



Biodiesel Production from Kapok Seed Oil (Ceiba Pentandra) Through the Transesterification Process by Using Cao as Catalyst

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I. INTRODUCTION

Nowadays, Indonesia's oil reserves are running low, reaching 4 billion barrels, with a consumption of about 1 million barrels a day then Indonesia's oil reserve will be exhausted the next 13 years maximum. Therefore, the role of renewables and gas development should be increased. One of the alternative energy oil and gas interest is biodiesel. Biodiesel is a promising alternative fuel that can be derived from waste oil, animal fat or vegetable oil that has been converted into methyl esters through transesterification with alcohol.

Biodiesel gives less pollution than petroleum fuels and can be re-used without modification to diesel engines. Many materials can be used as a source of biodiesel feedstock, one of them is kapok seed. Kapok seed has the potential to be used as raw material for biodiesel because it has advantages such as containing 40% oil by weight, easy to obtain, and relatively cheap.

Transesterification is the most common method used to produce biodiesel. Transesterification is the reaction of plant oils (triglycerides) with alcohols using

alkaline catalyst to produce biodiesel and glycerol. In the transesterification process, homogeneous alkaline catalysts that commonly used are NaOH, and KOH. One of the disadvantages in the use of homogeneous catalysts in the process of biodiesel production is the catalyst can not be re-used or can not be regenerated, because the catalyst is mixed with oil and methanol, and also the process of separating the catalyst from the product is more complex. The use of this homogeneous catalysts are also not environmental friendly because it requires a lot of water for the separation process, because of that, it is developed using solid catalysts in biodiesel production, such as CaO to overcome the lack of homogeneous catalysts.

II. RESEARCH METODOLOGY

a) Degumming Process

Degumming is a separation process of impurities such as latex or oil-slime. Slime composed of phosphatides, proteins, residues, carbohydrates, water and resin, without reducing the amount of free fatty acids contained in the kapok oil because of the gum will cause the emulsion of soap and would interfere the oil refining process [1]. the oil degumming process using H₃PO₄ kapok 0.1% pa oil volume, the reaction for 30 minutes at a temperature of 70 ° C.

b) Esterification Process

Esterification of Free Fatty Acid (FFA) aims to eliminate the FFA in the raw oil. The FFA will be converted into biodiesel. If the FFA levels are too large, it can cause saponification reaction with the catalyst, therefore FFA levels should be kept up to 1% [2]. Esterification using 1% H₂SO₄ mass of oil pa, methanol pa 1:6 mole ratio with oil. Operating conditions: temperature 60 ° C and reaction time 1.5 hours.

c) Catalyst Calcination Process

The calcination process is carried out before the the transesterification process. Because the CaO catalyst will be poisoned by CO₂ and convert into CaCO₃, thus reducing its activity as a catalyst. CO₂ is removed by calcination process on the CaCO₃ to produce a pure CaO. Calcination is performed at a temperature of 700 ° C for 2 hours..

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d) *Transesterification Process*

Transesterification performed by inserting methanol into a three neck flask with a variable ratio of 1:10, 1:15, 1:20 mol ratio of methanol. Secondly, adding CaO catalyst as many as 7% of the mass of the inlet oil. After a homogeneous mixture, kapok oil is inserted into a three neck flask and heated at a variable temperature 40,50,60°C in 1,2,3 hours of stirring. After the reaction, CaO catalyst is screened by using a filter paper (0.7 µm) and then we separate the FAME formed with glycerol using a glass funnel separator.

e) *Analysis*

Analysis conducted are the analysis of FFA content in the oil using titrimetric methods, analysis of biodiesel density, analysis of viscosity biodiesel using Ostwaldz viscometer, and GCMS analysis to view the content of the biodiesel produced

III. RESULTS AND DISCUSSION

a) *Effect Of The FFA Content On Oil*

Based on the analysis of FFA levels in kapok oil that used as raw material for the biodiesel production, oil content of FFA obtained is 9.317%. FFA value exceeds the maximum amount of FFA content to do the process of transesterification, which is a maximum of 1%. Therefore esterification process should be carried out.

Levels of FFA in the oil feedstock should be measured every period of time due to increased levels of FFA in the oil along with storage time. Factors that affect the speed increased levels of FFA in oil feedstock include temperature and humidity, therefore it is important to put the oil on the conditions of low humidity and low temperatures to prevent growth of FFA that resulting decreased efficiency and yield process.[3].

b) *Density Analysis Results*

The density is the weight per unit volume ratio. These characteristics related with calor value and the power generated by diesel engines per unit volume of fuel. If biodiesel has a density exceeding the provisions, the incomplete reaction will occur at the conversion of vegetable oil. Biodiesel that below the quality, should not be used for diesel engines because of will increase emissions, and cause damage to the machine [4].

From the analysis of all variable, according to the results obtained with the standard biodiesel according to SNI 04-7182-2006, which is the density still in the range 0.850 to 0.890 g/cm³.

c) *Viscosity Analysis Results*

Yield of biodiesel or Fatty Acid Methyl Ester (FAME) produced can also be estimated through the viscosity of biodiesel rates. Conversion of triglycerides into methyl esters through the transesterification process resulting a reduce on molecular weight of triglycerides and reduce its viscosity. Viscosity is one of

important parameter in the feasibility of using biodiesel in diesel engines [5].

Viscosity is the resistance of the fluid held in a capillary tube againsts the force of gravity which is usually expressed in the time required to flow at a certain distance. If the viscosity is higher, the higher resistance will be. It is very important because it affects the performance of injectors in diesel engines. Fuel atomization is also very dependent on the viscosity, higher viscosity makes atomized fuel into larger droplets with high momentum and have a tendency to collide with the cylinder wall relatively cool. This leads to an increase in deposits and fuel emissions. Instead with low-viscosity fuel will produce a very subtle spray and can not get into the combustion cylinder thus forming the fuel rich zone which led to the formation of soot. Viscosity also related to the viscosity lubrication or lubrication properties of fuel. Relatively high viscosity has better lubrication properties [4].

From the analysis viscosity on the whole variable from the transesterification process the results obtained with the standard biodiesel according to SNI 04-7182-2006, is still in the range 2.3 – 6 Cst.

d) *GCMS Analysis Results*

Table 1 : Library data of 3 best hits, GCMS analysis on Peak Number : 3, time : 16,72 minute, Area : 3197204978, %Area : 22,3 %

No	Content	Qual
1	Hexadecanoic acid, methyl ester	99
2	Hexadecanoic acid, methyl ester	98
3	Hexadecanoic acid, methyl ester	98

Table 2 : Library data of 3 best hits, GCMS analysis on Peak Number : 8, time : 18,70 minute, Area : 10213727390, %Area : 70,71 %.

No	Content	Qual
1	10,13-Octadecadienoic acid, methyl ester	96
2	9,12-Octadecadienoic acid (Z,Z)-, methyl ester	96
3	9,12-Octadecadienoic acid (Z,Z)-, methyl ester	96

Table 3 : Library data of 3 best hits, GCMS analysis on Peak Number : 9, time : 18,82 minute, Area : 485394187, %Area : 3,36 %.

No	Content	Qual
1	Octadecanoic acid, methyl ester	99
2	Octadecanoic acid, methyl ester	99
3	Octadecanoic acid, methyl ester	98

From the GCMS Analysis gained the content of methyl ester with % Area as 22,3 % (Hexadecanoic acid / Methyl Palmitic), 70,71%, and 3,36% (Octadecanoic acid / Methyl Stearic). We can conclude that Kapok seed oil can be used as a raw material for biodiesel production.

e) Effect of temperature on biodiesel yield

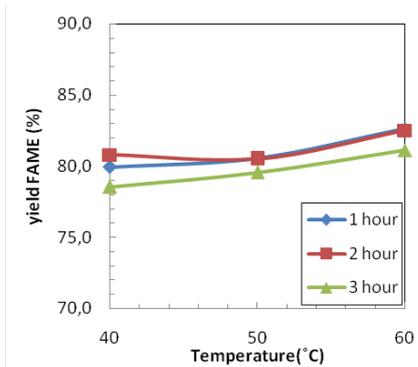


Figure 1 : The effect of temperature on biodiesel yield charts in kapok oil transesterification reaction at a ratio of oil: methanol = 1:10 mol ratio.

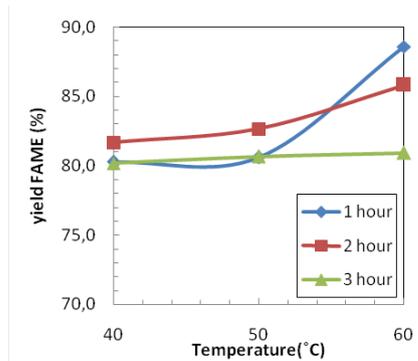


Figure 2 : The effect of temperature on biodiesel yield charts in kapok oil transesterification reaction at a ratio of oil: methanol = 1:15 mol ratio.

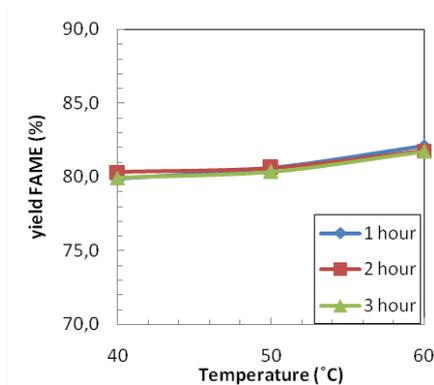


Figure 3 : The effect of temperature on biodiesel yield charts in kapok oil transesterification reaction at a ratio of oil: methanol = 1:20 mol ratio.

The usage of heterogeneous catalysts will make the reaction mixture consisting of three-phase system, oil-methanol-catalyst, where the reaction is slowed down due to the diffusion resistance between the phases. However, the reaction rate can be accelerated at higher temperatures [6]. Effect of temperature on reaction rate can be explained through the theory of chemical reaction kinetics. An increase in temperature will result in increasing fraction of molecules that have a high speed and therefore has a high kinetic rate [7]. Effect of temperature on the transesterification reaction of kapok oil is examined at 40, 50, 60 °C, in the reaction conditions, oil: methanol, 1:10, 1:15, 1:20 mole ratio and reaction time 1, 2, 3 hours. As shown in figure 1,2,3 reaction rate was slow at low temperatures and increases with increasing temperature, in accordance with the laws of kinetics reaction. Optimum yield of biodiesel obtained at a temperature of 60 °C at all the variables in this experiment.

f) Effect of time reaction on biodiesel yield

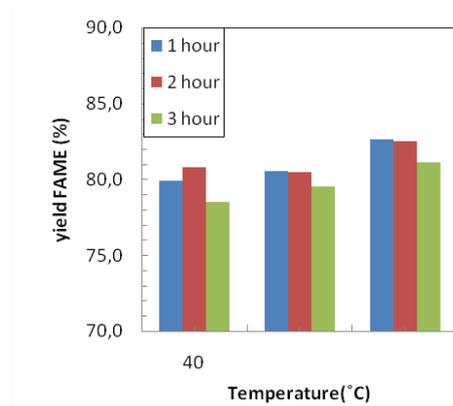


Figure 4 : The effect of reaction time on biodiesel yield charts in kapok oil transesterification reaction at a ratio of oil: methanol = 1:10 mol ratio.

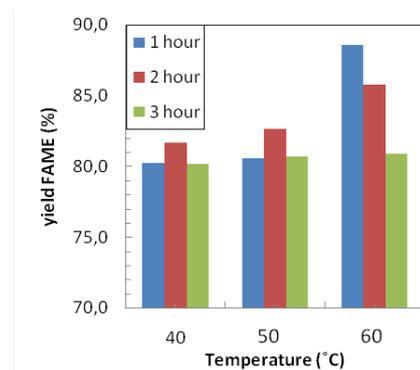


Figure 5 : The effect of reaction time on biodiesel yield charts in kapok oil transesterification reaction at a ratio of oil: methanol = 1:15 mol ratio.

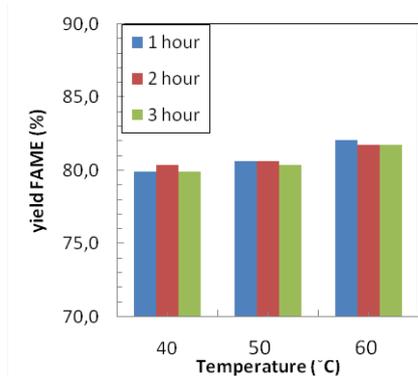


Figure 6 : The effect of reaction time on biodiesel yield charts in kapok oil transesterification reaction at a ratio of oil: methanol = 1:20 mol ratio.

The reaction time will be directly proportional to the percent yield of biodiesel obtained. The rate of conversion increases with the length of reaction time. Diglycerides and monoglycerides at the beginning of the reaction time, will increase and then decrease. In the end, the amount of monoglycerides will be higher than diglycerides. And monoglycerides required for the transesterification reaction [8]. From figure 4,5,6 the effect of reaction time on biodiesel yield produced can be seen. Optimum biodiesel yield obtained on a variable ratio oil: methanol = 1:15 mol ratio, temperature of 60 °C, and reaction time 1 hour, the resulting yield of 88.576%. While the increase in reaction time at 3 hour reaction time, have lowered the yield obtained in all variables, it because CaO catalyst can adsorb the product [9]. CaO catalyst has a tendency to absorb products when the reactants in a lack, thereby it reduce the activity of the catalyst due to the active surface of the catalyst is covered by the absorbed products (monoglycerides, diglycerides, triglycerides, and glycerol) that resulting yield decrease [2].

g) Effect of mol ratio on biodiesel yield

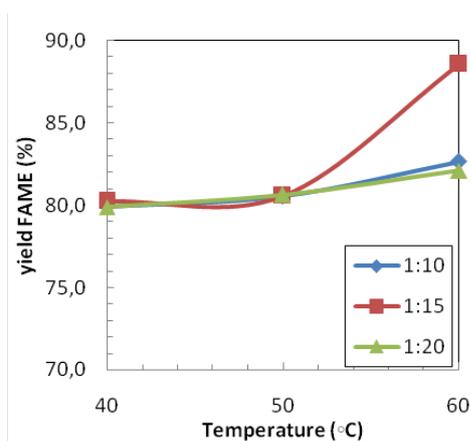


Figure 7 : The effect of mol ratio on biodiesel yield charts in kapok oil transesterification reaction at a ratio of oil: methanol = 1:10 mol ratio.

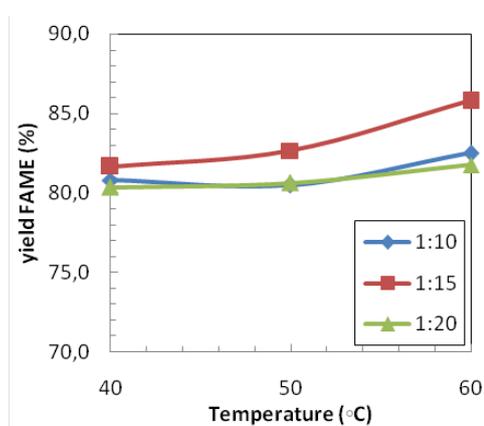


Figure 8 : The effect of mol ratio on biodiesel yield charts in kapok oil transesterification reaction at a ratio of oil: methanol = 1:15 mol ratio.

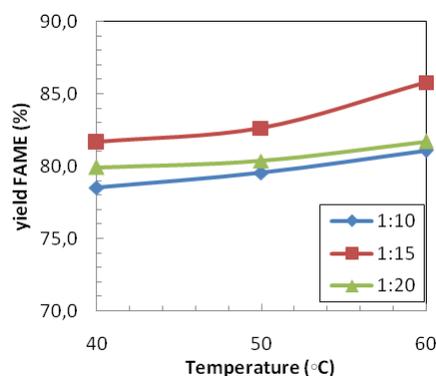


Figure 9 : The effect of mol ratio on biodiesel yield charts in kapok oil transesterification reaction at a ratio of oil: methanol = 1:20 mol ratio.

In stoichiometry, one mole of triglycerides requires three moles of methanol in the transesterification reaction to produce methyl esters (biodiesel). Transesterification is reversible reaction, that the excess methanol is made to run the reaction toward the formation of methyl esters (biodiesel). From the figure 7,8,9 can be seen that the yield increased with increasing molar ratio. As a comparison, biodiesel yield increased from 80.5% to 82.648% (variable 50 °C, time 1 hour) when mole ratio increased from 1:10 to 1:15. But the yield decreased when the mole ratio increased to 1:20 (80.621%). This happens because the catalyst has decreased due to higher content of methanol [6]. Therefore, the optimum mole ratio of oil: methanol for these experiments was 1:15.

h) CaO Catalyst Regeneration

In order to examine the regeneration of the catalyst CaO, CaO catalyst is separated from the reaction mixture using filter paper size of 0.7 µm. After that the catalyst was washed with methanol and dried in an oven at 100 °C for 1 hour [9]. Before the transesterification process, the catalyst recalcined back

at a temperature of 700 ° C for 2 hours. Transesterification process performed on variable ratio oil: methanol = 1: 10 mole ratio, at 60 °C and a reaction time of 1 hour.

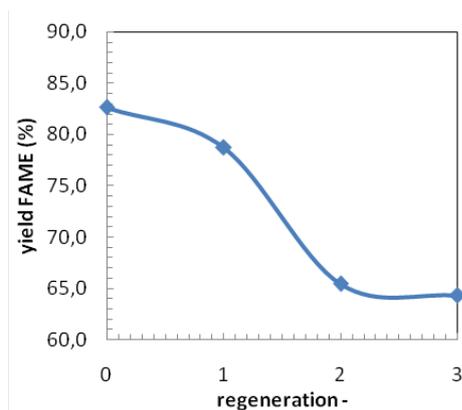


Figure 10 : CaO Catalyst Regeneration Charts.

The results obtained in Figure 10 show that the CaO catalyst can regenerated for 3 times, after being used as much as 3 times the yield of biodiesel have declined by 64.3%, so it is not continue to regenerate again.

IV. CONCLUSION

From this research several conclusions can be made:

1. Based on the results of GCMS analysis can be seen that the kapok seed oil can be used as raw material for biodiesel.
2. The highest levels of FAME produced under conditions of oil to methanol mole ratio 1:15, reaction temperature 60 ° C, and reaction time of 1 hour, amounting to 88.576%.
3. CaO catalyst can be regenerate back 3 times with the smallest yield obtained in the amount of 64.300%.

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