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Comparison Of The Characteristics Of CPO Oil And Waste Cooking Oil To The Influence Of Stirring Speed, Temperature And Reaction Time In The Transesterification

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Abstract *- Transesterification reaction plays an important role in converting vegetable oil or used oil into biodiesel. Reaction process may result in low yield, due to the conversion stage between oil and methanol takes place is not perfect and can lead to low-quality of biodiesel. In the transesterification reaction; mass of catalysts, mass of methanol, FFA value, reaction temperature, reaction time, and stirring speed is a major factor determining the quality of biodiesel produced. This study focused on the influence of reaction temperature, reaction time, and stirring speed on waste cooking oil transesterification reaction. This study focused on the influence of reaction temperature, reaction time and stirring speed on waste cooking oil transesterification reaction. Transesterification reaction takes place at a temperature variation 50, 55, 60, and 65° C, with a variation of reaction time 1, 1.5, and 2 hours, and the use of rapid mixing during the reaction process using a variation of 300, 400, 500, and 600 rpm. Results showed the higher temperatures used, the conversion of biodiesel produced will be higher, for a shorter time. Reactions were carried out at 65°C temperature gives lower yield of methyl ester. Using the stirrer speed is too high will cause the saponification reaction which reduce the yield generated. Use stirring speed is too high will cause the saponification reaction which will reduce the yield generated. Variables that produce the higher yield and quality of biodiesel is the use at 60° C temperature, 1.5 hours reaction time, and 500 rpm stirring speed. The results of these three variables biodiesel, has met SNI the standard and ASTM D 6751 for testing density, viscosity, cetane index, and flash point, but oil CPO has better value compared with waste cooking oil.*

Keywords *- Transesterification, saponification, cooking oil, temperature, rotation, reaction time*

I. INTRODUCTION

Biodiesel is the renewable energy replacing solar. Generally, biodiesel is the fuel of diesel machine which is made by renewable material or it is specifically the fuel of diesel machine consisting of *ester alkyl* from fatty acid. Biodiesel can be made from vegetable oil, animal oil, or the used cooking oil, by converting those materials (tryglicerida) to the methyl ester using a process called transesterification. Since transesterification is an equilibrium-controlled reaction, an excess of alcohol is used to shift the equilibrium forward as shown in Figure 1. Methanol is the most often preferred alcohol because of its affordable price compared to other alcohols. As a result, the operating cost as well as the capital cost of this process is reduced.

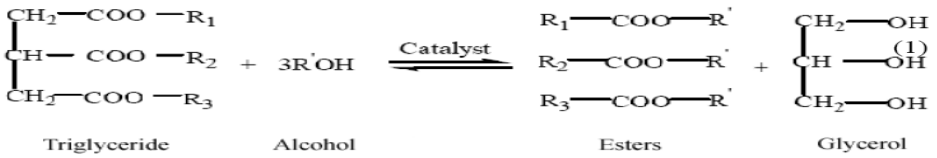


Fig. 1: Transesterification reaction from Tryglicerida

The biodiesel process is quite simple; however, it must be done appropriately and effectively from the view of raw materials, the equipments, and also for the manufacturing process of biodiesel itself, for instances; duration, temperature, quality and quantity of chemical substances used. The carelessness in unit pre-process and biodiesel manufacturing can cause the reducing of biodiesel quality produced and even the failure in manufacturing process. In biodiesel manufacturing process, transesterification reaction and biodiesel washing process are steps which are determined biodiesel yield quality that will be reached. Transesterification process is influenced by molar ratio of between catalyst and cooking oil, molar ratio of methanol with oil, FFA content of cooking oil, duration, temperature and the mixing speed. FFA content of cooking oil is the main factor

determining the success of tansesterification reaction. According to the study by Tiwari (2007) said that FFA content of cooking oil above 1% will decrease the yield of product and increase soap forming, so the process of separating biodiesel and glycerol more complicated [1]. Other studies done by Gerpen (2005) said that the higher of molar ratio between methanol and oil will produce high yield of biodiesel [2]. Furthermore, the study by Hossain *et al* (2010), factor influence in determining the yield of biodiesel content are time, temperature reaction, and mixing speed. In his research, temperature used is 55 ^{o}C, the duration of reaction is 2 hours, and the mixing speed is 250 rpm using used canola oil as feedstock is able to produce the highest yield of biodiesel [3].This study focuses on the influence of temperature, duration and stiring speed in transesterification reaction. The shorter duration of transesterification reaction will cause the failure of transesterification process. Meanwhile, the temperature used in the process, if the temperature is too high, and the mixing speed is too low or also too high, it will distrub the transesterification process.

The study about the biodiesel production use some mixing of vegetable oil and cooking oil is done by Piyanuch Nakpong and Sasiwimol Wootthikanokkhan in 2009 [4]. Vegetable oil consists of jatropha oil, rosella oil, coconut oil, while the comparison between vegetable oil and used cooking oil are 0,03-0,2 v/v. In the process of alkali-catalyst transesterification , methanol is used as feedstock in a reactor equipped with condenser, evaporator, thermometer, mixing large spoon in form of hot metal sheet with magnetic mixing; where all experiment are done in the same speed. In the experiment with the ratio of mixing vegetable oil and used cooking oil has obtained variation methyl ester with the difference of degree comparison is shown Table 1.The analysis characteristics of biodiesel from those mixtures in laboratory which is compared to the Thailand biodiesel standard (B100), it obtained the characteristics to where the condition has been met the standard except for low viscosity in biodiesel production from the mixing of coconut oil and used cooking oil. The result of biodiesel production has higher methyl ester in the minimum standard of B100 is the comparison between mixing used cooking oil and vegetable oil is 0.03 v/v The study done by Vicente *et al* (2004), transesterification reaction in sunflower seed oil with the temperature of reaction 65°C for 4 hours with the mixing speed 600 rpm [5]. In this study Vicente compare the use of four kinds of different catalysts but in the same condition of reaction, which are sodium hydroxide, potassium hydroxide, sodium methoxide, and potassium methoxide. The result of FAME conversion which is obtained for those four catalysts are 86.71%, 91.67, 99.33% and 98.46% orderly. The use of alkaly catalyst in transesterification can cause saponification reaction.

II. METHODOLOGY

2.1 Materials

Materials used in this research such as used cooking oil from frying residue or restaurant and CPO oil from coconut oil as feedstocks of biodiesel production. Methanol 90% is used as reactant in the transesterification reaction and KOH as catalyst in transesterification reaction, while H_2SO_4 as catalyst in esterification reaction.

2.2 Esterification and transesterification reaction.

This study uses the experimental method, instruments are made from some components for instances; heater which consists of two heaters with power 1000 -1200 watt, the mixing motor is matched with using circle inverter, control panel which is required to set the temperature where there is thermocopel, sensor temperature and control system with cycle if the temperature is reached setpoint will switch off the heater and cycle, but in the certain temperature, electricity motor and heater going on, stirring spoon use electricity motor whose power is 3/4 Pk where the spinning cycle can be set by using spinning inverter, control panel which is needed for arranging temperature where thermocopel is, temperature sensor and control system with spinning if the temperature has been reached, control will switch off the heater and spinning but in the specific temperature electricity motor and heater continuing again, stirring spoon in form of large spoon whose has certain oblique angle, the oil material is put on the reactor through cover and is not opened during circle duration of stirring occurred will cause saponification reaction.

2.3 Esterification reaction

Before doing transesterification reaction, esterification reaction process needs to be done in the series of biodiesel production process. Esterification reaction aims to decreasing the FFA content of used cooking oil. Esterification process which is done in this study consists of some steps as follow [6]: first of all used cooking oil (tryglicerida) was heated in reactor until the temperature raise 30-40^{o} C, then add methanol for the amount 20 %-v of oil into the reactor and stir for ± 5 minutes and add acid catalyst (H_2SO_4) for 0,25 %- of oil volume and stir for ± 120 minutes, then the product of esterification reaction is kept until becoming colder then continuing to the following step of transesterification.

2.4 Transesterification reaction

In this process as previously esterification is done, the following is by preparing some steps such as [7]: preparing the materials used to produce biodiesel, pre-washing of waste cooking oil as the main feedstock ; and chemical substances consisting of methanol with molar ratio of methanol and used cooking oil is 1 : 5 as reactant in the process of transesterification and also preparing 5.3 gram KOH / liter of the oil is used as catalyst, then the oil was done pre-heating which aims to omit the amount of water on it. Water existence in oil will cause hydrolisys which turns up soap whose existence can be solid or semi solid and will be very difficult handled. The methoxide solution is prepared by mixing 20 % of methanol from the amount of used cooking oil which is used as alkaly catalyst in form of KOH 5.3 gram/ liter of oil. This mixing is done in the closed equipment, so that it can produce potassium methoxide solution. After methoxide solution is formed, and water content below 1%. The Oil is moved into the reactor to be done the transesterification process using methoxide solution by applying various temperature, time reaction and stirring speed. The product from the transesterification is settled for 24 hours to separate glycerin from metyl ester (biodiesel) perfectly.

2.5 Biodiesel Analysis

The content of FAME in biodiesel can be known using method gas chromatography (GC). The analysis procedure with GC is explained as follows. [8]: Biodiesel sample is taken about 1 g, add benzene-alcohol with mass ratio 1:1 with biodiesel mass sample, both solution are mixed until homogeneous and taken as 0,5 µl for analysis by using the instrument called gas chromatography (GC).FFA determination procedure in used cooking oil and CPO oil are like this: the material are mixed smoothly and in the liquid when are taken as the sample. Weighing the sample for 50 ml hot neutral alcohol and 2 ml phenolphthalein (PP) indicator, titration with 0.1 NaOH solution which has been standardized until the color turns into pink and the color does not change for 30 seconds. Free fatty acid percent is expressed as oleic in the mostly oil and fat. For coconut oil and main coconut oil are expressed as laurat, meanwhile coconut oil is called palmitat. Free fatty acid is expressed as %FFA or as number of acid. Number of acid is the number of KOH used for neutralizing 1 gram. For example;

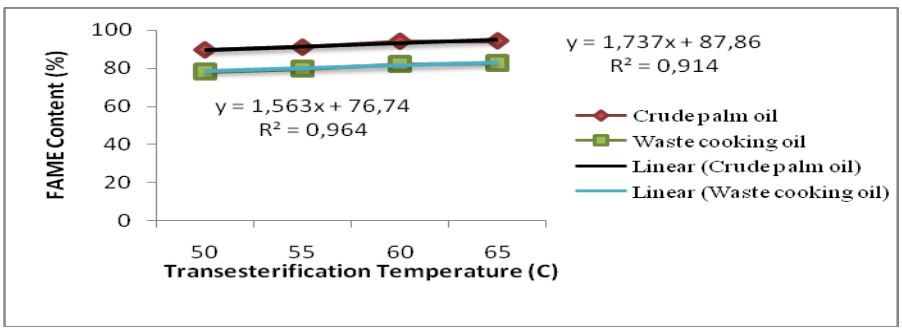
$$\%FFA = \frac{Volume\ of\ NaOH\ (mL)x\ Molecule\ Weight\ of\ Fatty\ Acid}{Weight\ of\ Sample\ x\ 1000} x100\% \quad (1)$$

Free fatty oil is determined as the containing of fatty acid mostly in particular oil. Therefore, free fatty acid is used as parameter for the particular oil. The analysis of density done based on procedure which is suitable with ASTM D 1298, while for the analysis of viscosity done based on procedure which is appropriate to ASTM D 445, then in the cetane index done based on procedure of ASTM D 86, next the analysis flash point done based on the procedure which is suitable with ASTM D 93-02A [9].

III. RESULT AND DISCUSSION

3.1 Characteristics CPO and cooking oil

In this study, it is found the instrument used for transesterification process in form of reactor which is functioned in the biodiesel manufacturing, such as the place where CPO oil conversion, used cooking oil, and others vegetable oil to be *Fatty Acid Methyl Ester* (FAME). For those instrument, in the reactor equipped automatic temperature control, heater, stirring and inverter for setting the mechanical stirrer with temperature control if the certain temperature electricity motor does not work and neither does heater or off. Generally, the transesterification temperature reaction can be about 30 - 65°C (methanol about 65° C). The higher the temperature, the higher the conversion is reached for the shorter period. Reaction done above boiling point of methanol can cause the decreasing the conversion and quality biodiesel which is produced. This happen because of methanol evaporating during the reaction occurs. The influence to the Fame in the used cooking oil and CPO with the temperature 50, 55, 60, 65 ^{O}C is described in the graphic in the figure 2.



In this reaction is also described the reaction time to FAME in the used cooking oil and CPO as the figure 3.

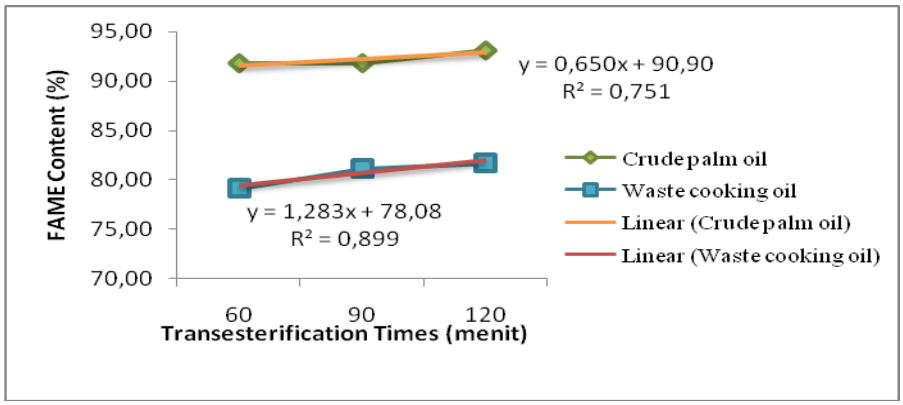


Fig 3: Reaction time to the content of FAME

This study shows that used cooking oil and CPO between stirring rotation with FAME content can be described in figure 4 .

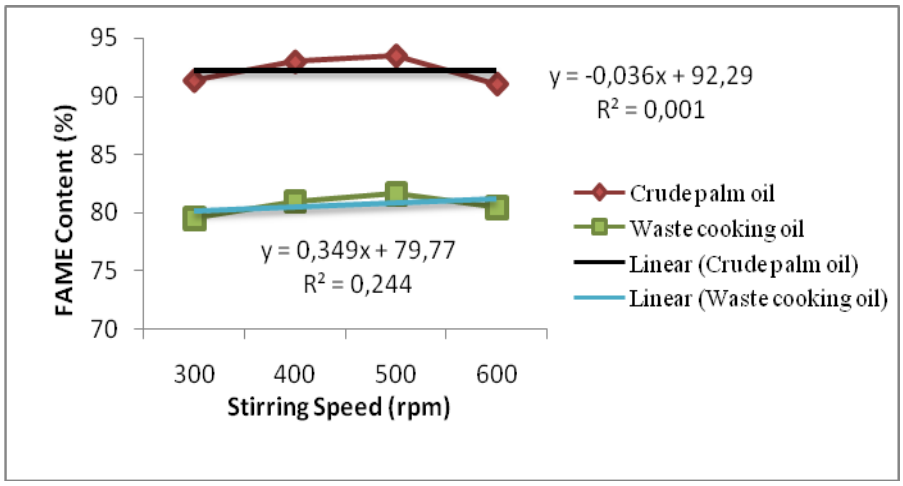


Fig. 4: Stiring speed to the content of FAME

3.2 Discussion

Figure 2 shows that the higher the transesterification reaction temperature, the higher the content of FAME biodiesel produced. The highest of FAME content happens in the temperatur 60°C. This happens because of increasing the temperature, so, the greater the inter-particle collisions, so the reaction goes further faster and greater the reaction constants. Transesterification reaction of CPO oil and waste cooking oil with methanol into Fatty Acid Methyl Ester (FAME) with methanol is an endothermic reaction, so if the temperature of reaction is raised, so the equilibrium will shift to the right / to the product.The increase in reaction rate is due to the increase in the reaction rate constant which is a function of temperature. The higher the temperature, the greater the reaction rate constant. This is according to the equation of Archenius as follows [10].

$\mathbf{k} = A \exp(-Ea/RT)$ (2)

In the transesterification, time of reaction influence to the conversion and FAME content which is produced. In this study, the influent of reaction time of transesterification to the content of FAME produced for both types of oil, is CPO oil and waste cooking oil are shown in figure 3 shows that the longer the time of transesterification reaction, the higher content of FAME biodiesel produced. This is caused the moving molecul in liquid has certain amount of potential energy in their bonds and amount of kinetic energy in their movement. By increasing the reaction time, molecul get the additional opportunity for reacting, colliding each others and changing kinetic energy to potential energy. The longer the time of reaction will increase CPO oil conversion and waste cooking oil into metyl ester (biodiesel) since the chance of colliding among reactants molecules is bigger.Figure 4 shows that optimal stirring produces the higest FAME content by using a stirrer speed of 500 rpm. If the strring speed used in the transesterification is low will cause the homogenization between vegetable oil (CPO oil and waste cooking oil) and methanol is less smooth. And if the stirring speed used is too high will

cause turbulence in the mixing process, where turbulence has high potential to produce soap reaction (saponfication reaction) that will reduce FAME biodiesel content.Stirring done in transesterification reaction will also increase the frequency of collisions between reactant molecules that accelerates the reaction and the reaction occurs perfectly. This is according to the equation of Archenius, the greater the collision occurs, the greater the reaction rate constant values, so in this case, stirring is very essential since the oil-catalyzed methanol is immiscible solution. The characteristics produced in this experiment for the raw material of CPO and waste cooking oil consists of density, viscosity is the characteristic which is influence to the atomization pattern, correlating and combustion performance, cetana index as the close in the number of cetana certain fuel, flash point is a temperature in fuel which is going to be started ignite, all of this are shown in Table 1

Types of Biodiesel	Density (g/ml)	Viscosity (cSt)	Cetane Index	Flash Point (^oC)
Crude palm oil Biodiesel (CPO)	0.8722	5.586	54.11	132.03
Waste cooking oil biodiesel (WCO)	0,8799	5,999	51,91	165,81
Indonesian National Standard	0,850 – 0,890	2,3 – 6,0	Min 48	Min 100

Table 1. Characteritics of CPO and waste cooking oil biodiesel

IV. CONCLUSION

In this analysis of experiment and discussion so that can be summed up as follows: the results shows the higher temperature used, the higher biodiesel conversion produced, for the shorter time. Reaction were carried out at temperature of 65°C cause the decreasing of yield produced. The use of stirring speed which is too high will cause saponification reaction which will reduce the yield generated. Variable produced the best yield and quality of biodiesel is the used of temperature of 60°C, reaction time is 1,5 hours, and the stirring speed is 500 rpm. Biodiesel characteristic from those three variables has been fulfilled Indonesian National Standard (SNI) and ASTM D 6751 standard for density, viscosity, cetana index and flash point.

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