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One-phase Transesterification of Palm Oil in to Biodiesel with Co-solvent Methyl Esters: The Effect of Adding Co-solvent to Kinetic Energy and Dipole Moment

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Abstract

In the transesterification process, the problem is the low solubility of oil in methanol, so the reaction will run slowly. The solution to this problem is to add a co-solvent to increase the solubility so that a one-phase reaction will be formed. The co-solvent methyl ester is the right choice because it is a product of the reaction itself so that it does not require a separation process. The operating conditions of the study were mass of palm oil 250 g, mass of NaOH catalyst 0.8%wt, stirring speed 100 rpm, reaction temperature 60°C, the molar ratio of oil:methanol = 1:6, reaction time (5,10,15,20,25,30 minutes), and the mass of co-solvent (0,5,10,15%wt). The first stage of the research was to make co-solvent, then proceed with the transesterification reaction by adding co-solvent which was carried out according to the research operating conditions. The optimum condition of the study was obtained at reaction time 30 minutes and the addition of co-solvent 5%, with yield 97.4171%. The density of FAME 0.88 g/mL and the concentration of FAME 99.963% which complied with SNI 7185-2015. The simulation results of ChemDraw for components of triglyceride+methanol+NaOH+co-solvent obtained kinetic energy 3479.0264 kJ/mol and dipole moment 43279.8007 debyes.

Keywords: transesterification; co-solvent; methyl esters; kinetic energy; dipole moment

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INTRODUCTION

The process of making biodiesel that is often carried out is the transesterification process because it can produce a relatively high yield. In the transesterification process of *Jatropha* oil with NaOH catalyst, yield 98% at 90 minutes reaction time with a reaction temperature of 60°C (Chitra *et al.*, 2005). The constraint in the transesterification process is the limited solubility of the reactants, namely triglycerides and methanol. The transesterification process with a

base catalyst has a slow reaction rate and often stops before complete conversion is achieved (Boocock *et al.*, 1996). Triglycerides are non-polar while methanol is polar so it is difficult to mix. One way to overcome the limited solubility of reactants in the transesterification process is by adding a co-solvent such as a tetrahydrofuran (Van Gerpen *et al.*, 2004).

Several studies of the transesterification process using co-solvent have been carried out. In transesterification of soybean oil with NaOH catalyst

1% wt., yield $98.35 \pm 0.75\%$ at molar ratio DEE: methanol = 0.5:1, oil: methanol molar ratio = 6:1, mass of adsorbent bentonite 20%, reaction temperature 34°C and agitator rotation of 500 rpm (Wu *et al.*, 2016). Transesterification of wasted cooking oil obtained a yield of 72.7% at a mass ratio of acetone: methanol = 1:2, a molar ratio of oil: methanol = 12:1, a reaction time of 15 minutes, a reaction temperature of 27°C and a stirring speed of 600 rpm (Julianto and Nurlestari, 2018). Transesterification of soybean oil with CaO catalyst 3% wt., obtained 98% yield at mass of co-solvent acetone 20%, molar ratio of oil: methanol was 6:1, the reaction time of 2 hours and reaction temperature (Laskar *et al.*, 2020). Transesterification of soybean oil with a catalyst mass of CaO 30 mg, obtained a concentration of 99% FAME in an iso-propanol co-solvent mass of 14.5%, and oil: methanol molar ratio = 20:1, a reaction time of 6.5 minutes and a reaction temperature of 65°C (Chueluecha *et al.*, 2017). Transesterification of palm oil with 5% wt. CaO catalyst obtained a yield of $98.5 \pm 1.5\%$ at a mass of 10% tetrahydrofuran co-solvent, a molar ratio of oil: methanol = 12:1, a reaction time of 90 minutes and a reaction temperature of 65°C (Roschat *et al.*, 2016). In transesterification of soybean oil with 1% wt. NaOH catalyst, the yield was 92% at a reaction time of 30 minutes, the reaction temperature was 50°C , the mole ratio of oil: methanol = 1:3 and mass of co-solvent diesel oil was 30% (Parida *et al.*, 2017).

Transesterification with co-solvent was also carried out under supercritical conditions. Supercritical transesterification of triglycerides from spirulina microalgae with CO_2 co-solvent, yield increased from 46% to 72% at 300°C and operating pressure of 20 MPa when the amount of CO_2 increased from 0.0005-0.003 g CO_2/g methanol (Tobar and Nunez, 2018). Transesterification of supercritical palm oil with acetone as co-solvent, obtained 86.3% yield at 20% co-solvent mass, oil: ethanol molar ratio = 23:1, reaction time 3.5 minutes, reaction temperature 350°C and operating pressure 8 MPa (Akkarawathkoosith *et al.*, 2019). In the transesterification process with co-solvent that has been carried out, the co-solvent used is relatively expensive and requires a separation process at the end of the reaction so that it adds to the length of the stages of the biodiesel manufacturing process (Daryono and Sinaga, 2017). An alternative co-solvent that is cheap and without a separation process at the end of the reaction is methyl ester which is a product of the transesterification reaction.

Several studies of the transesterification process using co-solvent methyl ester have been carried out. Transesterification of soybean oil with 0.8% wt. KOH catalyst, obtained a concentration of FAME 86% at a reaction time of 2 hours, a reaction temperature of 60°C , a mass of 10% FAME co-solvent, a stirring speed of 100 rpm and a mole ratio of oil: methanol = 1:6 (Park *et al.*, 2009). Transesterification of wasted cooking oil with a

catalyst $\text{SO}_4^{2-}/\text{SnO}_2\text{-SiO}_2$ 6% wt., obtained a yield of 88.2% at a reaction time of 1.5 hours, a reaction temperature of 150°C and a mole ratio of oil: methanol = 1:15 (Lam and Lee, 2010). In the transesterification of jatropha oil with NaOH catalyst, it obtained a yield of 88% at a reaction time of 30 minutes, a reaction temperature of 240°C , a mole ratio of oil: methanol = 1:6 and a mass of 15% FAME co-solvent (Sakthivel *et al.*, 2013). In transesterification of cottonseed oil with 1% wt. KOH catalyst, FAME concentration of 99.2% was obtained at 30 minutes reaction time, reaction temperature of 40°C , the mole ratio of oil: methanol = 1:4.5 and mass of co-solvent methyl oleate 34% (Le *et al.*, 2018). In the transesterification of canola oil with 1% wt. KOH catalyst, the yield was 97% at the reaction time of 1 hour, the reaction temperature was 30°C , the mole ratio of oil: methanol = 1:6 and the mass of co-solvent FAME was 20% (Nguyen *et al.*, 2020).

Research on the transesterification process with NaOH catalyst using FAME co-solvent that has been carried out has not yet obtained satisfactory yields in terms of co-solvent mass, reaction time, catalyst mass and reaction temperature. In transesterification of jatropha oil with 15% FAME co-solvent, the yield was 87% with 3% NaOH catalyst mass, reaction time of 30 minutes and reaction temperature of 240°C (Sakthivel *et al.*, 2013). This research uses 0.8% NaOH catalyst mass and a reaction temperature of 60°C , thereby minimizing operating costs. The research aimed to find the optimum conditions for the transesterification process of palm oil with NaOH catalyst and methyl ester co-solvent in terms of yield, physical properties of methyl ester and reactant solubility based on dipole moment.

MATERIALS AND METHODS

Materials

The materials used were palm oil, hydrochloric acid (Sigma Aldrich, 37%), ethanol (Sigma Aldrich, 96%), indicator PP, potassium hydroxide pellet (Riedel-de Haen, 99%), methanol (MERCK, 99.9%), acetone and sodium hydroxide pellet (Riedel-de Haen, 99%).

Raw Materials Preparation

Palm oil was analyzed for FFA and moisture content. The requirements for biodiesel raw materials are %FFA <1% and maximum water content of 1% (Van Gerpen *et al.*, 2004). If % FFA 1%, then an esterification reaction is carried out with an acid catalyst, but if FFA <1%, a transesterification reaction can be carried out directly. If the water content is >1%, then heating is carried out at a temperature of 110°C until the moisture content meets the requirements.

Moisture Test

Weighing 10 grams of oil then put in the oven at a temperature of $104\text{-}106^\circ\text{C}$ for 30 minutes, after

that it is cooled in a desiccator to reach room temperature to determine the water content of the oil using equation (1):

$$\text{Moisture (\%)} = \frac{\text{initial weight} - \text{final weight}}{\text{initial weight}} \times 100\% \quad (1)$$

FFA Test

Weighing 20 grams of oil and heat the oil to a temperature of 65°C. Add 50 mL of 96% ethanol and 3 drops of PP indicator into the *Erlenmeyer*, cool the solution to room temperature then titrate the solution with 0.1 N KOH solution until the color changes to pink. Record the required titration volume and to find out %FFA is calculated using equation (2):

$$\% \text{ FFA} = \frac{\text{ml KOH} \times \text{N KOH} \times \text{MW of fatty acid}}{\text{weight of sample} \times 1000} \times 100\% \quad (2)$$

Making of FAME Co-solvent

The transesterification reaction was carried out by weighing 250 grams of palm oil, mass of NaOH catalyst 1% wt. oil and oil: methanol molar ratio = 1:6. Palm oil was put in an *Erlenmeyer* and heated to a temperature of 60°C. Methanol and NaOH catalyst was put in a three-neck flask, stirred and heated to a temperature of 60°C. After the palm oil reaches a temperature of 60°C, it is put into a three-neck flask which has been equipped with a condenser containing methanol and a NaOH catalyst which has also reached a temperature of 60°C. The three ingredients were reacted with a stirring speed of 100 rpm, a reaction time of 1 hour and a reaction temperature of 60°C. The reaction product was then added with HCl 1 N to pH 7 to neutralize the catalyst and a separating funnel was added for 12 hours to form 2 layers. The top layer is FAME then the methyl ester concentration is analyzed by GC.

Palm Oil Transesterification Reaction with Co-solvent FAME

The transesterification reaction was carried out by weighing 250 grams of palm oil, mass of NaOH catalyst 0.8% wt. oil and oil: methanol molar ratio = 1:6. Palm oil and co-solvent FAME (0, 5, 10, 15% wt. oil) were added to *Erlenmeyer* and heated to 60°C. Methanol and NaOH catalyst were added to a three-neck flask, stirred and heated to a temperature of 60°C. After the palm oil and co-solvent reached a temperature of 60°C, they were put into a three-neck flask equipped with a condenser containing methanol and a NaOH catalyst which had also reached a temperature of 60°C. The three materials were reacted with a stirring speed of 100 rpm, reaction time (5, 10, 15, 20, 25, 30 minutes) and reaction temperature of 60°C.

After the reaction was completed according to the research time variable, 20 grams of sample was taken and HCl 1 N was added to pH 7 for neutralization of the NaOH catalyst so that the reaction stopped. The sample was then put in a separating funnel and allowed to stand for ± 12 hours to form 2

layers. The top layer is methyl ester, spent of palm oil and other impurities that are included, then distilled at a temperature of ± 110°C to separate the impurities that are still included. The residue from the distillation was then filtered to obtain a clear coloured methyl ester. The methyl ester was then calculated for the density and the value of the acid number and analyzed for the methyl ester concentration using GC. The bottom layer is a mixture of glycerol, spent methanol and the results of the catalyst neutralization reaction. Calculating yield FAME with equation (1):

$$\text{Yield FAME (\%)} = [(Wb - Wc) \times Cb] \times 100\% \quad (1)$$

Where *Wb* is the weight of product (g), *Wc* is the weight of co-solvent FAME (g) and *Cb* is FAME content (%).

Analysis of Density

An empty pycnometer was weighed and its mass recorded. The sample was inserted into the pycnometer and its mass was recorded. Density was calculated by equation (2):

$$\text{Density (g/mL)} = (Wpic1 - Wpic2) / Vpic \quad (2)$$

Where *Wpic1* is the weight of pycnometer fill (g), *Wpic2* is the weight of pycnometer empty (g) and *Vpic* is the volume of pycnometer (mL).

Analysis of Acid Number (Shao and Agblevor, 2015)

0.5-g sample was put into *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 it was pink. The pink colour should last for at least 15 seconds. The acid number was calculated by equation (3):

$$\text{Acid number} = (56.1 \times VKOH \times MKOH) / Ws \quad (3)$$

Where *VKOH* (mL) is the volume of KOH, *MKOH* (M) is the concentration of KOH and *Ws* is the weight of sample (g).

Simulation with ChemDraw Professional 15.0

To determine the effect of the addition of NaOH catalyst and FAME co-solvent on the palm oil transesterification reaction, simulations were carried out with ChemDraw Professional 15.0. The simulation results will get the kinetic energy and dipole moment parameters for each system: triglyceride + methanol, triglyceride + methanol + NaOH and triglyceride + methanol + NaOH + co-solvent FAME. From these parameters, it can be seen how far the influence of NaOH catalyst and FAME co-solvent on the reactivity and solubility of the reactants.

RESULTS AND DISCUSSION

Raw Material Preparation

The results of the analysis of palm oil raw materials obtained 0.12% FFA and 0.05% moisture content, so that palm oil can be directly used for the transesterification process.

Making of Co-solvent FAME

From the results of the GC analysis, FAME co-solvent was obtained with a concentration of 98%.

Palm Oil Transesterification Reaction with Co-solvent FAME

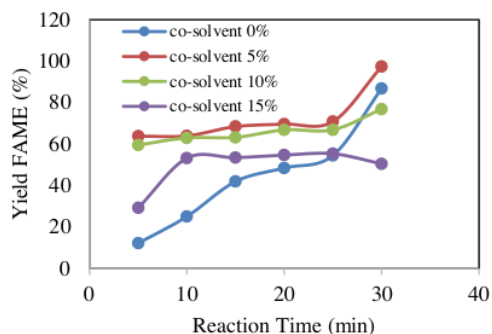


Figure 1. Relation of Reaction Time and FAME Yield with The Variation of Mass Co-solvent

In Figure 1 it can be seen that in general the longer the reaction time, the FAME yield will increase. With the addition of co-solvent FAME 15%, the highest FAME yield of 55.7047% was achieved at a reaction time of 25 minutes and at a reaction time of 30 minutes the FAME yield decreased. This happens because at 25 minutes the reaction has reached equilibrium, so the addition of reaction time will shift the reaction equilibrium towards the reactants because the transesterification reaction is reversible. The increase in FAME yield in the reaction time of 10 minutes to 25 minutes was not very significant, so it can be said that the reaction time of 10 minutes was the optimum condition for the addition of co-solvent FAME 15%. For the addition of co-solvent FAME 0% and 10%, the optimum conditions have not been obtained because the FAME yield continues to increase along with the addition of reaction time. With the addition of 5% co-solvent, the highest yield of 97.4171% is the optimum yield, because if the reaction time is increased to 35 minutes, the possibility is that the yield will increase slightly so it will not have much effect, the yield will remain or it will decrease because the transesterification reaction is reversible.

In the palm oil transesterification process with mass of NaOH catalyst 0.8% wt, the optimum FAME yield of 97.4171% was obtained at a reaction time of 30 minutes with the addition of 5% FAME co-solvent, where this result is quite far when compared to the transesterification process without the co-solvent that obtained the highest FAME yield was 86.7834% at a reaction time of 30 minutes. The more

FAME co-solvent is added, the faster the reaction reaches equilibrium. This happens because the added FAME co-solvent is a product of the reaction itself so that if the equilibrium reaction has been reached, the reaction will quickly shift towards the reactants. By shifting the reaction towards the reactants, the FAME yield obtained decreases, because the formed FAME will be hydrolyzed into fatty acids again (the transesterification reaction is reversible) (Eevera *et al.*, 2009; Daryono *et al.*, 2021).

Analysis

From Table 1, it is known that the density values for all research variables meet the biodiesel standard of SNI 7182-2015, namely 0.85-0.89 g/mL. The biodiesel standard SNI 7182-2015 for the acid number is a maximum of 0.5 mg-KOH/gr. Several research variables did not meet the biodiesel standard for acid number. This happens because during the reaction there is contact between fatty acids with air and water which may be involved so that oxidation and hydrolysis reactions occur to form free fatty acids. The longer the reaction time, the greater the possibility of reactions between fatty acids and other impurities forming free fatty acids. The FAME concentration based on the biodiesel standard SNI 7182-2015 is at least 96.5%.

Table 1. Data on Density, Acid Number and Concentration of FAME

Co-solvent (%)	Time of Reaction (min)	Density (g/mL)	Acid Number (mg KOH/g)	Concentration of FAME (%)
0	5	0.88	0.42	29.7855
	10	0.87	0.56	49.2382
	15	0.88	0.56	82.0302
	20	0.88	0.56	86.7423
	25	0.88	0.56	98.3922
5	30	0.88	0.7	98.0732
	5	0.89	0.28	98.5139
	10	0.89	0.28	98.7344
	15	0.87	0.42	98.1862
	20	0.88	0.56	97.9816
10	25	0.88	0.56	98.501
	30	0.88	0.56	99.963
	5	0.89	0.28	98.0818
	10	0.87	0.42	98.2011
	15	0.87	0.42	97.8281
15	20	0.87	0.42	99.9648
	25	0.87	0.56	97.0489
	30	0.89	0.7	97.5191
	5	0.88	0.42	91.0855
	10	0.88	0.42	92.9215
15	15	0.87	0.42	93.288
	20	0.88	0.42	99.6471
	25	0.88	0.56	97.2464
	30	0.88	0.56	91.4921

Several research variables did not meet the biodiesel standard for FAME concentration. This is because at the time of the reaction FAME has not been formed optimally and there are still many unreacted triglycerides so that the concentration of FAME obtained is small.

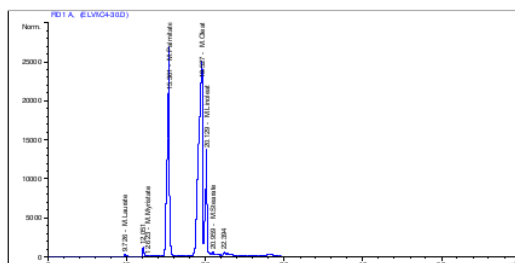


Figure 2. GC Chromatogram for Transesterification with FAME Co-solvent 5% and Reaction Time 30 min

Figure 2 shows the chromatogram of the results of the GC analysis for FAME formed in the transesterification reaction of palm oil with FAME co-solvent 5% and reaction time 30 minutes which is the optimum research condition.

Table 2. Composition of FAME for Transesterification with FAME Co-solvent 5% and Reaction Time 30 minutes

FAME	Composition (%)
Methyl laurate	0.0421
Methyl miristate	0.0298
Methyl palmitate	6.5206
Methyl oleate	92.6177
Methyl linoleate	6.4626
Methyl stearate	0.1435

Table 2 shows the composition of FAME formed in the transesterification reaction of palm oil with 5% FAME co-solvent and reaction time 30 minutes which is the optimum research condition. The FAME composition formed is identical to the fatty acids that make up palm oil triglycerides, namely methyl palmitate, methyl oleate, methyl linoleate and methyl stearate (Marlina *et al.*, 2020a).

Table 3 shows a significant difference in the kinetic energy and dipole moment of the reactants before and after the addition of NaOH catalyst and FAME co-solvent. The higher the kinetic energy, the more reactive the compound (Marlina *et al.*, 2020b). If the compound is more reactive, the greater the collision fraction between the reactants, so the reaction speed will also be greater.

Table 3. Data on Kinetic Energy and Dipole Moment of Simulation Results from ChemDraw Professional 15.0

Component	Kinetic Energy (kJ/kmol)	Dipole Moment (debye)
TG + methanol	2764.4168	4.1036
TG + methanol + NaOH	3388.7315	8054.5281
TG + methanol + NaOH + FAME	3479.0264	43279.8007

In addition to the addition of a catalyst and co-solvent, the collision fraction of the reactants is also affected by stirring and reaction temperature. The amount of kinetic energy is also influenced by the dipole moment parameter. The greater the dipole moment, the higher the molecular interactions within the components (Waluyo *et al.*, 2020).

One of the functions of the catalyst in the transesterification process is to increase the solubility of the reactants involved in the reaction. From the table, it is known that the addition of NaOH catalyst in the transesterification reaction causes the solubility of the reactants, namely triglycerides and methanol to increase. It is known from the increase in the dipole moment before and after the addition of NaOH catalyst, from 4.1036 debyes to 8054.5281 debyes. The more dissolving of the reactants, the faster the transesterification reaction will occur. The addition of co-solvent will increase the solubility and dipole moment (Waluyo *et al.*, 2020). Table 3 shows that there is a significant difference in the value of the dipole moment in the component before and after the addition of FAME co-solvent, namely from 4.1036 debyes to 43279.8007 debyes. The increase in the value of the dipole moment is also quite significant for the reactant + NaOH and reactant + NaOH + co-solvent, namely from 8054.5281 debyes to 43279.8007 debyes. The increase in the dipole moment will accelerate the transesterification reaction time.

CONCLUSION

The addition of FAME co-solvent will increase the kinetic energy and dipole moment of the reactants so that it will speed up the transesterification reaction time. The greater the kinetic energy, the more reactive the components and increase the collision fraction. The greater the dipole moment, the greater the solubility of the reactants, thus accelerating the reaction. From the simulation results of ChemDraw Professional 15.0 for triglyceride + methanol + NaOH + co-solvent FAME components, the kinetic energy is 3479.0264 kJ/mol and the dipole moment is 43279.8007 debyes. The optimum condition of the research was the palm oil transesterification process with NaOH catalyst mass of 0.8 wt%, the reaction time of 30 minutes and the addition of 5% FAME co-solvent, with FAME yield of 97.4171%. At optimum conditions, the FAME density value was 0.88 g/mL

and the FAME concentration was 99.963%, which complied with SNI 7185-2015.

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