Optimization of Esterification and Transesterification Process for Biodiesel Production fom Used Cooking Oil

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Abstract
Various treatments were carried out to produce biodiesel optimally. This research was conducted to process used cooking oil (UCO) into biodiesel. UCO with high FFA has saponification in the transesterification reaction. Transesterification experiment UCO with % FFA 4.261 and acid number 8.42 mg-KOH/g produced saponification. This experiment was carried out at different temperatures, speeds, and times. It is necessary to carry out initial esterification treatment. Simultaneously the biodiesel synthesis process is carried out by esterification and transesterification processes. The esterification process with 3%wt H_2SO4 catalyst, 1:6 molar ratio of oil and alcohol with temperature treatment of 50 °C, speed of 300 rpm for 60 minutes can reduce % FFA in UCO to 2.115 and acid number 4.208 mg-KOH/g. The transesterification process using a NaOH catalyst of 0.5% wt, the molar ratio of oil and alcohol 1:6 with a temperature treatment of 60°C, a speed of 400 rpm for 60 minutes, can produce biodiesel with an acid number of 0.28 mg-KOH/g and 0.141 of % FFA according to SNI 7182-2015 standard. These results after purification on biodiesel.

***Keywords*:** *Biodiesel, Used Cooking Oil, Esterification, Transesterification*.

1. Introduction

The need for fuel oil is increasing, resulting in a decrease in reserves of petroleum resources. This fact motivates many countries to look for alternative renewable fuel sources, including biodiesel. Much research has been done to make biodiesel. Biodiesel is made to fuel diesel engines derived from vegetable oils. Biodiesel is produced by converting triglycerides to oils obtained from palm oil, soybeans, jatropha, sunflower seeds and other plants. Another material used for biodiesel is UCO (Leung, Wu, & Leung, 2010).

Cooking oil is one of the staple ingredients widely consumed, including in Indonesia. Used cooking oil (UCO) has the potential to be converted into biodiesel. Biodiesel made from UCO for vehicle fuel must have quality characteristics by the requirements (Tsautsos & al, 2019). In Surabaya, UCO is dumped into the ground or sewers as a pollutant. Several

community groups collect UCO from the local waste bank. A waste bank is a campaign to handle waste by buying back trash in terms of a deposit like a banking system (Pariatamby & Tanaka, 2014). Based on survey results from the central waste bank in Surabaya, UCO reaches 4000-5000 litres in a month (February 2020). It has the potential to be used as biodiesel. Biofuel production technology from palm oil and palm kernel oil is one of the national Flagship programs (IESR, 2019).

In general, biodiesel made from UCO is processed by mixing oil with Methanol through a transesterification process. The quality of biodiesel resulting from this process is influenced by factors such as temperature, time, the molar ratio of Methanol, catalyst, stirring, and the content of fatty acids and water in cooking oil (Buchori & Widayat, 2007). Several studies have been conducted to produce optimal biodiesel both in terms of quality and cost. The results of the review of articles related to biodiesel synthesis with the transesterification process obtained the optimum values for several factors; reaction temperature (50°C-60°C), reaction time (>90 minutes), Methanol: oil molar ratio (5.6-7.8:1), Catalyst (NaOH, KOH), mixing speed 200 rpm, 400 rpm, 600 rpm, and 800 rpm. Optimum results at 400 rpm for 60 minutes. Low rotation will result in low biodiesel formation, while high stirring will increase saponification. (Gashaw & Theshita, 2014). The initial filtration treatment of the material will also affect the characteristics of biodiesel. (Setiawati & Edwar, 2012).

Biodiesel is an alkyl ester produced from biological sources such as vegetable oil, animal fat and even used cooking oil. Biodiesel is produced from the conversion of triglycerides to oils obtained from palm oil, soybeans, jatropha, sunflower seeds and other plants. Another material that can be used for biodiesel is UCO (Leung, Wu, & Leung, 2010). Biodiesel can be produced from several synthesis processes. The methods that can be used are direct use and blending, microemulsion process, thermal cracking process, and the most commonly used process are transesterifications (Gashaw & Theshita, 2014) and (Sulaiman, 2016). The direct use and blending method on diesel engines are not recommended and still has the potential to cause engine problems. Because mixing and other chemical modifications are required, the microemulsion method can be used to overcome the problem of high viscosity in vegetable oils. Micro-emulsion is defined as a colloidal equilibrium dispersion of the microstructure of an optically isotropic liquid. The thermal cracking or pyrolysis method is the conversion of substances by heating in the absence of air. Biofuels can be produced from the pyrolysis of vegetable oils. The transesterification method is a chemical reaction involving vegetable oil and alcohol with a particular catalyst. Catalysts are used to increase the reaction rate and yield of biofuels. This method is most commonly used (Gashaw & Theshita, 2014).

The characteristics of biodiesel produced from used cooking oil must meet certain quality or requirements (Tsautsos & al, 2019). Biodiesel from UCO in several southern European countries is identified according to the needs of EN ISO 12937, EN14104 for moisture content and acid content. For physical characteristics such as density, viscosity, flash point, and fog point, it is adjusted to the requirements of EN ISO 12185, EN ISO 3104, EN ISO 3679, and EN 23015. Another reference used to measure the characteristics and needs of biodiesel is SNI 7182-2015. This standard was prepared to establish the quality requirements and test methods

of biodiesel and has an essential role in maintaining the quality of biodiesel marketed domestically to protect consumers producers and support the development of the biodiesel industry (BSN, 2015).

UCO is palm oil that has been used several times. The analysis of the physicochemical properties of used palm oil have shown damage and have changed physicochemical properties such as colour, acid number and water content. Through linear equation regression analysis, cooking oil from traders can be predicted with the frequency of use with 16-54 times of frying (Aminullah et al., 2018). Cooking oils used repeatedly will reduce its quality, including turbidity, high levels of free fatty acids and peroxides, aroma and viscosity (Kusumastuti, 2004).

UCO has different characteristics. UCO collected in several European countries has a free fatty acid (FFA) content of 1.19% with an average water content of 0.19% (Tsautsos & al, 2019). UCO with 4.38% FFA was obtained from cooking oil in India (Gopalakrishnan & al, 2017). In local cooking oil (in Indonesia), the FFA content is 4.2% by weight (Gopalakrishnan & al, 2017), the water content is 0.3%, with FFA 2.3% (Kusumastuti, 2004). In the transesterification reaction, the used cooking oil material has a water content of FFA < 1%. If the material used is more than 1%, an alkaline catalyst is needed to neutralize the FFA (Gashaw & Theshita, 2014).

Vegetable oils must be converted to other forms to decrease viscosity, increase volatility and remove free fatty acids. One way that can be taken is to convert it into alkyl esters (biodiesel) (Hidayati et al., 2017). UCO has the potential as a biodiesel feedstock. This material is easy to obtain and low cost for biodiesel production (Gashaw & Theshita, 2014).

The process commonly used to synthesize UCO is a transesterification reaction. Transesterification is one method to synthesize biodiesel. This reaction is carried out by mixing oil and alcohol to form methyl esters and glycerol with the help of a catalyst. The transesterification method is the most commonly used and low-cost method. Commonly used catalysts are alkaline catalysts such as NaOH and KOH, while acid catalysts are widely used, such as HCl and H_2SO_4. (Leung, Wu, & Leung, 2010)**.**

Transesterification with NaOH catalyst is used to make biodiesel from used cooking oil. Pre-treatment was performed by filtering UCO with 1µm, 5µm, and 16µm size filters. Sodium methoxide solution (NaOH + Methanol) was dissolved in UCO, preheated at a temperature of 50ºC. The cooking oil transesterification process uses a temperature of 65ºC. The optimum yield of biodiesel was obtained at 16µm filter treatment with a time of 65 minutes (Setiawati & Edwar, 2012). The reaction of 8 grams of sodium hydroxide and 400 ml of Methanol dissolved in 2 litres of UCO at a temperature of 60ºC produces biodiesel with better quality than petrol diesel fuel (Ojiego & al, 2014).

83.4% biodiesel and 26.6% glycerol were obtained from the transesterification reaction of UCO at a temperature of 70ºC using a KOH catalyst (Gopalakrishnan & al, 2017). 3% KOH/bentonite was used in the transesterification reaction, which produced a yield of 90.70% biodiesel with a ratio of 1:4 for 3 hours at 60ºC and a stirring speed of 750 rpm (Soetaredjo & al, 2011). The use of calcium oxide (CaO) as a heterogeneous base catalyst was studied to

manufacture biodiesel from UCO. The highest yield, 53%, was obtained when the transesterification reaction was carried out with Methanol: oil ratio of 15:1, the amount of catalyst was 3%, the reaction temperature was 60ºC, and the reaction time was 2 hours with a stirring speed of 600rpm (Hidayati et al., 2017). Catalyst factors, temperature, time and stirring speed are very important in the transesterification process.

A catalyst is a substance that increases the rate of a reaction. The catalyst is not permanently involved in the reaction, so at the end of the reaction, the catalyst is not combined with the reaction product compounds. When the reaction is complete, the same mass of catalyst is obtained. Catalysts can be classified into three major groups, namely homogeneous catalysts, heterogeneous catalysts and enzyme catalysts (biocatalysts). In the transesterification process, catalysts can be classified into basic homogeneous catalysts, homogeneous acid catalysts, alkaline heterogeneous catalysts, heterogeneous acid catalysts, and enzyme catalysts (Sulaiman, 2016). Homogeneous base catalysts generally use KOH and NaOH. These catalysts are very easy to obtain and require low costs. In comparison, homogeneous acid catalysts used such as HCl and H2SO4. Heterogeneous catalysts in solid bases such as CaO are often used, while heterogeneous acid catalysts commonly used are Tungsten oxide, Zn-Cr Oxide, and silica. Several solid catalysts, both basic and acidic, can both be used in esterification and transesterification reactions (Mat et al., 2012). An example of an enzyme catalyst that is often used is Lipase, but the use of enzymes as a catalyst requires a high cost.

The formulation of the problem in this study is how to process waste cooking oil which has a high acid number, to become biodiesel according to quality standards. This study aims to treat waste cooking oil with high acid numbers. This waste is taken from the local waste bank. In this study, an experiment was conducted on the transesterification process directly on the oil, and the second experiment was carried out by pre-treatment with the esterification process followed by the transesterification process.

2. Methodology

This research is an experimental study on the transesterification process of UCO to produce biodiesel. Experiments were carried out with the treatment of several different factors and levels. There are two Experiments. **Experiment I**. Transesterification. Mass of catalyst and molar ratio are fixed variables. Three factors and three levels used a factorial design experiment. The materials used in this experiment are UCO collected from the local waste bank, sulfuric acid (H_2SO_4) 98%, sodium hydroxide (NaOH) 99%, isopropyl alcohol (IPA) 99%, phenolphthalein (PP) 0.1%, potassium hydroxide (KOH) 0.1M and 98% Methanol. The equipment used in the experiment were a magnetic stirrer with heater and speed control, digital scale, stopwatch, thermometer, Erlenmeyer, beaker glass and separator funnel and condenser for reflux. UCO is filtered to remove dirt and other deposits. Initial characteristics of UCO are identified. The second experiment was carried out by giving pre-treatment to the UCO. **Experiment II.** Esterification and Transesterification. Esterification was given before the Transesterification process. Esterification uses two different variables. The molar ratio of oil and Methanol is 1:6 and 1:9, with the weight of the catalyst to oil being 3% and 5%.

Source: Research Document, 2020
Figure 1. Experimental equipment

Experiment I, the molar ratio of oil and Methanol was 1:5. The UCO is directly processed by transesterification by mixing 0.4 gram NaOH and 20 ml of Methanol. Stir in a separate place at a temperature of 50ºC and a speed of 400 rpm until both are dissolved. Carry out the transesterification process by treating different temperatures (50, 60, 70ºC), mixing (300, 400, 500 rpm), and times reaction (30, 60, 90 minutes) according to a predetermined combination. There are 3^3 or 27 experimental combinations without repetition. Each experiment was carried out on 100 ml of cooking oil respectively. The resulting solution was placed in a separating funnel and allowed to stand for 1 hour.

Experiment II. The second experiment was carried out by giving pre-treatment to the UCO. Esterification was given to reduce the % FFA and avoid saponification during the transesterification process. Esterification process using acid catalyst H_2SO_4. The molar ratio of oil and Methanol is 1:6 and 1:9, with a catalyst weight of 3% and 5% by weight of oil. The esterification results are then separated and followed by the transesterification process. The transesterification process uses a NaOH catalyst of 0.5% by weight of oil. The molar ratio of oil and Methanol is 1:6. Each esterification and transesterification process are calculated to yield and acid number. Yield is the ratio of the weight of the processed product to the weight of the oil used.

$$\text{yield} = \frac{\text{weight of product}}{\text{weight of oil}} \text{ x } 100\% \quad (1)$$

The procedure for determining the acid number is as follows. Weight 5gr of oil and put it in a 250ml Erlenmeyer. Then add 20 ml of IPA (Isopropyl alcohol) stirred with a magnetic stirrer at a temperature of 50°C for 30 minutes. After cooling, add 3-4 drops of PP indicator, shaken to mix. Then titrate with KOH until the colour changes to pink. Record how many ml of KOH are used. Then calculate the acid number with Formula 2.

$$\text{Acid number} = \frac{\text{volume KOHxNxBM}}{\text{oil weight}} \quad (2)$$

Where,
The volume of KOH is the number of ml used for the titration.
N is Molar KOH (0.1).
BM is the molecular weight of KOH (56.1).
Oil weight is the weighted amount of oil. The biodiesel produced is then washed with warm water (50 ºC).

3. Result and Discussion

The oil (UCO) used in the experiment was taken from the local waste bank. The initial identification results of UCO are as follows.

Table 1. Initial Characteristics of UCO

Characteristics	Results
Acid number	8.42 mg-KOH/g
% FFA	4.23
Molecular weight	880,4 gr/mol
Colour	Dark brown

Source: Test result, 2020

Experiment I.

From the results of the transesterification process carried out on UCO, it did not produce biodiesel as desired. All different combinations of temperature, time and speed result in saponification or solidification. All 27 experimental combinations are failed. This is due to the high levels of FFA in the UCO (4.23%). In accordance with the research conducted (Gashaw & Theshita, 2014) (Ojiego & al, 2014) (Gopalakrishnan & al, 2017). A homogeneous base catalyst can also not be used on materials with FFA above 3% (Leung, Wu, & Leung, 2010) (Sulaiman, 2016). The form of saponification or saponification can be seen in Figure 2. Saponification or compaction can be in the form of lumps or solid evenly (homogeneous).

Source: Research Document, 2020

Figure 2. Saponification Formed

Experiment II.

Esterification using 100gr of UCO. Prepare 3% and 5% by weight of H_2SO_4, and reflux with a magnetic stirrer at different temperatures and speeds. The molar ratio of oil to Methanol was used in two comparisons. The results of the process are then calculated to yield and %FFA. The following is the result of the esterification process.

Table 2. Result of the Esterification Process

Molar ratio	Catalyst Amount	Temperature, Speed	Yield	% FFA
1:6	H_2SO_4 3%	50°C, 300 rpm	96%	2.115
1:9	H_2SO_4 5%	60°C, 400 rpm	98%	3.524

Source: Experimental Result, 2020

Based on the result, the first treatment reduced %FFA to 2.115 with a yield of 96%. These results can be continued into the transesterification process. For the second treatment, the %FFA level was still above 3 % even though the yield was higher. Less than 3% free fatty acid content in oils is needed (Gashaw & Theshita, 2014) (Ojiego & al, 2014) (Gopalakrishnan & al, 2017). The use of basic homogeneous catalysts (such as NaOH, KOH) also cannot be carried out on materials with FFA above 3% (Leung, Wu, & Leung, 2010) (Sulaiman, 2016). Materials with high FFA will cause saponification in the transesterification process.

The transesterification process was carried out using a NaOH catalyst of 0.5% by weight of oil. The molar ratio of oil and Methanol is 1:6. In this study, experiments were carried out at a temperature of 60°C at a stirring speed of 400rpm. Variations of time used are 30 minutes and 60 minutes. Experiment with 30 minutes; biodiesel has not been formed. Glycerol and biodiesel are still not completely separated. The triglycerides in UCO react with alcohols to form esters and glycerine. It is necessary to provide sufficient opportunity for the reactant molecules to collide with each other. However, after the equilibrium is reached, the additional reaction time does not affect the reaction but can cause the product to decrease due to the back reaction, namely methyl esters are formed into triglycerides. The presence of long-chain free fatty acid in used frying oil decreases the yield compared to palm olein vegetable oil (Danian et al., 2015). Meanwhile, for a processing time of 60 minutes, biodiesel and glycerol can be separated completely. The following are the results of the transesterification experiment.

Table 3. Transesterification Results with 0.5% NaOH Catalyst, Methanol Oil Ratio 1:6

Time	Yield	Acid number	% FFA
30 minutes	Not formed	-	-
60 minutes	98.87%	0.561	0.282

Source: Experimental result, 2020

The transesterified biodiesel was then washed with 50°C warm water. The ratio of the volume of biodiesel and water is 2:1. This washing is done to remove the remaining catalyst and other impurities in the biodiesel. The washing process carried out can reduce the acid

number and purify biodiesel. The following are the results of the washing carried out on the transesterified biodiesel.

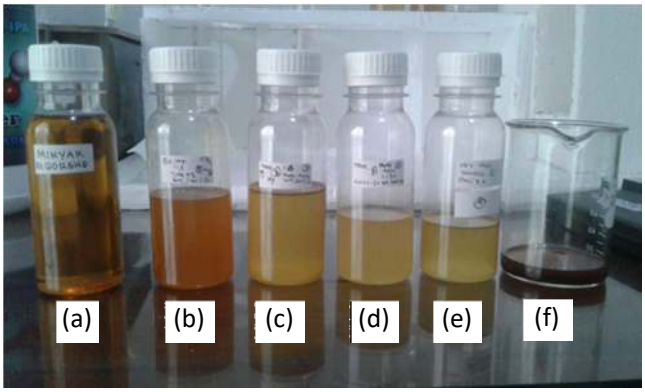
Table 4. Effect of Washing on Acid Number and Color of Biodiesel

Properties	Raw	Washing 1st	Washing 2nd
Acid number mg-KOH/g	0.561	0.2805	0.2805
%FFA	0.282	0.1409	0.1409
Colour	Cloudy yellow	Clear yellow	Clear yellow

Source: Experimental result, 2020

In the two processes of esterification and transesterification, biodiesel productivity can be calculated. The yield produced in the esterification process is 96%, while for the transesterification process, it is 98.87%. So, the biodiesel production resulting from the esterification-transesterification process is 96% x 98.87%, which equals 94.92%. The biodiesel produced has an acid number of 0.2805 mg-KOH/g. This number is still included in the standard of SNI 7182-2015, where the maximum for acid number is 0.5 mg-KOH/g.

The biodiesel synthesis process from UCO also produces residues such as Methanol and water, glycerol, and washing water filtrate. They need additional treatment before reuse or as feedstock. In designing a biodiesel reactor, it is necessary to pay attention to how to process residue to be reused so that the biodiesel synthesis process can be carried out efficiently. Figure 3 shows the change in colour characteristics of the esterification and transesterification processes.



Source: Research Document, 2020

Figure 3. Characteristics change from raw material and after the process.
(a) UCO, (b) esterification, (c) transesterification, (d) 1st washing, (e) 2nd washing, (f) glycerol

Making biodiesel from UCO can be carried out with the esterification-transesterification process or just transesterification depending on %FFA (free fatty acids) in the feedstock. The first method that has been carried out shows that biodiesel is not formed. Saponification occurs in the transesterification process even though experiments have been carried out with various combinations of treatments. This is in accordance with the recommendations and results of

reviews conducted by several researchers, including (Gashaw & Theshita, 2014) (Ojiego & al, 2014) (Gopalakrishnan & al, 2017). The use of basic homogeneous catalysts (such as NaOH, KOH) also cannot be carried out on materials with FFA above 3% (Leung, Wu, & Leung, 2010) (Sulaiman, 2016).

Producing biodiesel from UCO, it is necessary to calculate the acid number first. This is so that the appropriate process can be determined. Oil materials for biodiesel production with different acid numbers will require different treatments therefore experiments with optimal treatments (molar ratio, type and weight of catalyst, temperature, speed, time) need to be carried out with many repetitions in order to obtain valid results. The results of biodiesel need to be tested in full according to the parameters contained in SNI 7182-2015 so that it can be used safely.

4. Conclusion

Temperature, speed and reaction time, and the molar ratio of oil and alcohol, affect the esterification and transesterification processes. The optimal temperature used in the transesterification process is 60°C. The results showed that the temperature used ranged between 50, 60, and 70°C, likewise, with the speed and length of reaction time. The results of previous studies show that the speeds that are often used are between 300, 400, and 500 rpm. Improper speed will cause saponification in the transesterification process with a homogeneous base catalyst. The reaction time used is also different for each researcher. This really depends on the type of material and the catalyst used.

This experiment used different temperatures for the esterification and transesterification processes. In the esterification process, the temperature used is 50°C, the molar ratio of oil alcohol is 1:6 with a reaction speed of 300 rpm for 60 minutes. The catalyst used is a strong acid catalyst (H_2SO_4) as 3%. As for the transesterification process, it uses a homogeneous catalyst NaOH base as much as 0.5% of the weight of the oil. The molar ratio of oil and alcohol was 1:6 with temperature, time and stirring speed of 60°C, 60 minutes and 400 rpm. From the experimental results, it can be concluded that pre-treatment is necessary to make biodiesel from used cooking oil with a high %FFA. Esterification with acid catalyst H_2SO_4 can reduce %FFA in the material. The transesterification process is then carried out to produce biodiesel with an acid number of 0.28 mg-KOH/g according to SNI 7182-2015 standard.

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