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The Synthesis of CME (Coconut Methyl Ester) With Organic Catalyst: Experiments, Data Analysis and Kinetics Model

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Abstract

Biodiesel is one of the biomass materials or renewable energy, which is needed today to replace fuel from fossil energy, which can reduce global warming and has a high renewability cycle. Biodiesel is obtained from plants, so it is also known as biofuel. One type of biodiesel group is CME (Coconut Methyl Ester) which is biodiesel obtained from coconut oil as raw material. In this study, a synthesis of used coconut oil and methanol has been carried out with an organic catalyst based on coconut coir called the ASK catalyst. The results of transesterification have provided some important information, including: the yield are 15-19.5% after usage of the ASK catalyst consisting of amorphous phase and crystalline phase ClK$_{0.8}$Na$_{0.2}$, with the density and viscosity of products are 790-800 kg/m$^3$ and 0.6-1 mm$^2$/s. Yield data obtained are then used to build the kinetics equation. The equation is $Y = 1 - e^{-k't^n}$, with the value of $n = 1$ and $k'$ for temperatures of 50$^\circ$C and 60$^\circ$C were 0.20 and 0.21, respectively, and a minimum activation energy of $Q = 1.1$ kJ/mol, which can determine reaction time needed at a specified temperature to achieve a certain yield value.

Keywords : CME; biodiesel; FAME; organic catalyst; kinetics

Introduction

According to data from the Directorate General of New and Renewable Energy and Energy Conservation of the Ministry of Energy and Mineral Resources, it is estimated that in 2030 there will be a gap between demand and supply for crude palm oil (CPO) for biodiesel where demand is estimated at around 17 million tons while stock or availability is only around 15 million tons (Direktorat Bioenergi EBTKE, 2020). To anticipate this, a diversification is possible to be developed in order to support those huge demand because biodiesel is one of variables in the government's renewable energy program whose total energy consumption from all variable value is targeted to increase from 9% MTOE in 2019, to 23% MTOE in 2025 (Direktorat Bioenergi EBTKE, 2020). Coconut as one of feedstock for biodiesel which had become a potential ogled after palm, both in terms of availability (Govindasamy, 2019) and from a technical point of view because of its physical-chemical properties (Canakci & Sanli, 2008; Habibullah et al., 2015; Lukić et al., 2016), and its application which tend to be superior to another feedstock (Broatch et al, 2014; Choung Chiong et al., 2018; Chupka et al., 2013; Habibullah et al., 2015; Lin et al., 2011; Sugami et al., 2017; Thangaraja & Srinivasan, 2019; Yuliansyah et al., 2019).
Meanwhile, from an economic point of view, usage of coconut oil as a raw material in production costs is less competitive due to raw material price, so this situation can be taken into consideration to use used cooking oil as raw material for biodiesel production, where many studies have been succeeded in proving that (Budiman et al. 2017; Lukić et al., 2016; Talebian-Kiakalaieh et al., 2012; Wijaya et al., 2016).

Furthermore, according to people's beliefs from generation to generation, all parts of coconut also have many benefits, such as skin or husk. Usage of coconut husk as a biodiesel catalyst has been carried out by several researchers (Husin et al., 2018; Vadery et al., 2014) which provides information that the yield produced is around 90% so that it is possible to use it on a commercial scale with used coconut cooking oil to support 3R (reuse, reduce, recycle) program campaign which is needed for environmental sustainability.

**Method**

The research flow chart which become reference for this research activity is given in Figure 1. The rectangular symbol represents an activity or process while the diamond symbol represents a process with certain conditions where a decision is needed.

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**Figure 1. Research flow chart**
Based on flow chart in Figure 1, research activities in general can be grouped in the arrangement of activity stages, consisting of: preparatory activities; initial characterization activities; synthesis activities; final characterization activities; and modelling.

In preparation of used oil, we got it from coconut cooking oil which we used for frying for 3 times. The determination of 3 times of frying was carried out as a research limitation so that the stability of materials was expected to be measured in same treatment. After being cooled to room temperature, the used oil was then filtered to separate it from remaining food ingredients, then we collected it in the container. Meanwhile, in term of preparation of the ASK catalyst, the wet coconut coir was first dried in open air or in the sun for approximately 5 hours. After dried, it was then heated using an oven at 350°C for 1 hour and then cooled and collected in the provided container.

Initial characterization activities of used oil using FTIR (Fourier Transform Infra Red) Spectroscopy and GC (Gas Chromatography) techniques to determine quality of the material. Then Fuel Property Test was done in the form of calculating acid number, iodine number, and density of used oil.

Transesterification of CME was carried out by using following parameters: (1) volume ratio of oil : methanol were 3:1 and 2:1; (2) variation of catalyst concentration were 5 and 10wt.%; (3) stirring speed was 500 rpm; (4) reaction temperature was 50°C; and (5) reaction time was 1 hour. After the reaction time is complete, the material is then cooled for 10 minutes and then put into a separating funnel. Two layers then will be formed, upper layer in the form of methyl ester products (CME) and lower layer in the form of glycerol. After approximately 12 hours, the CME was then accommodated in a beaker glass to be weighed and the yield calculated based on following equation:

\[
\% \text{ Yield} = \frac{WP}{WR} \times 100
\]

Whereas :
WP = weight of the separated product (g)
WR = weight of the reactant (g)

After the synthesis process with transesterification technique was completed, it was continued with characterization of the catalyst using XRF (X Ray Flouresence) and XRD (X Ray Diffraction) as a follow-up analysis. CME products then entered the final characterization stage, by density test, and a kinematic viscosity test using a viscometer, and the best result will be analyzed with several physical tests of other fuel products, which are CME with 0.25 wt.% KOH catalyst, PME (Palm Methyl Ester), and B0 or “Solar” as a pure fossil fuel.

Result

Raw Material Characterization
The analysis of Figure 2 showed several functional groups that appear in peak which can be divided into 3 areas, first, the wave number area of 500-1500 cm$^{-1}$, which is the fingerprint area. According to a reference (Dachriyanus, 2004), this area was generally neglected because it contained very complex absorbent forms. This was because all types of molecular bending vibrations absorbed in this region. Meanwhile, the second was the area of wave number 1684-1755 cm$^{-1}$, in this area there was bond stretching, which shows the interpretation of the C=O carbonyl group. Third, the area of wave number 2800-3000 cm$^{-1}$, in this area there is also a stretching or interpretation of the CH and CH$_2$ group bonds (Dachriyanus, 2004). The -OH group (indicating the presence of excess water content) which was absorbed from the wave number above 3000 cm$^{-1}$ was not found (+-98% transmittance), so it was ensured that the material did not contain water. The significant difference between the new oil and the used oil is that the triglycerides from the new oil showed no absorption at the wave number around 500 cm$^{-1}$ because the hydrolysis process which produced fatty acid was not occurred.

Table 1. GC and fuel property test of used oil

| Appearance | reddish yellow |
|------------|----------------|
| Acid Value | mg KOH/g sample | 0,33 |
| Iodine Value | g I$_2$/100 g sample | 4,4 |
| Density | g/ml | 0,92 |
| Composition | | |
| C-8 | 6,22% |
| C-10 | 5,28% |
| C-12 | 42,63% |
| C-14 | 16,94% |
| C-16 | 12,06% |
| C-18 | 16,60% |
| C-18-1 trans | 0,05% |
| C-20 | 0,23% |
Table 1 showed the composition of chemical bonds in the material which were dominated by saturated fatty acids in the form of lauric acid (C-12) which is 42.63%. According to a reference (Lin et al., 2011) where pure coconut oil was contained 46.5% of lauric acid, this result showed suitability. Meanwhile, other compositions varied, C-14 acid or myristic acid was 16.94%, C-18 or stearic acid was 16.60%, C-16 acid or palmitic acid was 12.06%, and several other fatty acids. As for unsaturated fatty acids which was C-18-1 trans acid or elaidic acid, the percentage was only 0.05%. These saturated fatty acids indicates the stability of the material, which will be strengthened by the iodine number test. Furthermore, the acid value of 0.33 mg KOH/g was far below the research reference limit of 5 mg KOH/g (Budiman et al., 2017; Wijaya et al., 2016). This means that the free fatty acid content of the material was still good, so there was no need for an esterification process before carrying out the transesterification process. While the iodine value or iodine value of 4.4 g I$_2$/100 g indicates the stability of the used oil, because the more I$_2$ reacts, the more double bonds or unsaturated bonds in the material. These bonds can make the material easily degraded or oxidized. In comparison, in the another edible oils such as palm oil and olive oil, each of which has an iodine value of 57.5 and 80.2 g I$_2$/100 g (Peamaroon, Jakmunee, & Moonrungsee, 2021) while non-edible oil such as Simarouba glauca (paradise tree) oil has an iodine value of 46 g I$_2$/100 g (Atabani et al., 2013).

**Transesterification**

| CME | vol.ratio of oil:methanol | wt.% catalyst | Temperature (°C) | Time (hr) | rpm | %yield |
|-----|---------------------------|---------------|------------------|-----------|-----|--------|
| 1   | 3:1                       | 5             |                  |           | 15  |        |
| 2   | 3:1                       | 10            |                  | 1 hr      | 500 | 16     |
| 3   | 2:1                       | 5             | 50               | 1 hr      | 500 | 18.3   |
| 4   | 2:1                       | 10            |                  |           | 19.5|        |

In table 2 above, it can be seen that the largest yield with a value of 19.5% occurred at a CME-4 (10 wt.% catalyst concentration with a 2:1 oil:methanol volume ratio). Changes in concentration or volume ratio from 3:1 to 2:1 in 5wt.% catalyst increased yield by 18%, the same value of increase was also found in changes in oil:methanol volume ratio from 3:1 to 2:1 in 10wt% catalyst concentration.

**Catalyst Characterization**
Characterization using ED (Energy Disperse) XRF presented in the form of elements and their stable oxide compounds as table 3 and 4. Na, K, and Cl, were the dominant elements in the catalyst with an amount of 8.91; 7.44; and 2.37 weight percent, respectively, while the most dominant form of oxide obtained is Na$_2$O and K$_2$O with 9.85 and 8.15 weight percent. In the use of ED-XRF, there are several elements whose energy spectrum is not detected (generally elements of organic compounds such as C, H, O and N).

In general, the main elements of catalysts have been identified, i.e. active elements or compounds with catalytic properties such as metals, alkali metals or halide elements and compounds (Lukić et al., 2016). This is also specifically in accordance with the reference where in coconut coir that have been converted into husk ash, one of the constituent elements is a potassium element-based compound (Vadery et al., 2014).

Figure 3 is X-ray diffraction spectrum of the ASK sample data processed using the GSAS software. The graph with a steep up-and-down pattern is the main graph, while the purple graph below showed description of wRp, or was called weight ratio pattern, which shows the quality or accuracy of fitting with a limit of 0.1, so that the value of wRp was less than 0 showed that the results of data processing are accurate. The presence of
amorphous phase was indicated by the background data that was mountainous and wide starting at an angle of $2\theta = 5^\circ$ to $60^\circ$. While the presence of a crystalline phase was indicated by the presence of diffraction peaks at 20 diffraction angles of 29° and 41°. Seeing large intensity of the diffracted background light, it can be assumed that the ASK sample is dominated by the amorphous phase (Sugondo & Badruzaman, 1999). This is very possible considering that the dominant chemical element in the ASK sample consists of organic elements such as C, O, N, etc which were not detected by XRD because they were light atoms.

The results of GSAS analysis of the ASK catalyst showed that the crystalline phase contained in the ASK sample was the crystalline phase Cl$\text{K}_{0.8}\text{Na}_{0.2}$. Another method is needed to determine the amorphous phase in ASK samples and this was not carried out in this analysis. It can be concluded temporarily that the presence of Cl$\text{K}_{0.8}\text{Na}_{0.2}$ crystalline phase in the amorphous ASK sample, together with main compounds i.e. $\text{K}_2\text{O}$, and $\text{Na}_2\text{O}$, can produce a maximum CME yield of 19.5%. This was because using of heterogeneous catalysts in some previous studies had shown that the presence and number of crystal phases can have an effect in the adsorption activity of catalyst elements where it can lower FFA (Free Fatty Acid) levels in esterification reactions and increase product yields in transesterification reactions (Chen et al., 2013; Husin et al., 2018; Sakti La Ore et al., 2020; Vadery et al., 2014).

**Product Characterization**

Furthermore, three CME products with one additional product (for kinetic purpose) are characterized at the end with physical testing generating data in the following table 5:

| Sample | Density at 40°C (g/ml) | Kinematic Viscosity at 40°C (mm²/s) | Parameter | Volum ratio (oil:methanol) | Temp. (°C) | Catalyst (wt.%) | Time (hr) | rpm |
|--------|------------------------|-------------------------------------|-----------|---------------------------|------------|-----------------|-----------|-----|
| CME-1  | 0.802                  | 1                                   |           | 3:1                       | 50         | 10%             | 1         | 500 |
| CME-2  | 0.789                  | 0.8                                 |           | 2:1                       | 50         | 10%             | 1         | 500 |
| CME-3  | 0.798                  | 0.8                                 |           | 2:1                       | 50         | 5%              | 1         | 500 |
| CME-4  | 0.788                  | 0.6                                 |           | 2:1                       | 60         | 5%              | 1         | 500 |

The reaction rate will be faster as some parameters increase: the surface area of reaction contact, reactant concentration, reaction temperature and catalyst. Based on the table 5 above, there is a wedge between the two, which were the concentration and reaction temperature. Based on this, the first effect observed was the reactant
concentration which was represented by the volume of the reactants, assuming that the reaction proceeds in a first order (Sofyan, Hafizah, & Manaf, 2020).

Then it can be analyzed that the addition of the volume of oil by 11% from a ratio of 2:1 to 3:1 with fixed parameters of reaction temperature, %catalyst, rpm, and reaction times of 50°C, 10%, 500 rpm and 1 hour, respectively, will increase the viscosity value by 20% (0.8 mm²/s to 1 mm²/s) and increase the value of a density of 2% (0.789 gr/ml to 0.802 gr/ml). On the other hand, a decrease in volume oil from a ratio of 3:1 to 2:1 will decrease its viscosity and density by the same percentage. Thus, reducing the ratio to 2:1 already fulfills limit of reactant value where the trend of product density value is smaller than density of reactant.

In the same table, it can also be analyzed that variations in temperature increase from 50°C to 60°C with fixed parameters volume ratio, %catalyst, rpm, and reaction time of 2:1, 5%, 500 rpm, and 1 hour, respectively, will reduce the viscosity by 25% and reduce the density value by 1%. This matter also has complied with the limit of the density of the reactants to the products. the reactant concentration parameters, which were represented by the volume ratio of oil: methanol and temperature parameters that were analyzed with the product, had been running normally according to the first order reaction.

CME-1 with the highest density and viscosity values was then selected to be compared with the results of physical testing from other fuel samples, which are the CME with 0.25 wt.% KOH catalyst we made as per the result of previous research (Wahyuningsih & Wega, 2015), PME (Palm Methyl Ester) or B100 palm, and Diesel fuel or B0 as figure 4. The CME product with 0.25 wt% KOH catalyst was synthesized with the same transesterification scheme as the research methodology so that the recapitulation in table 6 was obtained.

![Figure 4. Sample product variants include (a) CME with organic catalysts; (b) CME with KOH inorganic catalyst; (c) PME; and (d) B0](image-url)
Tabel 6. Recapitulation of CME with reference fuel

| Product Value       | CME (ASK catalyst) | CME (KOH catalyst) | B100 PME | B0 |
|---------------------|--------------------|--------------------|----------|----|
| yield (%)           | 16                 | 78                 | ND       | -  |
| density (kg/m³)     | 802                | 859                | 857      | 837 |
| kinematic viscosity (mm²/s) | 1,0              | 3,3                 | 4,5      | 3,8 |

From table 6 above it is seen that the CME product with ASK organic catalyst have smaller density and viscosity values than the CME product with KOH catalyst, this was due to the less reactive character of catalyst when compared to KOH catalyst for the same material and time parameters. While from the results of viscosity comparison between products, both CME with KOH catalyst and CME with ASK catalyst have lower viscosity values than PME and B0. This is an advantage as explained earlier (Choung Chiong et al., 2018; Habibullah et al., 2015; Lin, Cunshan, Vittayapadung, Xiangqian, & Mingdong, 2011; Thangaraja & Srinivasan, 2019; Yuliansyah et al., 2019), where with lower viscosity the flow of fuel easily flows well to the combustion chamber, causing the fuel to be properly atomized, resulting in more perfect combustion results and resulting in lower hydrocarbon emission, so CME products with organic catalysts ASK might be promoted for further research.

The above experimental CME yield data was then processed by the same method as the previous study (Sofyan, Hafizah, & Manaf, 2020), produced the Avrami kinetic equation with a value of n = 1 and k' value for 50°C and 60°C were 0,20 and 0,21; with a minimum activation energy of Q = 1,1 kJ / mol. Through this equation was built a model to determine a reaction time at a specified temperature to achieve a certain yield value, as shown as figure 5.

![Figure 5. Kinetic model for CME with organic catalyst](image-url)
The model showed that CME with a reaction temperature of 60°C or 333 K is better than CME with a reaction temperature of 50°C or 323 K because it can produce a larger yield with a determined time, for example at a reaction time of 3 hours, CME T 333 K and CME T 323 K produce yields of 45% and 47% respectively. In the model has also been shown other information that to achieve a yield of >50%, then the minimum reaction time needed is 4 hours.

**Conclusion**

The synthesis of CME (biodiesel) from used coconut oil through transesterification techniques using The ASK organic catalysts can produce yield products in the range of 15% to 19.5%. The CME products with organic catalysts have a tendency of lower density and viscosity values compared to other methyl ester or biodiesel products. These characteristics provide an advantage in diesel engines application.

The CME yield data obtained from calculations was then used to construct kinetic equations and a model that can predict yields in a predetermined reaction time and temperature. Thus, with this model, the time redundancy for trials can be reduced. Also with recommendations of reaction times that are longer than the current experiment, in addition to increasing effectiveness and productivity, later The CME products can also be further characterized so that more comprehensive data can be obtained.

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