

Waste cooking oil processing for fatty acid methyl ester and mono glycerides production with magnetite catalyst

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Abstract

The objective of this research was to produce biodiesel using waste cooking oil and various magnetite catalysts with the esterification-transesterification process. Magnetite catalysts tested were α -Fe₂O₃, α -Fe₂O₃/Al₂O₃, α -Fe₂O₃/ZSM-5 catalysts. Catalysts were prepared through chemical precipitation and calcination. The esterification-transesterification process was carried out with the conditions WCO: methanol molar ratio of 15:1, catalyst (1% wt of oil), heated at 65°C for 3 hrs. The results showed biodiesel production using α -Fe₂O₃-ZSM-5 catalyst obtained higher %FAME (83.28%), yield (91.915%) and monoglyceride content (16.72%) compared to others due to larger pore volume. Biodiesel produced passed the requirement of Indonesian National Standard (SNI) based on density, acid number and viscosity.

1. Introduction

Palm oil is one of the most important agricultural products in Indonesia. The national Crude Palm Oil (CPO) production has increased by 8% from 32.18 MT in 2017 to 34.71 MT in 2018. It is predicted that by 2019 it will grow by 5% (Deha, 2019). CPO was used as raw material in cooking oil production. Cooking oil is widely used not only in household capacity but also in industries. Consequently, used cooking oil is abundant as waste. Waste cooking oil (WCO) refers to vegetable oil that has been used in food production and no longer viable for its intended use. WCO is a potentially problematic waste stream which requires proper management. The problem arises lately as many restaurants and hotels sold WCO to other dealers, which then recycle it. The recycled oil supposedly should not be used for cooking as it is unfit for consumption, causing health hazards when it is consumed or used in processing products (Tsai, 2019).

WCO can be converted to cooking oil through adsorption. The adsorbent used could be bentonite and zeolite (Widayat *et al.*, 2006). However, WCO can also be converted to FAME through esterification (Lotero *et al.*, 2005) and transesterification reaction as it contains 96.43%w triglyceride, which is a potential raw material for fatty acid methyl ester (FAME) or biodiesel production (Juan *et al.*, 2011; Yusup *et al.*, 2015). Biodiesel has numerous advantages such as low carbon

content, biodegradable, more efficient combustion and non-toxic than other fuels (Tan *et al.*, 2015). Biodiesel is environmentally friendly due to the easily reabsorbed combustion gases by plants for photosynthesis. Furthermore, it will reduce exhaust emissions without compromising the performance and efficiency of the engine. Use of 100% biodiesel will reduce CO₂ and SO₂ emissions by 100% as well as CO and HC emissions by 10-50% (Havendri, 2008).

Iron sand has the potential to be developed as a catalyst due to hematite (α -Fe₂O₃) magnetic mineral content. Several studies on biodiesel synthesis using TiO₂ supported hematite catalysts from waste cooking oil have been carried out by Zhai *et al.* (2010), Anuradha *et al.* (2014) and CaO supported by Ezzah-Mahmudah *et al.* (2016) which shown successful biodiesel production with a yield of 92.1%, 92.2% and 92% respectively. However, the use of TiO₂ as catalyst support has significant drawbacks such as small specific surface areas, high cost and potential leaching (Bagheri *et al.*, 2014). Therefore, the need of other catalyst support is crucial. Al₂O₃ and ZSM-5 are potential solutions because of large surface areas, lower cost, easily accessible and no potential leaching hazard. Furthermore, Al₂O₃ and ZSM-5 were widely used as catalysts in biodiesel production, shown by researches namely Marinkovic *et al.* (2017), Mohadesi *et al.* (2015), Zabeti *et al.* (2009), Fawaz *et al.* (2019), Estephane *et al.* (2016) and many more. In this research, waste cooking oil is reacted with

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methanol using α -Fe₂O₃, α -Fe₂O₃/ZSM-5 and α -Fe₂O₃/Al₂O₃ catalysts to produce biodiesel.

2. Materials and methods

2.1 Materials

Waste cooking oil was obtained from several nearby restaurants in Tembalang, Semarang. Chemical reagents used are KOH (85%, Merck, Germany), H₂SO₄ (95%, Merck, Germany), methanol (99.9%, Merck, Germany) and distilled water. ZSM-5 (99%) was purchased online from Alibaba.com and Al₂O₃ (99.9%) purchased from PT INALUM Indonesia. Iron sand as α -Fe₂O₃ resources obtained from Cilacap District Central Java Indonesia.

2.2 Preparation of catalyst

Iron sand was separated by magnetic separation method to obtain higher iron content. Hematite (α -Fe₂O₃) initially undergone chemical precipitation and followed by calcination to obtain the hematite phase (α -Fe₂O₃). A total of 20 g of iron sand filled in beaker glass is added with 40 mL of HCl 37%. The mixture was stirred at 70°C for 30 mins and followed by filtration using Whatman filter paper (No. 1). The filtrate was then mixed with PEG (polyethylene glycol) 4000 which previously has been melted with a volume ratio of 1:5. Then, it was stirred again at 70°C for 40 mins until precipitation is formed. The precipitate was washed with distilled water while the catalyst was separated by filtration in vacuum condition. The catalyst then was dried in the oven at 120°C for 2 hrs and calcined in the tube furnace at 750°C. The results of catalyst preparation were presented in Widayat *et al.* (2019).

2.3 Waste cooking oil pretreatment

WCO was pretreated using esterification reaction. WCO was heated to 70°C and added with sulfuric acid in methanol. This is considered as the starting point of the reaction. Esterification performed by using methanol is carried out with the following conditions: methanol: waste cooking oil molar ratio of 5:1 and H₂SO₄ 1%w of WCO at 70°C for 300 mins. After esterification is carried out, the mixture is moved to separating funnel and washed with warm water. Bottom product and water are discarded while excess methanol is separated from esterification product.

2.4 Transesterification

Transesterification method refers to Ezzah-Mahmudah *et al.* (2016) with slight modifications. Transesterification of waste cooking oil was done by conventional methanol reflux process. A mixture of waste cooking oil, methanol (molar ratio of 15:1) and catalyst (1% wt of oil) was heated at 65°C for 3 hrs. The

mixture was discharged and centrifuged at 3500 rpm to separate the solid catalyst. After catalyst is separated, liquid product is moved to separating funnel and let to settle to 3 layers for 12 hrs. Glycerol at the bottom layer, FAME at the middle and excess methanol at the top. FAME was washed with warm water and done until it is clear which indicates there were no methanol or impurities left in FAME. Water that still contains FAME was removed by evaporation process and the remaining product (FAME) was collected for yield determination and characterization.

2.5 FAME characterization

The product was analyzed using GC-MS (Gas Chromatography Mass Spectroscopy). The yield is the ratio of the product obtained to the raw material, or as written on Equation 1:

$$\%Yield = \frac{\text{Weight of Biodiesel (gram)}}{\text{Weight of transesterification feed (gram)}} \times 100\% \quad (1)$$

FAME was also analyzed for its density, viscosity and acid number.

3. Results and discussion

3.1 Production and characterization of biodiesel

In the production of biodiesel, waste cooking oil is used as raw material. Prior to production, waste cooking oil was pretreated to reduce impurity through heating and filtering. The pretreated waste cooking oil can be seen in Figure 1(a) Waste Cooking. The waste cooking oil is then analyzed using GC-MS to determine its content. In addition to GC-MS analysis, %FFA (free fatty acid) analysis was carried out. From the analysis, it was found that %FFA contained in waste cooking oil was 6.175%. According to Canakci and Van Gerpen (1999) and Ramadhas *et al.* (2005) oils with high FFA (>2%) cannot be processed directly with transesterification as it can cause saponification. Saponification reaction occurs between free fatty acids and basic catalysts. Saponification will decrease the effectiveness of the catalyst, which will consequently reduce FAME yield and complicate the separation between FAME and glycerol. Waste cooking oil used in this study has a FFA content above 2% (6.178%). Therefore, the esterification process needs to be carried out to reduce the FFA content. Through esterification, FFA content was successfully reduced to 1.238% which is in accordance with the requirement of the transesterification process. The results of the transesterification process (biodiesel) can be seen in Figure 1(b) Transesterification results (biodiesel).

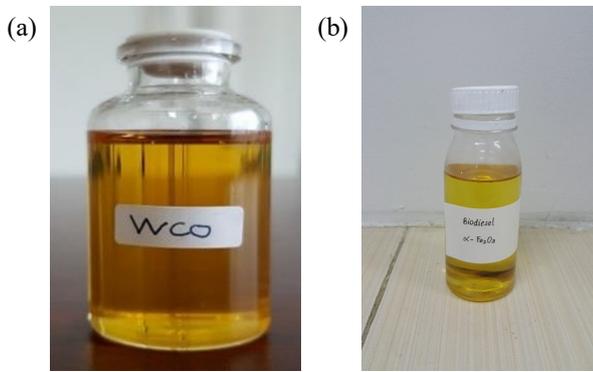


Figure 1. (a) Waste cooking oil (b) Transesterification results (biodiesel)

3.2 Catalyst pore characterization

Figure 2(a) and 2(b) shows the surface area and pore volume of α -Fe₂O₃-ZSM-5/Al₂O₃ respectively. The surface area of the catalysts increases after impregnated with ZSM-5 and Al₂O₃. This enlargement is due to the large surface area of the supporting catalyst, 447 m²/g for ZSM-5 (Suzuki *et al.*, 1983) and 129 m²/g for Al₂O₃ (Huang and Stumm, 1972). Impregnation of α -Fe₂O₃ to support catalyst will cause pore volume to enlarge. Therefore, a larger surface area of supporting catalyst will result in larger pore volume. However, the pore volume of α -Fe₂O₃/ZSM-5 catalyst is much higher compared to α -Fe₂O₃/Al₂O₃ who has a smaller surface area. This is due to the overload of α -Fe₂O₃ used (50% w), where it would fully fill the pore carrier – leading to blockage – and eventually shrink the surface area.

3.3 Yield, Fatty Acid Methyl Ester (FAME) and monoglyceride content of biodiesel

Figure 3 shows the yield and FAME content of the biodiesel produced. It could be seen that yield and FAME obtained was directly proportional to the results of the pore volume obtained in BET analysis. Pore volume represents the active surface in which contacts between the reactants in the reaction process occurs. The greater the active surface of the catalyst, the better the catalyst activity is expected (Ozkar, 2009). Based on the difference in the catalyst, the highest yield and FAME content was produced using α -Fe₂O₃/ZSM-5. This is in

accordance with the earlier statement as ZSM-5 has the largest surface area 447 m²/g (Suzuki *et al.*, 1983) among the three catalysts.

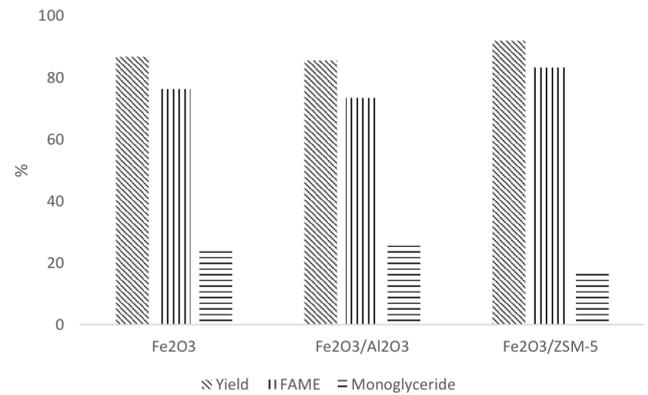


Figure 3. Yield, FAME and monoglyceride content of biodiesel

In addition, it can be concluded that α -Fe₂O₃ has a fairly good catalytic ability in biodiesel production. This conclusion was taken because α -Fe₂O₃ has twice the pore volume of α -Fe₂O₃/ZSM-5. Although the former has higher pore volume, FAME produced was in reality not too far apart. This is probably caused by α -Fe₂O₃ which belongs to the class of metal oxides. Metal oxides in heterogeneous catalysts consist of positively charged metal ions which act as electron acceptors, while negative oxygen ions act as proton acceptors. The presence of these active sites causes the catalyst to provide an adsorptive site for methanol where the O-H bond will be easily broken into methoxide ions and hydrogen cations. Methoxide anion in the catalyst will react with triglyceride molecules (Refaat, 2011). The formation of this nanocomposite (metal oxide) catalyst will increase catalytic activity due to increased catalyst active site.

3.4 Density, acid number and viscosity of biodiesel

Based on Table 1, the density, acid number and viscosity of biodiesel obtained have different values for each variable. It can be concluded that density, acid number and viscosity obtained in this study has good

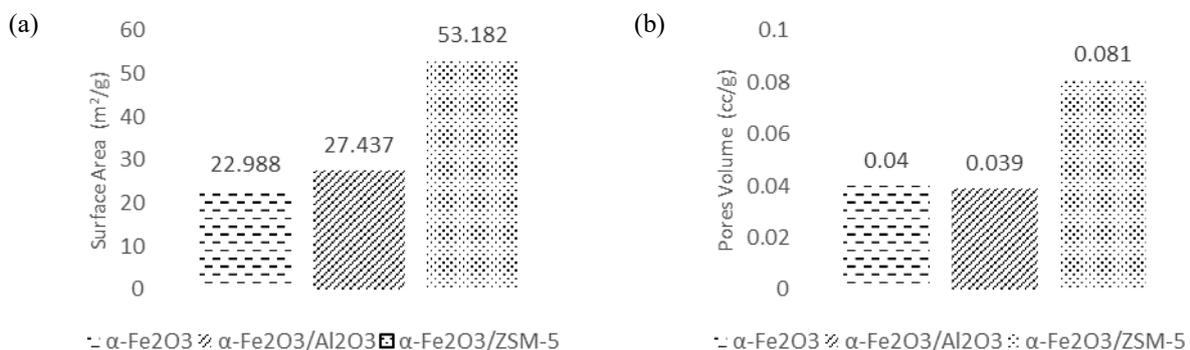


Figure 2. (a) Surface area (b) Pore volume of α -Fe₂O₃-ZSM-5/Al₂O₃

quality because it meets the SNI 7182-2015 biodiesel standard (SNI, 2015).

Table 1. Density, acid number and viscosity of FAME

Parameters	Variable			SNI
	α -Fe ₂ O ₃	α -Fe ₂ O ₃ / ZSM-5	α -Fe ₂ O ₃ / Al ₂ O ₃	
Density (kg/m ³)	888.3	884.6	888.9	850 – 890
Acid Number (mg-KOH/g)	0.27	0.362	0.27	Max 0.5
Viscosity (mm ² /s)	5.685	5.312	5.889	Max 6

It cannot be concluded how catalyst influences the differences in biodiesel density, but biodiesel obtained in this study can be said to have good quality because it meets the SNI 7182-2015 biodiesel standard (SNI, 2015). Density is mass per unit volume. Consequently, higher density means it contains more mass which would produce higher emission after combustion (Tuccar *et al.*, 2018). Therefore, the density of the biodiesel produced has to meet the standard quality that is regulated by the government to control the emission.

Furthermore, it also cannot be concluded how catalyst influences the differences in biodiesel acid

number. However, it was indicated that the alkalinity of the catalyst affects the value of the acid number. Acid number is used to determine the presence of acid in a sample of biodiesel. In this study, the acid number is obtained from the amount of %FFA in WCO after the esterification process. The small values of acid number obtained were the result of the effectiveness of catalyst in reducing the acid content during the esterification process. When compared to the SNI acid number standard, biodiesel produced in this study has met the requirement. This is an advantageous property as the high acid number will lead to corrosion in the system (Patel and Shah, 2015).

Viscosity is an important property of fuel which affects the formation of NO_x. Low viscosity will yield lower NO_x emission, which will lead to a decrease in the greenhouse effect (Tuccar *et al.*, 2018). Based on the SNI viscosity standard for biodiesel, it can be said that biodiesel produced in this study has relatively good quality as it has lower viscosity which would contribute to lower exhaust emission.

3.5 GC-MS Results

Results of the GC-MS analysis on biodiesel produced were shown in Figures 4, 5 and 6. Figure 4

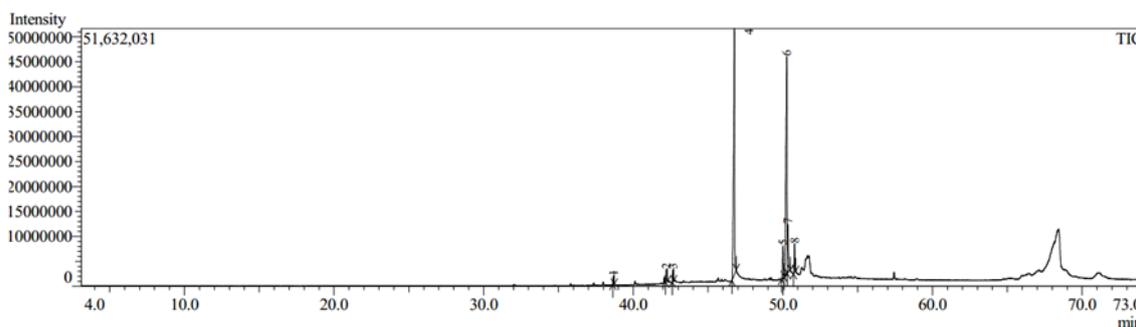


Figure 4. GC-MS result of biodiesel synthesis using α -Fe₂O₃ catalyst

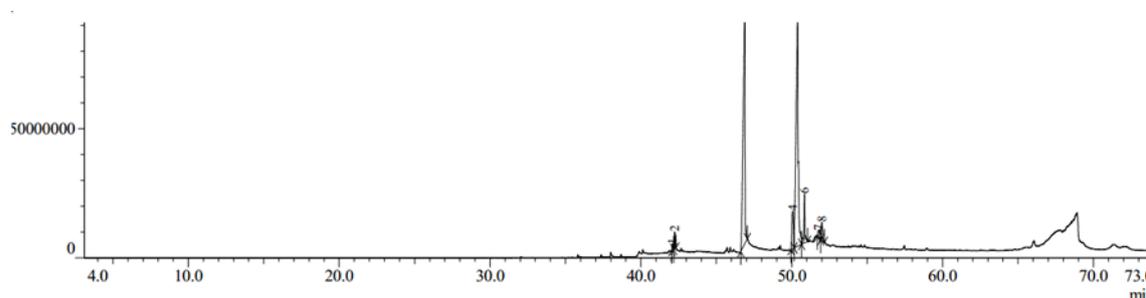


Figure 5. GC-MS result of biodiesel synthesis using α -Fe₂O₃/Al₂O₃ catalyst

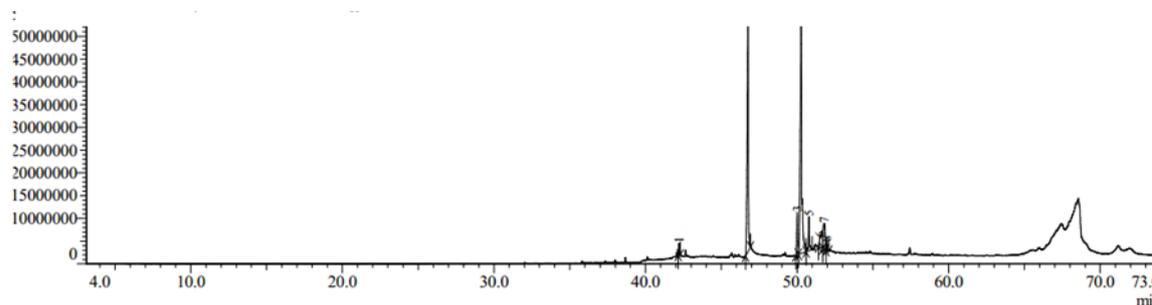


Figure 6. GC-MS result of biodiesel synthesis using α -Fe₂O₃/ZSM-5 catalyst

shows the result of biodiesel produced using $\alpha\text{-Fe}_2\text{O}_3$ catalyst. The highest area percentage of 42.63% is Hexadecanoic acid, 2-hydroxy-1,3-propanediyl ester, a monoglyceride component. The retention time is found to be 46.760 mins. It is followed by glycerol (di-(9-octadecenyl)-glycerol) which has an area percentage of 38.76%, with retention time 50.257 min.

Figure 5 shows the chromatograph result of biodiesel produced using $\alpha\text{-Fe}_2\text{O}_3/\text{Al}_2\text{O}_3$ catalyst. The highest percentage of component is found to be glycerol (di-(9-octadecenyl)-glycerol) with an area percentage of 44.43%, retention time of 50.389 mins. Hexadecanoic acid, 2-hydroxy-1,3-propanediyl ester (monoglyceride component) is the second highest component found, with area percentage of 40.18%, retention time of 46.893 min.

Figure 6 shows the result of biodiesel production using $\alpha\text{-Fe}_2\text{O}_3/\text{ZSM-5}$ catalyst. The highest percentage is found to be the same component as in biodiesel production using $\alpha\text{-Fe}_2\text{O}_3/\text{Al}_2\text{O}_3$ catalyst. With area percentage of 42.29%, retention time of 50.270 mins, di-(9-octadecenyl)-glycerol is the highest component contained. Followed by Hexadecanoic acid, 2-hydroxy-1,3-propanediyl ester (monoglyceride component) with area percentage of 35.36%, retention time of 46.768 mins.

Based on the results, it could be concluded that the $\alpha\text{-Fe}_2\text{O}_3\text{-ZSM-5}/\text{Al}_2\text{O}_3$ catalysts are not selective in producing methyl esters. This could be seen from the formation of monoglyceride component instead of methyl esters. Monoglyceride is the result of direct esterification and/or transesterification of glycerol with fatty acid molecule (Bart *et al.*, 2010). In this study, the production of biodiesel uses simultaneous esterification and transesterification process. Methyl esters which were converted from free fatty acids in WCO during the esterification process will further react with the glycerol present from transesterification, thus forming monoglyceride. The mechanism of monoglyceride

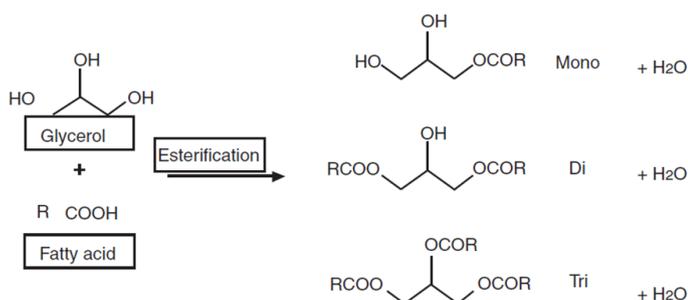


Figure 7. Glycerolysis reaction mechanism (Felizardo *et al.*, 2011)

formation or known as glycerolysis is shown in Figure 7.

4. Conclusion

Waste cooking oil can be converted into free fatty acid methyl ester (FAME), glycerol and monoglyceride such as Hexadecanoic acid, 2-hydroxy-1,3-propanediyl ester. Highest biodiesel yield was obtained with the use of $\alpha\text{-Fe}_2\text{O}_3\text{-ZSM-5}$ catalyst (83.28%). Density, acid number and viscosity of product obtained in this study is in accordance with SNI 7182-2015 biodiesel standards. $\alpha\text{-Fe}_2\text{O}_3\text{-ZSM-5}/\text{Al}_2\text{O}_3$ catalysts were not selective in biodiesel production, proven with the formation of monoglyceride.

Conflict of Interest

The authors declare no conflict of interest.

Acknowledgments

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