

CO-SOLVENT TETRAHYDROFURAN IN THE PRODUCTION OF BIODIESEL FROM USED COOKING OIL USING THE TRANSESTERIFICATION METHOD

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Received 17 January 2023 Accepted 16 February 2023 Published 01 March 2023

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DOI 10.29121/ijetmr.v10.i3.2023.1274

Funding: This research received no specific grant from any funding agency in the public, commercial, or not-for-profit sectors.

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ABSTRACT

Used cooking oil is a waste oil derived from cooking oil that has the potential to be produced into biodiesel because it contains fatty acids and triglycerides like other vegetable oils, however, its utilization is still low. This study aimed to analyze the effect of co-solvent THF based on reaction time and oil: methanol molar ratio in the manufacture of biodiesel using used cooking oil as raw material. Transesterification is a method used in biodiesel production by reacting raw materials with alcohol. KOH1% is used as a catalyst in the process of converting used cooking oil into biodiesel. In its operation, reaction time variations of 5, 7, 10, 13, and 15 minutes and molar ratios of 1:3, 1:6, 1:9, 1:12, and 1:15 were used at a temperature of 65oC. The results showed that when entering the reaction time of 10 minutes the density and viscosity tend to be stable and meet the standards of SNI 7182:2015, namely the density is stable at 0.88 gr/ml and the viscosity is in the range of 4.6238 cSt-5.7336 cSt. Meanwhile, based on the molar ratio, it was obtained only at a molar ratio of 1:6 which is in accordance with the characteristics of biodiesel based on SNI 7182:2015 which has a density of 0.88 gr/ml and a viscosity of 4.6238 cSt. The best % yield was at a 1:6 molar ratio with a reaction time of 13 minutes, that is 88.8199% with a heating value of 33.0398 MJ/kg and a cetane number of 66.9. Based on the methyl ester test with GC-MS, the methyl ester (biodiesel) content in the product was 87.24%, dominated by Oleaic (9(Z)-Octadecenoic) acid or 49.33% oleic acid and Palmitic (Hexadecanoic) acid. or palmitic acid by 34.08%.

Keywords: Used Cooking Oil, Co-Solvent THF, Transesterification, Biodiesel

1. INTRODUCTION

The utilization of used cooking oil as raw material for biodiesel is currently not thoughtfully developed in Indonesia. In fact, a study by Traction Energy Asia 2019 shows that of the 28.4 kilolitres (KL) of used cooking oil produced in Indonesia, the potential for biodiesel is 5.7 KL litres Traction Energy Asia. (2019). This potential may meet the national biodiesel needs of up to 32%. However, only 3 million KL of used cooking oil has been collected in Indonesia, so only 570 thousand KL is used as biodiesel. The remaining 2.43 million KL is produced as a recycled cooking oil and 184 thousand KL is exported Aprobi (2022). However, used cooking oil has high levels of FFA, this is due to the formation of free fatty acids in used cooking oil that

occurs during frying so that a hydrolysis reaction occurs in triglycerides, producing free fatty acids, diglycerides, monoglycerides, and glycerol which can be indicated from the acid number Setyawati and Edwar (2012). This condition may interfere with the biodiesel production process because the transesterification process uses an alkaline catalyst which if it reacts with free fatty acids it will experience saponification so it needs to be pre-treated on the raw materials. In this study, the handling of FFA uses an adsorbent from activated charcoal from palm oil shells, this refers to the study of Fadillah et al. (2017) which found that adsorbents from activated charcoal from oil palm shells had reduced effectiveness of 91.82%, this percentage is significant compared to coir-rice husk which is 57.06% and activated charcoal from coconut shell by 33.33%. Transesterification is a slow reaction process because it takes place through a two-phase system. This can be overcome by adding unreactive co-solvent. The purpose of adding co-solvent is to form a solution system from two phases into one phase. The use of Co-solvent THF is to overcome the difference in solubility, in this case, THF is the best co-solvent; cheap, non-reactive, low boiling point (67°C), and non-toxic, therefore it can be separated by co-distillation with methanol and can be recycled at the end of the reaction Sidi et al. (1996). THF also has hydraulic and hydrophobic properties and can bind water and alcohol for its hydrophilicity, while its hydrophobicity can dissolve organic compounds Ontario et al. (2003). In this study, the addition of Co-solvent THF in the transesterification reaction of making biodiesel from used cooking oil is strongly influenced by the reaction and the molar ratio of the resulting product.

2. MATERIAL AND METHOD OF STUDY 2.1. MATERIALS AND TOOLS

Three neck flasks, return cooler, magnetic stirrer, thermometer, a set of distillation apparatus, cooler, stopwatch, Pyrex brand of glassware, hotplate, filter paper, separating funnel, analytical balance, oven, Otswald viscometer, and pycnometer. Used cooking oil, potassium hydroxide, methanol, tetrahydrofuran, palm shell activated charcoal, demineralized water, and phenolphthalein indicator.

2.2. PROCEDURE OF STUDY 2.2.1. PRETREATMENTOF USED COOKING OIL

The raw material of used cooking oil was obtained from mobile fried food vendors. Free Fatty Acid (FFA) levels were reduced by being adsorbed for 24 hours with 35 mesh palm shell activated carbon, then titration was carried out to determine the FFA levels in the sample using a titrant of 0.1 N KOH solution and the use of phenolphthalein as an indicator. If the FFA content is less than 0.5% then the raw material could be continued to the transesterification stage.

2.2.2. TRANSESTERIFICATION STAGE

The temperature of the used cooking oil was increased to 60° C, after the temperature was reached, the oil was contacted with methanol, KOH catalyst, and Co-solvent THF which had been mixed in a separate container. The transesterification was carried out under 65° C operating conditions, 1%-wt KOH catalyst from oil, co-solvent 1:1% vol methanol, and stirring speed of 75-150 rpm. The molar ratio of oil: methanol = 1: 6 at various times of 5, 7, 10, 13, and 15 minutes. The best reaction time is indicated by the highest % yield and characteristics

according to SNI 7182:2015, the best reaction time is used as reaction time at molar ratio of 1:3, 1:6, 1:9, 1:12, and 1:15.

3. RESULT AND DISCUSSION 3.1. EFFECT OF ACTIVATED CARBON ON FREE FATTY ACID LEVELS

In the pretreatment of used cooking oil derived from crispy fried foods at mobile fried food vendors, the free fatty acid content was reduced by the adsorption method using activated charcoal from palm oil shells. The temperature used for frying is not too high for a relatively short time so that the free fatty acid content formed is slight at 1.130%. After going through adsorption, the FFA content decreased to 0.512%. FFA reduction by adsorption method is more economical and easier and more effective than FFA reduction by esterification using acid and methanol catalysts Usman et al. (2019). Oil with 0.512% FFA content has met the requirements for the transesterification stage.

3.2. EFFECT OF REACTION TIME AND MOLAR RATIO ON % YIELD

3.2.1. EFFECT OF REACTION TIME ON % YIELD

Observation of the effect of reaction time on % yield of biodiesel produced from the transesterification process with fixed variable operating conditions; amount of feed (70 ml), reaction temperature (65°C), stirring speed (75-150 rpm), molar ratio of oil and methanol (1:6), amount of co-solvent (1:1 %vol methanol), weight of catalyst (1 %wt of oil), while the variable is the reaction time (5, 7, 10, 13, and 15 minutes).



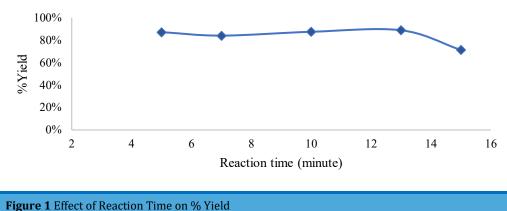


Figure 1 shows that % yield tends to increase slightly with increasing reaction time. The addition of time provides more opportunities for collisions between reactants so it gives more opportunities to react. The more volume of product produced, the % yield increases. After the best reaction time was reached, the % yield began to decrease, and an equilibrium occurred, so the addition of reaction time exceeding 13 minutes resulted in a decrease in % yield. The decrease occurs because the transesterification reaction is a reversible reaction so when the reaction has reached equilibrium, the addition of reaction time causes the reaction to shift towards the reactants (to the left) and results in a decrease in % yield (Setyawati et

al, 2012). At the reaction time of 5 minutes, the yield reached 87.1429%, while at the reaction time of 7 minutes the yield decreased to 84.0373% and the yield increased again to 87.4534% in 10 minutes, this is because, at the reaction time of 5 minutes the cooking oil has not been used yet completely converted into biodiesel, this refers to the characteristics of biodiesel such as the amount of density and viscosity. The product densityat 5 minutes has the same density as the raw material, which is 0.92 g/ml and has a high viscosity of 14.9928 CST. Thus, at the reaction time of 5 minutes, the raw material has not been properly converted into biodiesel. However, at a reaction time of 7 minutes, the density and viscosity of the biodiesel product were in accordance with SNI 7182:2015, so it could be said to be well converted, where % Yield continued to increase at a reaction time of 13 minutes until it reached 88.8199%, but at 15 minutes conversion decreased to 71.2733%. The best reaction time was obtained at 13 minutes and the co-solvent was very influential compared to without the use of co-solvent, which generally required a minimum of 1 hour to achieve the same yield.

3.2.2. EFFECT OF MOLAR RATIO ON % YIELD

Observations on the effect of the molar ratio of reactants on % yield of biodiesel produced in the transesterification process, the fixed variables used were the best reaction time taken from the previous data (13 minutes), reaction temperature (65oC), amount of feed (70 ml), stirring speed (75-150 rpm), the weight of catalyst (1%wt of oil), and amount of co-solvent (1:1% vol methanol). Meanwhile, the independent variable is the molar ratio of oil and methanol, namely 1:3, 1:6, 1:9, 1:12, and 1:15.

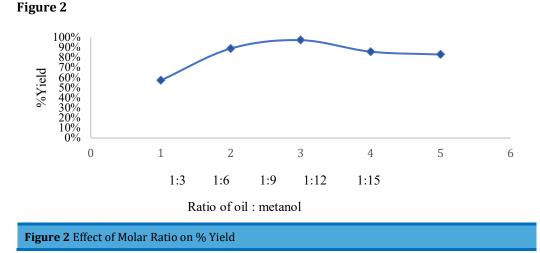


Figure 2 shows a significant increase in yield with an increase in the ratio of reactants to 1:9. In a reversible reaction, the addition of excess methanol will push the reaction to the right, so that the conversion of oil into biodiesel increases Razak et al (2015). In this study, the highest yield was obtained at the oil: methanol ratio of 1:9 with a yield of 97.1429%, then the graph decreased at the molar ratio of 1:12 and 1:15. The transesterification reaction is an equilibrium reaction so that if equilibrium has been reached, the addition of moles of methanol and the reaction time does not affect the increase in yield of methyl ester. Excessive use of methanol will cause an increase in the formation of glycerol in proportion to the formation of methyl esters (biodiesel), but glycerol will dissolve in excess methanol, making it difficult to separate biodiesel and glycerol Zukhriyah et al. (2022).

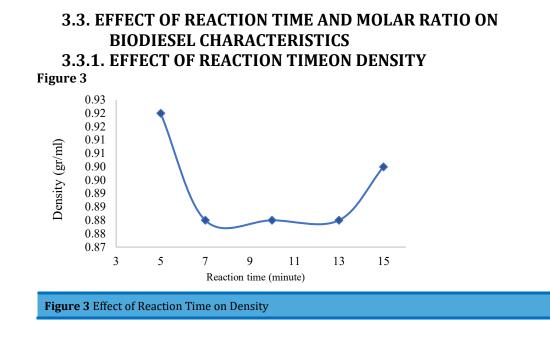
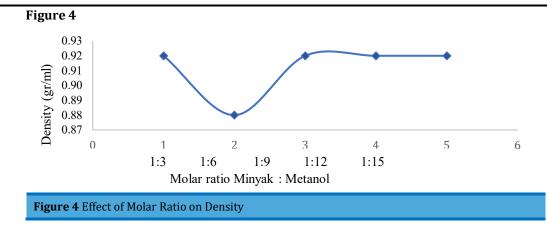


Figure 3 shows the relationship of the effect of reaction time on product density. It can be explained that at the beginning of the treatment the density was still too high, similar to the density of the raw material, which was 0.92 gr/ml, but at reaction times of 7, 10, and 13 minutes the density was consistent at 0.88 gr/ml which complied with SNI 7182:2015 biodiesel. At the reaction time of 15 minutes the density increased to 0.90 g/ml, this was related to a decreased yield, allowing the presence of other compounds such as the length of the carbon chain, therefore the greater the number of double bonds, the higher the density value of biodiesel Yanowitz et al. (2016). However, the accuracy of the balance used is only 1 digit behind the comma, making it difficult to read the weight, but the density of this product is close to SNI 7182:2015 biodiesel. Thus the biodiesel obtained has met the biodiesel density standard.

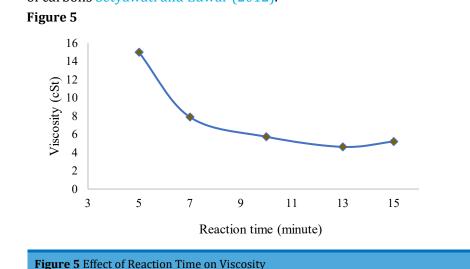
3.3.2. RELATIONSHIP BETWEEN MOLAR RATIO AND PRODUCT DENSITY

Figure 4 Shows that the product that meets the quality requirements of SNI 7182:2015 is only at a ratio of 1: 6, which is 0.88 gr/ml. This is because the use of too little methanol (1 : 3) has not been able to convert the raw material into biodiesel, so it has similar characteristics to the raw material, namely 0.92 g/ml, while the product has a larger density of 0.92. gr/ml has an indication that the reaction is not perfect in the conversion of oil. This condition corresponds to the % yield obtained which allows the presence of impurities in the product, such as residual catalyst and methanol, glycerol, soap, water, and fatty acids that are not converted to methyl esters or other content in the product that can affect the density of the product. biodiesel Setyawati and Edwar (2012).



3.3.3. REACTION TIME TO PRODUCT VISCOSITY

Figure 5 shows that in this study kinematic viscosity was found. The value of kinematic viscosity tends to decrease with the length of reaction time. According to Setyawati and Edwar (2012), the length of reaction time indicates that the resulting product has a mixed composition consisting of compounds with a smaller number of carbons Setyawati and Edwar (2012).



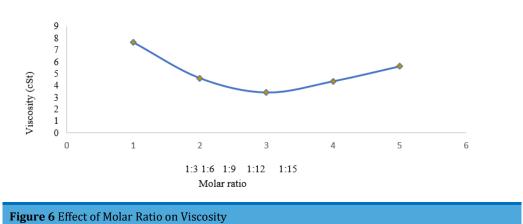
At the reaction time of 5 minutes, the conversion was not perfect so the viscosity value was high (14.9928 cSt), but at the reaction time of 10 to 15 minutes, the viscosity value met the requirements and quality of SNI biodiesel of 4.6238-5.7336 cSt. The test results showed that the biodiesel yield in the transesterification process at 10, 13, and 15 minutes was in the range of 2.3 - 6.0 mm^2 /s. This indicates that some biodiesel produced are in accordance with the viscosity value according to the SNI 7182:2015 standard.

3.3.4. RELATIONSHIP BETWEEN MOLAR RATIO AND PRODUCT VISCOSITY

Figure 6 shows the molar ratio to viscosity. It can be observed that the viscosity value is in the range of SNI 7182:2015 biodiesel, this indicates that at a reaction time of 13 minutes, the variation of the methanol molar ratio tends to be stable, except in the 1:3 molar ratio the mixture viscosity of 7.5840 cSt exceeds the biodiesel

standard, this is due to these conditions the yield has only reached 57.1429% so that the product mixture still contains a lot of triglycerides.





Viscosity is a very important property in the storage and the use of fuel. Too high viscosity affects the performance of the fuel injection device and makes it difficult to ignite, causing poor atomization and increasing engine deposits Setyawati and Edwar (2012). However, too low a viscosity can also result in a leak in the fuel injection pump. The viscosity of the fuel also has a direct effect on the ability of the fuel to mix with air, causing incomplete combustion.

3.3.5. EFFECT OF REACTION TIME AND MOLAR RATIO ON MOISTURE CONTENT

In the process of making biodiesel, it allows for the emergence of a mixture of water. This mixed water may come from raw materials, catalysts, and during the purification process. However, the amount of water in the raw material (used cooking oil) is very small due to the hydrolysis reaction when frying repeatedly in cooking oil, thus breaking down H_2O compounds and reacting with other compounds to form new compounds. KOH catalyst has hygroscopic properties so it has a good ability to absorb water molecules when exposed in the open Sangha et al. (2005). In the purification process, demineralized water is used as the purification medium, so the possibility of too fast separation between biodiesel and purified water will result in a large amount of water in biodiesel. Therefore, at the end of the purification stage, the product is dried by heating it at a temperature of 110°C to evaporate the remaining water in the product.

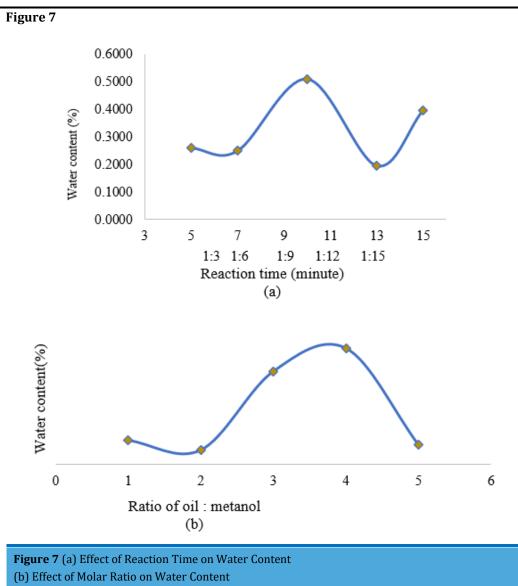


Figure 7 shows the relatively high-water content. The standard water content allowed for biodiesel is a maximum of 0.05%. The high-water content is caused by several technical errors such as poor separation during the washing process of biodiesel with demineralized water, drying biodiesel that is not long enough so that the water content is still high in biodiesel, incomplete drying of biodiesel products that have gone through the washing process, and a balance with high accuracy is required for the accuracy of weighing the product.

Water can exist in free form and can cause damage to the interior of the furnace surface during combustion especially if it contains dissolved salts. Water can also cause sparks at the tip of the burner which can extinguish the flame, lower the flame temperature, or prolong ignition. However, water content is not a big problem in diesel vehicles that have a water sediment filter. Water sediment has a function as a filter to separate water from fuel, so that diesel oil entering the combustion chamber remains free of water (Final B30, Ministry of Energy and Mineral Resources, 2020).

3.4. FLASH POINT AT MOLAR RATIO OF 1:6 AND REACTION TIME OF 13 MINUTES

The flash point of biodiesel according to SNI is at least 100 °C and based on the Decree of the Director General of EBTKE No.189/2019 the flash point of biodiesel is at least 130°C, so it is within safe limits against fire hazards while in storage, handling, and transportation below that temperature. Based on the data of the study, the flash point is 178.3°C, this number is a safe number because it is in accordance with the standards and quality of biodiesel, so it is safe in handling and storage. The more water content in the fuel, the more energy needed to evaporate the water so that the flash point will be higher, and vice versa Fadilah et al. (2019).

3.5. CALORIFIC VALUE AT MOLAR RATIO OF 1:6 AND REACTION TIME OF 13 MINUTES

In the calorific value test, it was found that the calorific value under the best conditions (molar ratio 1:6 and reaction time 13 minutes) was 7.891.4335 Cal/gr or equivalent to 33.0398 MJ/kg while the calorific value of diesel fuel (diesel) which is 44 MJ/kg. This low calorific value can be caused because biodiesel does not contain aromatic compounds like diesel fuel, but methyl esters with different levels of saturation. Unsaturated esters have lower energy content by weight but because of their higher density, they have more energy per unit volume Wahyuni et al. (2011). The amount of unsaturated ester content has a calorific value that is not too high, the composition of this ester can be identified by GC-MS testing.

3.6. CETANE NUMBER OF BIODIESEL PRODUCTS

The cetane number can be shown by itself in the diesel engine combustion chamber when the fuel ignites, this is with a high cetane number there will be an acceleration of combustion and the thermodynamic efficiency is get better.

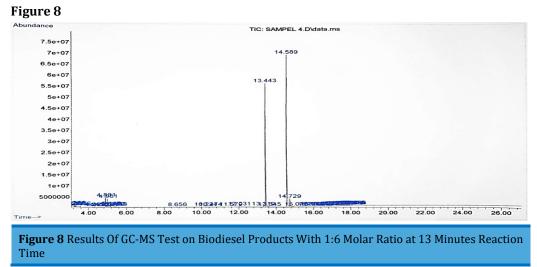
The cetane number test was carried out on biodiesel products under the best conditions, the biodiesel cetane number was 66.9. This figure shows the feasibility of the product to be used as diesel fuel because this figure has met the SNI biodiesel, which is at least 51. The cetane number obtained is also equivalent to previous studies where biodiesel has a cetane number of 62 for palm oil-based biodiesel, 51 for Jatropha, and 62.7 for coconut-based vegetables Soerawidjaja (2003).

The composition of fatty acids in biodiesel, such as long-chain saturated fatty acids (arachidic, lauric, myristic, palmitic, and lauric acids), is a high cetane number composition. Thus, the fatty acid composition is closely related to the cetane number of biodiesels. The cetane number has a significant effect on timing, on the fuel injected, and will cause a smooth engine noise and a good start.

3.7. COMPOSITION OF CHEMICAL COMPOUNDS IN TESTING USING GC-MS

Composition testing with GC-MS was carried out at a 1:6 molar ratio and a reaction time of 13 minutes. It was obtained that the resulting product contains the most methyl esters at 87.24%, while other compounds are 12.76%. This proves that the resulting product is biodiesel due to its high methyl ester content. The composition of this biodiesel product is shown in Figure 8. Figure 8. shows the readings of the composition of the methyl ester types tabulated in Table 1. Results

of the GC-MS show that there are 2 main peaks that have the largest content in the product composition, namely Oleaic (9(Z)-Octadecenoic) acid or oleic acid of 49, 33% at a retention time of 14,589 minutes and Palmitic (Hexadecanoic) acid or palmitic acid of 34.08% at a retention time of 13,443 minutes, the composition of these compounds affects the high cetane number in the product.



The methyl ester content affects the measurement of the calorific value and cetane number of biodiesel products, where oleic acid is an unsaturated fatty acid, so it has a low calorific value but the content of palmitic acid which is a saturated fatty acid in principle has a high calorific value which can balance the calorific value so it does not too low.

Compound Composition (%)
49,33
34,08
3,44
0,16
0,18
0,05
12,76
100

In this study, the biodiesel produced has a calorific value of 7,891.4335 cal/gr. Referring to the research of McCormick et al. (2001) which measured the cetane number of oleic acid methyl ester that the value was 59.3, while palmitic acid was measured in the study of Knothe et al. (2005), the CN was 74.5. This figure corresponds to the measurement of the CN biodiesel in this study, which is 66.9.

4. CONCLUSION

Co-solvent Tetrahydrofuran was very influential in the manufacture of biodiesel in shortening the time, biodiesel has started to convert well at a reaction time of 10 minutes. Meanwhile, transesterification without the use of co-solvent takes a long time (+1 hour) to convert raw materials into biodiesel products.

Based on the effect of reaction time, it is concluded that the longer the reaction time, the greater the conversion. The best reaction time is at a reaction time of 13 minutes with a %yield of 88.8199%. When entering the reaction time of 10 minutes the density and viscosity tend to be stable and meet SNI 7182:2015 with a stable density at 0.88 gr/ml and viscosity in the range of 4.6238 cSt-5.7336 cSt. Meanwhile, based on the molar ratio of density and stable viscosity, it is obtained only at a molar ratio of 1:6 which is in accordance with the characteristics of biodiesel based on SNI 7182:2015 which has a density of 0.88 gr/ml and a viscosity of 4.6238 cSt.

Based on the GC-MS test, the methyl ester content in biodiesel products was 87.24%, dominated by Oleaic (9(Z)-Octadecenoic) acid or oleic acid at 49.33% and Palmitic (Hexadecanoic) acid or palmitic acid of 34.08%.

CONFLICT OF INTERESTS

None.

ACKNOWLEDGMENTS

None.

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