH* (*Riedel-de Haen*, 99%), *NaOH* (*Riedel-de Haen*, 99%), *methyl acetate* (*Sigma Aldrich*, 99.9%), indicator PP, *acetone* (*Merck*, 99.8%), *CH₃COOH* (*Sigma Aldrich*, 99.7%).

B. Raw Material Preparation

Palm oil was analyzed for FFA concentration and water content. If the FFA concentration < 3% and water content ≤ 0.6% can be directly used for the interesterification reaction. If the FFA concentration > 3% esterification reaction was carried out and if the water content > 0.6% was evaporated in an oven at 110°C until the mass was constant.

C. Interesterification Reaction with Base Catalyst

Palm oil (250 grams), methyl acetate (molar ratio of oil: methyl acetate = 1: 6), and *KOH* catalyst (0.25; 0.5; 0.75% oil mass) were weighed according to the study variables. Palm oil was put into a three-neck flask, methyl acetate, and the catalyst was put into a separate *Erlenmeyer*, then each material was heated at 60°C. After the reaction temperature of 60°C was reached, methyl acetate and catalyst were put into a three-neck flask containing palm oil that has been heated to a temperature of 60°C with a hot plate. After all the ingredients were put into a three-neck flask that has been equipped with a condenser for reflux, stirring was carried out with a magnetic stirrer with stirring speed (200, 300, 400, 500, and 600 rpm), and the calculation of the reaction time was started according to the study variable. After the reaction time of 1 hour was reached, 20 grams of reaction sample was taken for each study variable to be carried out for the separation and purification process.

The optimum conditions for the stirring speed of the interesterification reaction with the KOH catalyst were used for the variable interesterification reaction with the NaOH catalyst with variations in the mass of the catalyst (0.25; 0.5; 0.75% mass of oil). Other operating conditions were the same as the interesterification reaction with the KOH catalyst.

D. Separation and Purification

The reaction sample was then added with hot water at 60°C as much as 50% of the sample volume and 0.03% acetic acid was also added from the sample volume. The mixture was then stirred with a magnetic stirrer speed of 300 rpm for 20 minutes. The sample was then inserted into the separating funnel for 1 hour to separate the water washing and methyl esters. In the separating funnel 2 layers will be formed; namely the top layer was a non-polar compound, namely methyl ester, methyl acetate and residual reaction oil. While the lower layer was a polar compound that was triacetin, water and the catalyst neutralization reaction. Washing was repeated until the water was clear and pH neutral. After the washing water was clear and pH neutral, the top layer was then distilled at 105°C to separate the methyl acetate and the remaining washing water. Distillation was carried out until no distillate was dripping [16]. Distillate residues, namely methyl esters, and residual reaction oil, were weighed as product mass. Calculate the yield with equation (1):

$$\text{Yield (\%)} = \frac{\text{mass of product}}{\text{mass of initial oil}} \times 100\% \quad (1)$$

E. Analysis

1) *Analysis of Density*: Empty pycnometers were weighed and their mass recorded. The sample was put into a pycnometer, and its mass was recorded. Density was calculated by equation (2):

$$\rho = \frac{\text{mass of pycnometer contains} - \text{mass of pycnometer empty}}{\text{volume of pycnometer}} \quad (2)$$

2) *Analysis of Acid Value* [17]: 0.5-gram sample was added to the *Erlenmeyer*, and 50 ml acetone was added. The sample was then added with 3 drops of PP indicator and titrated with 0.1 M KOH solution until pink. The pink color must last for at least 15 seconds. The acid value was calculated by equation (3):

$$\text{Acid value} = \frac{56.1 \times \text{volume of KOH} \times M \text{ KOH}}{\text{mass of sample}} \quad (3)$$

3) *Analysis of Methyl Esther*: Analysis of FAME composition using Gas Chromatography (GC) Shimadzu brand with data:

- Column: BPX5 0.25 μ m, 12 m x 0.53 mm ID
- Initial temp: 100°C, 0.5 min
- Rate 1: 50°C / min
- Temp: 280°C
- Rate 2: 10°C / min
- Final temp: 360°C, 5 min
- Gas carrier: He, 5 min
- Detector: FID
- Injection mode: on column (OCI-5)
- Injection volume: 1 μ l

The method of this research can be seen in Figure 1 below.

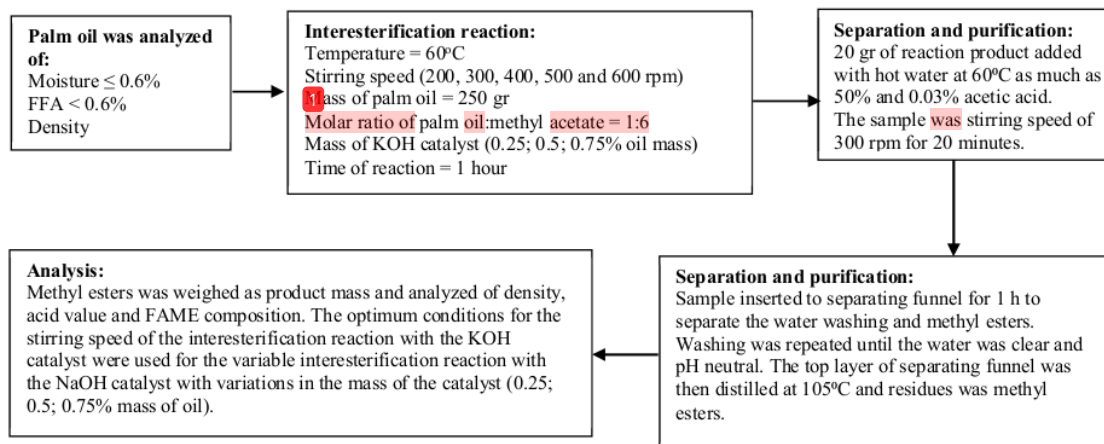


Fig.1 Set up a diagram to interesterification reaction with hot water washing.

III. RESULT AND DISCUSSION

A. Raw Material Preparation

From the initial analysis, results obtained water content 0.015%, FFA 0.1569%, and density 0.93 gr/ml, so that oil can be directly used in the interesterification reaction.

B. Interesterification Reaction with Base Catalyst

In Figure 2, it can be seen that in all the stirring speed variables, the highest methyl ester yield was obtained at the KOH catalyst mass of 0.75% wt. oil.

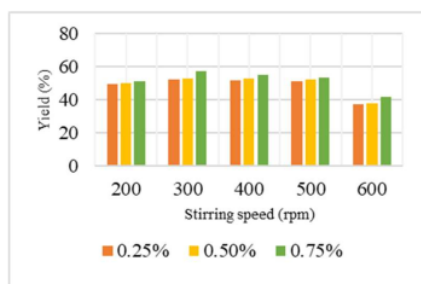


Fig. 2 Relationship between stirring speed (rpm) and yield (%) with variations in the mass of the KOH catalyst (%) at a reaction temperature of 60°C, the molar ratio of oil: methyl acetate = 1:6 and reaction time of 1 hour

The optimum conditions of the study were obtained at 0.75% wt KOH catalyst mass, oil and stirring speed of 300 rpm with a yield of 57.3%. This is because the stirring speed of 300 rpm of the interesterification reaction has reached equilibrium so that if the stirring speed is increased, it will shift the reaction equilibrium towards the reactants because the interesterification reaction is reversible. At the mixing speed of 200 rpm, the methyl ester yield is still low because the material has not mixed properly so the reaction has not reached equilibrium.

The stirring speed is very influential in making the homogeneous reaction mixture and enlarging the collision fraction between the reacting components. This is under equation (4):

$$kA(T) = A e^{-E/RT} \quad (4)$$

Where k (the reaction rate constant) and A (collision frequency factor) are directly proportional. The greater the stirring speed, the collision frequency factor will also be greater, so the reaction rate constant is also greater.

The highest yield of methyl ester was found in the catalyst mass of KOH 0.75% wt. oil. This is because with the amount of catalyst KOH 0.75% wt. it is enough to meet the needs of the reaction catalyst to achieve equilibrium. However, this condition has not yet reached the optimum condition of the study, because it is possible that if the catalyst mass is

increased the yield of methyl esters will also increase. Kusumaningtyas et al. [9] obtained the highest yield on the KOH catalyst mass of 0.5% wt. oil, but there is no known stirring speed used. Casas et al. [5] obtained a 20% FAME mass fraction with a CH_3OK : oil catalyst molar ratio = 0.2 and a stirring speed of 600 rpm. Chuepeng and Komintarachat [10] obtained a 72% yield with a molar ratio of NaOH : WCO catalyst = 0.015 at a reaction time of 3 hours and the stirring speed is unknown.

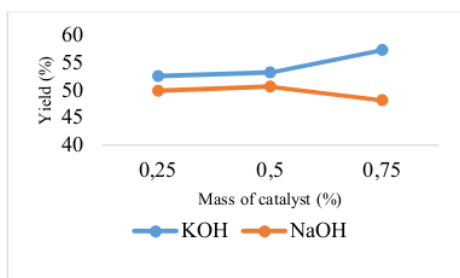


Fig. 3 Relationship between catalyst mass (%) and yield (%) with variations in catalyst type at a stirring speed of 300 rpm, reaction temperature 60°C, molar ratio of oil: methyl acetate = 1:6 and reaction time 1 hour

In Figure 3, it appears that the highest yield is obtained on the KOH catalyst. In the optimum NaOH catalyst yield of 50.65% was obtained at the catalyst mass of 0.5% wt. oil. This is due to the 0.5% wt NaOH catalyst amount. It is enough to meet the needs of the reaction catalyst to achieve equilibrium. If the amount of catalyst is added, it will shift the reaction's equilibrium to the reactants because of the reversible reaction's nature. In contrast to the KOH catalyst, the highest methyl ester yield of 57.30% was obtained at the mass of the 0.75% wt KOH catalyst. oil. This is because with the amount of catalyst KOH 0.75% wt. it is enough to meet the needs of the reaction catalyst to achieve equilibrium. But this condition has not yet reached the optimum condition of the study because it is possible that if the catalyst mass is increased, the yield of methyl esters will also increase.

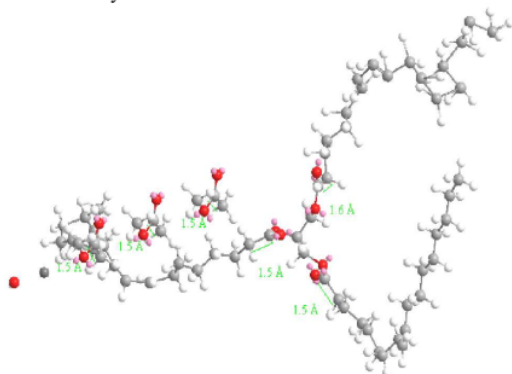


Fig. 4 Molecular dynamic of triglyceride and methyl acetate after adding NaOH catalyst.

The catalyst's function in accelerating the reaction and as a solvent is very important for the reaction. The catalyst's role in accelerating the reaction can be seen from its ability to make the compound unstable so that it is more easily broken.

With ChemDraw software, the C-C bond's distance before and after the catalyst is added. Fig. 4 shows the molecular dynamic of triglycerides and methyl acetate after adding NaOH catalyst while Fig. 5 shows the molecular dynamic of triglycerides and methyl acetate after adding KOH catalyst.

TABLE I
LENGTH OF THE C-C BOND ON THE TRIGLYCERIDE AND METHYL ACETATE MOLECULES WITH NaOH CATALYST

| Bond of C - C | Length of the bond early (Å) | Length of the bond end (Å) |
|--------------------------------------|------------------------------|----------------------------|
| C(7)-C(10) R_3 triglyceride | 1.509 | 1.456 |
| C(8)-C(12) R_2 triglyceride | 1.509 | 1.544 |
| C(9)-C(14) R_1 triglyceride | 1.509 | 1.564 |
| C(69)-C(70) methyl acetate | 1.509 | 1.506 |
| C(74)-C(75) methyl acetate | 1.509 | 1.507 |
| C(79)-C(80) methyl acetate | 1.509 | 1.508 |
| The average of C-C bond length | 1.514 | |

Table I shows that the NaOH catalyst's addition changes the C-C bond's length to triglycerides and methyl acetate. The C-C bond's length, which was originally 1.509 Å, changed to an average of 1.514 Å. This is because the NaOH catalyst makes the triglyceride and methyl acetate molecules unstable, so the C-C bond becomes weak. With weak bonds, it will be more easily broken by Na^+ ions from NaOH. With the breaking of the C-C bond on triglycerides and C-C on methyl acetate, a substitution reaction will occur: the exchange of the place where the R group on the triglyceride occupies the R group on methyl acetate and vice versa according to the mechanism of the transesterification reaction.

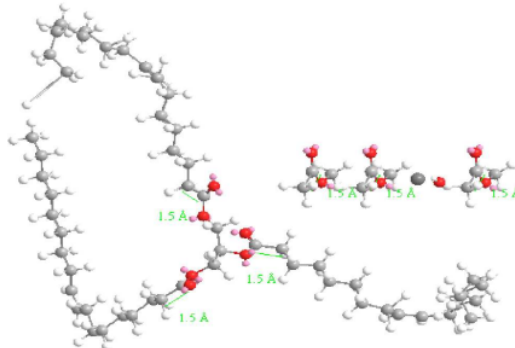


Fig. 5 Molecular dynamic of triglyceride and methyl acetate after adding KOH catalyst.

Table II shows that with the addition of the KOH catalyst, there is a change in the C-C bond length in triglycerides and methyl acetate. The C-C bond's length, which was originally 1.509 Å changed to an average of 1.515 Å. This is because the KOH catalyst makes the triglyceride and methyl acetate molecules reactive, so the C-C bond becomes weak. When viewed from the average C-C bond length, the KOH catalyst produces an average bond length greater than NaOH. This shows that the KOH catalyst makes the molecule more active in moving so that it is easier to break than the NaOH catalyst.

TABLE II
LENGTH OF C-C BOND ON TRIGLYCERIDE AND METHYL ACETATE
MOLECULES WITH KOH CATALYST

| Bond of C – C | Length of the bond early (Å) | Length of the bond end (Å) |
|--|------------------------------|----------------------------|
| C(7)-C(10) R ₃ triglyceride | 1.509 | 1.504 |
| C(8)-C(12) R ₂ triglyceride | 1.509 | 1.522 |
| C(9)-C(14) R ₁ triglyceride | 1.509 | 1.532 |
| C(69)-C(70) methyl acetate | 1.509 | 1.508 |
| C(74)-C(75) methyl acetate | 1.509 | 1.516 |
| C(81)-C(82) methyl acetate | 1.509 | 1.508 |
| The average of C-C bond length | 1.515 | |

Table III shows that the KOH catalyst's addition produces more tremendous kinetic energy than the NaOH catalyst. The greater the kinetic energy, the more active the molecule moves [18]. This relates to the C-C bond's length, where the addition of the KOH catalyst results in a more considerable C-C bond length. If seen from the dipole moment, it can be seen that the addition of the KOH catalyst produces a more excellent dipole moment than the NaOH catalyst. The greater the dipole moment, the greater the solubility of the material [19].

TABLE III
CALCULATION OF TOTAL ENERGY AND DIPOLE MOMENT ON COMPONENTS WITH CHEMDRAW

| Component | E potentia I (kJ/kmol) | E kinetic (kJ/kmol) | E total (kJ/kmol) | Dipole Moment (debye) |
|------------------------|------------------------|---------------------|-------------------|-----------------------|
| TG+methyl acetate+NaOH | -2977 | 2977 | 0 | 11457 |
| TG+methyl acetate+KOH | -3670 | 3671 | 0 | 20330 |

So, it can be said that triglycerides and methyl acetate are more soluble with the KOH catalyst compared to the NaOH catalyst. With more easily triglycerides and methyl acetate dissolve, it will accelerate the transesterification reaction.

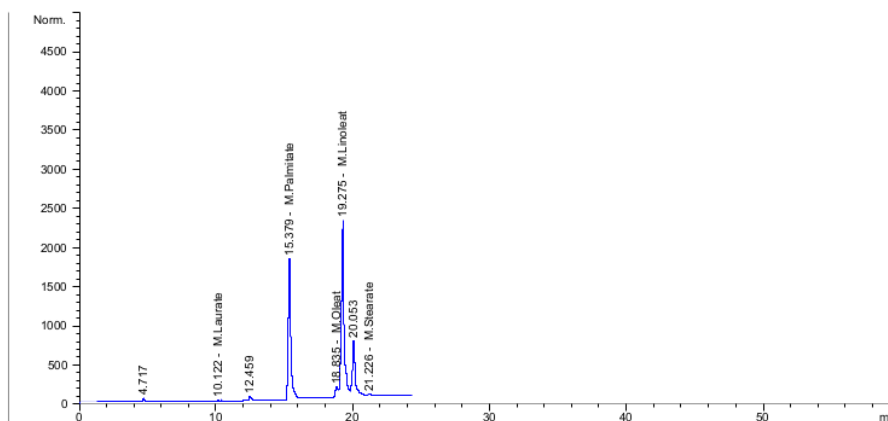


Fig. 6 FAME chromatogram on the mass KOH catalyst of 0.75% wt. oil, stirring speed 300 rpm, reaction temperature 60°C, molar ratio of oil:methyl acetate = 1:6 and reaction time 1 hour

C. Analysis

Data on density, acid value, and methyl ester composition were obtained from the results of the analysis and calculations as reaction products. The data is made in the form of a table showing the effect of the research variables, namely the mass of the KOH catalyst and the reaction time on the density and acid value of methyl ester.

TABLE IV
DATA ON DENSITY AND ACID VALUE OF METHYL ESTER BY
INTERESTERIFICATION WITH KOH CATALYST

| Mass of KOH (% wt. oil) | Stirring speed (rpm) | Density (gr/ml) | Acid value (mg KOH/gr sample) |
|-------------------------|----------------------|-----------------|-------------------------------|
| 0.25 | 200 | 0.9124 | 0.5723 |
| | 300 | 0.9124 | 0.5685 |
| | 400 | 0.9123 | 0.5274 |
| | 500 | 0.9124 | 0.5499 |
| | 600 | 0.9124 | 0.5611 |
| 0.5 | 200 | 0.9124 | 0.5274 |
| | 300 | 0.9124 | 0.4377 |
| | 400 | 0.9123 | 0.4152 |
| | 500 | 0.9124 | 0.4388 |
| | 600 | 0.9124 | 0.5274 |
| 0.75 | 200 | 0.9124 | 0.4096 |
| | 300 | 0.9124 | 0.3927 |
| | 400 | 0.9124 | 0.4040 |
| | 500 | 0.9124 | 0.4489 |
| 600 | 0.9124 | 0.4713 | |

Table IV shows that the density of methyl esters is between 0.9123-0.9124 gr/ml. This value does not meet ASTM D1298, namely biodiesel density between 0.86-0.89 gr/ml [20]. This is because there is still a lot of palm oil that is not converted to biodiesel, so that the density of biodiesel is still close to the initial oil density of 0.93 gr/ml. The acid value that does not meet the standard is for all KOH catalyst mass variables 0.25% wt. oil and catalyst mass variable KOH 0.5% wt. oil with a stirring speed of 200 and 600 rpm. Based on ASTM D664, biodiesel's acid value is maximum 0.5 mg KOH/gr sample [20]. This is because many triglycerides do not react and there is still water in biodiesel. The reaction between triglycerides and water will produce free fatty acids which affect the acid value.

TABLE V

COMPOSITION OF FAME FROM GC ANALYSIS ON KOH CATALYST MASS 0.75% WT. OIL, STIRRING SPEED 300 RPM, REACTION TEMPERATURE 60°C, MOLAR RATIO OF OIL:METHYL ACETATE = 1:6 AND REACTION TIME 1 HOUR

| FAME (Fatty Acid Methyl Esters) | Composition (%) |
|---------------------------------|-----------------|
| Methyl Laurate | 0.7736 |
| Methyl Palmitate | 67.4598 |
| Methyl Oleate | 5.6366 |
| Methyl Linoleate | 26.0750 |
| Methyl Stearate | 0.0549 |

GC chromatogram results for FAME resulting from triglyceride and methyl acetate transesterification reaction on the mass of catalyst KOH 0.75% wt. oil, stirring speed of 300 rpm, reaction temperature of 60°C, molar ratio of oil: methyl acetate = 1:6, and reaction time of 1 hour are presented in Figure 6. The FAME composition, which is from triglyceride and methyl acetate transesterification reaction on the KOH catalyst mass is 0.75% wt oil, stirring speed 300 rpm, reaction temperature 60°C, molar ratio of oil: methyl acetate = 1:6, and reaction time 1 hour are presented in Table V. The biggest FAME composition is methyl palmitate 67.4598% is according to the largest composition of palm oil namely palmitate acid.

IV. CONCLUSION

Stirring speed and type of catalyst are two factors that determine the success of the transesterification reaction between triglycerides and methyl acetate to produce methyl esters and triacetin. From the analysis and calculation results obtained the highest FAME yield of 57.30% at a reaction temperature of 60°C, the molar ratio of palm oil: methyl acetate = 1:6, reaction time 1 hour, KOH catalyst, stirring speed of 300 rpm, and catalyst mass 0.75% wt. oil. From the results of the ChemDraw software calculation. This research found that the KOH catalyst was superior to the NaOH catalyst from its ability to make the reactants more reactive and the energy kinetic and dipole moment higher than the NaOH catalyst. From the analysis results, biodiesel has an acid value of 0.3927 mg KOH/gr and meets ASTM D664 for a maximum acid value of 0.5 mg KOH/gr.

NOMENCLATURE

| | | |
|--------|----------------------------|--|
| ρ | density | gml^{-1} |
| k_A | the reaction rate constant | |
| A | factor collision frequency | |
| E | activation energy | kJmol^{-1} |
| R | ideal gas constant | $8,314 \text{ Jmol}^{-1}\text{K}^{-1}$ |
| T | absolute temperature | K |

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