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Transesterification of Palm Oil with NaOH Catalyst Using Co-solvent Methyl Ester

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Abstract : Palm oil is very potentially for turned in to biodiesel. In the process of making biodiesel with transesterification conventional required a long time because the reaction takes places in two phase. Adding the co-solvent can be a solution to makes the reaction time shorter. Methyl ester can be used as a co-solvent to makes the reaction in single phase between the oil and methanol so decrease the reaction time in biodiesel process. Methyl ester as a co-solvent is not need to separate after the last process because methyl ester is the main product of the reaction. The purpose of this research is to find out the effect of addition of co-solvent methyl ester and catalyst NaOH to produced the biodiesel from palm oil which has the good standard. According to the result was obtained that the best results of this process achieved at mass of co-solvent of 15% and reaction time of 20 minutes with mass of FAME 28.06 gr. Methyl ester density and acid value regards with SNI 04-7182-2006.

Key words : palm oil, transesterification, co-solvent, methyl ester, biodiesel.

Introduction

Biodiesel is an alternative energy that has been developed extensively to reduce dependence on fuel oil (BBM). Biodiesel is a fatty acid methyl ester produced from a chemical process between vegetable oil and alcohol. The process is mostly done for the manufacture of biodiesel is transesterification reaction, because it is easy and can produce methyl ester is quite high. The main problem in the transesterification reaction is oil solubility in methanol is very low, in which the reaction occurs in the methanol phase. To achieve single-phase reaction system takes a long time. One way to overcome the limitations of the mass transfer is reaction of one phase by adding a co-solvent¹. With the addition of co-solvent, the reaction becomes faster, the reaction temperature can be lower and the ratio of materials: methanol can be minimized. In the research that has been done, the co-solvent used in the reaction transesterification such as THF, DEM, n-hexane and diethyl ether are relatively expensive and necessary process of separation at the end of the reaction by heating. This will certainly add to the cost of biodiesel production itself. In addition, by using methyl ester as a co-solvent will save on production costs because it does not need a co-solvent separation process at the end of the reaction.

Transesterification of castor oil get a yield of 94.5% at the reaction temperature 60°C, the reaction time of 60 minutes, the mass of methanol 20% by weight oil, the mass of NaOH 1%, and biodiesel produced meets

the standard BIS (Bureau of Indian Standards)². Conversion gain 97.85%³ in the transesterification reaction of sunflower seed oil with optimum conditions were 1 hour reaction time, reaction temperature of 60°C, the mole ratio of oil:methanol = 1:6, and the mass of KOH = 1% by weight oil³. Co-solvent is a substance that serves to increase the solubility of the oil in methanol. With the increased solubility of the oil in methanol, the reaction of one phase can be formed so that the methyl ester will be formed more quickly. Co-solvent soluble in alcohol, fatty acids and triglycerides⁴.

Transesterification of waste cooking oil, get a 95% yield and density of the methyl ester of 876.2 kg/m³ at a ratio molar of oil:methanol = 1:4, the catalyst NaOH as much as 1% by weight oil, the reaction temperature 60°C, the stirring speed 600 rpm and reaction time 1 hour⁵. Get a 85% yield in transesterification waste cooking oil with NaOH catalyst⁶. Get a 85% yield in the transesterification of Jatropha oil at a molar ratio of oil:methanol = 1:4, the weight of NaOH catalyst 0.325 g, the reaction temperature of 65°C, reaction time of 75 minutes and the volume of oil 15 mL⁷. In the study of biodiesel obtained with a density of 0.870 g/cm³ and the acid value 0.413 mg KOH/g⁷.

The transesterification of palm oil with a microwave, get a yield of 97.06% at a frequency of 2 times the material entry with a reaction time of 10 minutes, the catalyst KOH 1% by weight oil, the molar ratio methanol:oil = 6:1 and power 180 W⁸. Density biodiesel produced meets SNI 04-7182-2006 ie from 0.85 to 0.89 g/mL⁸. Get a 97% conversion at a molar ratio methanol:oil = 1:9, weight CaOnano particle catalyst impregnated on zeolite 15%, waste cooking oil volume of 500 mL, the reaction time of 6 hours, and the reaction temperature 70°C⁹. In the study of biodiesel obtained with a density of 946.73 kg/m³. Transesterification of algae oil with CaOnano catalyst of egg shells and gain a conversion of 96.3% and the acid value of 1.25 mg KOH / g at the reaction temperature of 55°C, the mole ratio of methanol:oil = 1:9, the weight of the catalyst 1.25% by weight oil¹⁰.

Table 1 Fatty acid composition of palm oil¹¹

Fatty acid	% Weight
Myristic acid	1.1
Palmitic acid	42.5
Stearic acid	4.2
Oleic acid	41.3
Linoleic acid	9.5
Caprylic acid	0.8
Capric acid	0.5
Lauric acid	0.3
Palmitoleic acid	0.2
Heptadecanoic acid	0.1
Linolenic acid	0.3
Arachidic acid	0.3

Get a concentration of FAME of 86% by mole ratio of oil:methanol = 1:6, KOH 0.8% wt., the reaction temperature of 70°C, reaction time of 2 hours, stirring speed of 100 rpm and a co-solvent FAME 10% wt. oil¹². Get a yield 88.2% on the transesterification of waste frying oil with FAME co-solvent in the reaction time of 1.5 hours, the reaction temperature 150°C, the mole ratio of oil:methanol = 1:15, and the catalyst SO₄²⁻/SiO₂SnO₂ 6% wt¹³. In the transesterification of castor oil get a concentration of 95.5% at the reaction temperature of 60°C, the catalyst CH₃ONa 1% wt., a co-solvent n-hexane 15% wt., reaction time of 2 hours, the mole ratio of oil:methanol = 1:6, and stirring speed of 600 rpm¹⁴.

In situ transesterification of mahogany seed oil with a co-solvent n-hexane, yield 88.18% gain in the mass ratio of seed:methanol:n-hexane = 1:3:4 and a reaction time of 4 hours¹⁵. Density methyl ester obtained also regards SNI 04-7182-2006 with limits 0.85 to 0.89 g/mL¹⁵.

Table 2 Indonesian specifications for biodiesel (SNI 04-7182-2006)

No.	Property	Unit	Limits
1.	Density @ 40°C	kg/m ³	850 - 890
2.	Kinematic viscosity @ 40°C	mm ² /s (cSt)	2.3 - 6.0
3.	Ester content	% . wt.	Min. 96.5
4.	Cetane no.	-	Min. 51
5.	Flash point, closed cup	°C	Min. 100
6.	Cloud point	°C	Max. 18
7.	Acid value	mg KOH/g	Max. 0.8

Experimental

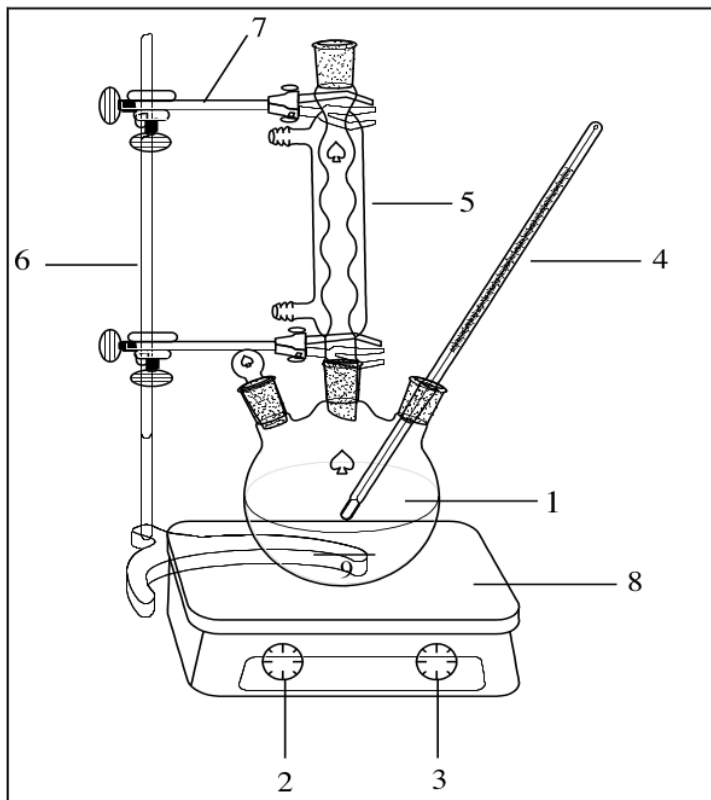
Preparation of Co-solvent FAME

- Palm oil packaging FFA analysis. If the FFA > 2% esterification reaction was carried out but if FFA < 2% can be directly carried out the transesterification reaction.
- Transesterification reaction was carried out with the oil weighing 250 grams, the catalyst NaOH 1% by weight oil, stirring speed of 100 rpm, 1 hour reaction time, reaction temperature of 60°C, and the molar ratio of oil:methanol = 1:6.
- The reaction product was added 1 N HCl to neutralize the catalyst and inserted separating funnel for ± 12 hours to separate into two layers.
- The top layer was FAME used as a co-solvent.

Transesterification of Palm Oil with Co-solvent FAME

- Palm oil as much as 250 grams was included in the three-neck flask which was equipped with condenser. Furthermore, the transesterification reaction of oil with the addition of co-solvent FAME and stirring with a magnetic stirrer at 100 rpm.
- Variable fixed research include catalyst NaOH 1% wt. oil, stirring speed of 100 rpm, the reaction temperature of 70°C, the concentration of methanol 99.9%, the concentration of co-solvent FAME 98.02% and the molar ratio of oil:methanol = 1:6.
- The independent variables were reaction time (5, 10, 15, 20, 25, 30 minutes), and the mass of co-solvent FAME (0, 5, 10, 15% mass of oil).
- After the reaction was completed in accordance variable time of the study, as many as 30 gram sample was taken and added HCl 1 N to pH 7 to stop the reaction. Result of reaction was included in the separating funnel and allowed to stand for ± 12 hours in order to form two layers.
- The top layer was a methyl ester, unreacted oil and other impurities are entrained, then distilled at a temperature of ± 100°C to separate the impurities.
- The bottom layer was a mixture of glycerol, residual methanol, the remainder of the catalyst and the results of the neutralization reaction.
- Upper layer distillation was residue then filtered to obtain crude methyl ester colored clear. Crude methyl ester was weighed and calculated density and the acid value.

Equipment description



Explanation :

1. Three neck flash
2. Button for temperature
3. Button for speed stirrer
4. Termometer
5. Condenser
6. Support
7. Stative
8. Hot plate
9. Magnetic Stirrer

Figure 1 Transesterification Reactor

Results and Discussion

From the analysis and calculation of the data obtained mass, density and acid value of the methyl ester.

Table 3 Results of % FFA and moisture content of palm oil early

Property	Value (%)
% FFA	0.124
Moisture Content	0.0496

From table 3 the data obtained on the analysis of raw materials, namely oil palm with % FFA < 2% and moisture content < 1%, so it can be done directly transesterification process¹⁶.

Table4 Result of density, mass and acid value of methyl ester

Mass of Co-solvent(% wt. oil)	Time of Reaction (min)	Density (ρ) (g/ml)	Mass of FAME (gr)	Acid Value (mg KOH / g sample)
0	5	0.88	16.68	0.28
	10	0.89	17.23	0.28
	15	0.89	17.49	0.28
	20	0.88	17.84	0.28
	25	0.88	18.56	0.28
	30	0.88	19.62	0.28
5	5	0.89	11.98	0.28
	10	0.89	15.21	0.42
	15	0.88	16.02	0.42
	20	0.88	18.98	0.42
	25	0.88	26.74	0.42
	30	0.88	27.08	0.42
10	5	0.89	19.32	0.56
	10	0.88	20.08	0.56
	15	0.89	21.15	0.7
	20	0.88	26.80	0.7
	25	0.89	26.87	0.7
	30	0.88	27.10	0.42
15	5	0.88	15.88	0.7
	10	0.89	17.47	0.28
	15	0.88	21.58	0.56
	20	0.89	28.06	0.28
	25	0.89	28.37	0.56
	30	0.89	28.66	0.28

From table 4 showed different each study variable. The result of the calculation of density values obtained that meet SNI 04-7182-2006 with limits 0.85 to 0.89 g/mL. The result of the calculation the acid value was also obtained grades that meet SNI 04-7182-2006 with max. 0.8 mg KOH/g. Mass crude methyl ester was obtained vary in each study variable, but generally with the longer reaction time and a growing mass of co-solvent was added then the mass of methyl esters also will further increase. In the mass of co-solvent 0% FAME mass obtained 19.62 grams of the reaction time of 30 minutes. In the co-solvent mass of 5% FAME mass obtained 26.74 grams on reaction time 25 minutes. In the co-solvent mass 10% FAME mass obtained 26.8 grams of the reaction time of 20 minutes. In the co-solvent mass 15% FAME mass obtained 28.06 grams of the reaction time of 20 minutes.

Conclusion

- The longer reaction time and a growing mass of co-solvent was added then the mass of methyl esters also will further increase.
- The optimum conditions were found in mass 15% co-solvent and a reaction time of 20 minutes was by FAME mass of 28.06 grams, the density of 0.89 g/mL and the acid value of 0.28 mg KOH/g.
- Density of methyl ester was obtained results varied in each study variable, but the result has regards SNI 04-7182-2006 with limits 0.85 to 0.89 g/mL.
- Obtained results acid number was varied in each study variable, but the result has fulfilled SNI 04-7182-2006 with max. 0.8 mg KOH/g.

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