

Effect of Time on the Quality of Biodiesel from Cooking Oil Used Using Lignite Coal Catalyst with Assistance Ultrasonic

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Abstract— Waste from used cooking oil can cause harm to various aspects of our lives. Efforts to reduce used cooking oil waste are by producing biodiesel. Biodiesel production is carried out using the transesterification method of oil and methanol with the help of a heterogeneous catalyst. One alternative material that can be developed as a heterogeneous catalyst is low-rank coal. This research aims to determine the quality of biodiesel produced from used cooking oil by varying the transesterification time using a low-rank coal catalyst with the help of ultrasonic waves. Used cooking oil and methanol (1:2) were transesterified at a temperature of 60°C with varying times of 25 minutes, 30 minutes, 35 minutes, and 40 minutes using 2 grams of low-rank coal catalyst with the help of ultrasonic waves with a frequency of 47 kHz. This research produced biodiesel with the highest yield of 77.78% at a transesterification time variation of 35 minutes with characteristics of a density of 40°C of 859.5 kg/m³, a kinematic viscosity of 40°C of 4.01 cSt, a water content of 0.026% and a number acid 0.44 mgKOH/g. In general, the biodiesel produced meets SNI 7182:2015.

Keywords— Biodiesel, low-rank coal, transesterification, ultrasonic, used cooking oil.

I. INTRODUCTION

Indonesia plays a strategic role in the global food system through plantations and agriculture. According to the Central Statistics Agency (BPS), in 2022, the area of rice fields will reach 7.46 hectares involving around 27.68 million farmers. This makes Indonesia the main producer of several world strategic agricultural commodities such as palm oil. Indonesia became the world's largest producer of palm oil in 2019, and one of its products is cooking oil. Long-term and repeated use of cooking oil can cause various fatal diseases [1]. Therefore, efforts are needed to process used cooking oil waste into something useful, one of which is converting used cooking oil waste into biodiesel.

Biodiesel is produced from a transesterification process using vegetable oil or animal fat as raw materials which is reacted with alcohol compounds such as methanol and a catalyst is added to increase the conversion. The added catalyst can be a heterogeneous catalyst (solid) or a homogeneous catalyst. Heterogeneous (solid) catalysts have advantages compared to homogeneous catalysts, namely that they do not mix with the product so the purification process is easier. One example of a heterogeneous catalyst is low-rank coal (lignite).

Low-rank coal is a quality of coal that is often found in Indonesia. One example of low-rank coal is lignite (brown coal) which has high water content, low calories, and high burning properties. With the large number of coal mining industries in Indonesia and looking at the existing brown coal reserves. Based on data from the Geotechnology Research Center-LIPI in 2015 it reached 86.11% of the total coal reserves in Indonesia.

In general, the efficiency and productivity of the biodiesel production process can be improved by reducing the reaction time or lowering the reaction temperature through several new technological innovations. One way is with the help of ultrasonic waves. The use of ultrasonic waves is one way that can be done to speed up reaction time and increase yield [9]. Thus, this research is focused on determining the quality of biodiesel from used cooking oil, with a low-grade coal catalyst using ultrasonic waves. By using this catalyst, it is hoped that it will be able to increase the conversion of used cooking oil into biodiesel which is useful as a cheap and affordable alternative fuel.

II. RESEARCH METHODS

Research Tools and Materials

The tools used in this research were a 200 mesh sieve, crusher, stirring rod, bulb, burette, porcelain cup, funnel, separating funnel, Erlenmeyer, FTIR, chemical beaker, measuring cup, hot plate, watch glass, magnetic stirrer, digital balance, Oven, pycnometer, dropper pipette, measuring pipettes, submersible pumps, hoses, states & clamps, thermometers, and Ostwald viscosity. Meanwhile, the materials used in this research are aluminum foil, distilled water, low-rank coal, ethanol p.a., PP indicator, H₂SO₄ solution, oxalic acid solids, NaOH solids, technical methanol, and used cooking oil.

Research path

The research was carried out in the basic laboratory of the Chemical Engineering Department of Samarinda State Polytechnic and the testing was carried out at the Undip Integrated Laboratory Technical Implementation Unit (UPT) to test the characteristics of lignite coal catalysts. The coal catalyst is first activated and then its characteristics are tested. The used cooking oil and biodiesel are analyzed at the integrated laboratory of the East Kalimantan Institute of

Technology (ITK).

Schematic Diagram of Work Procedures

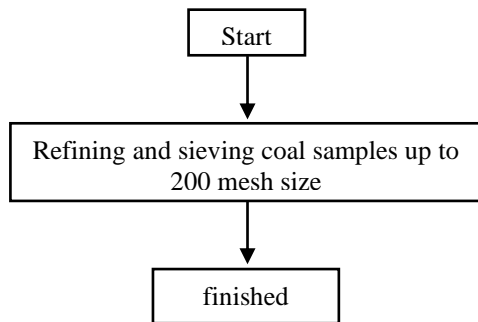


Fig. 1. Flow diagram for making catalysts from low-rank coal

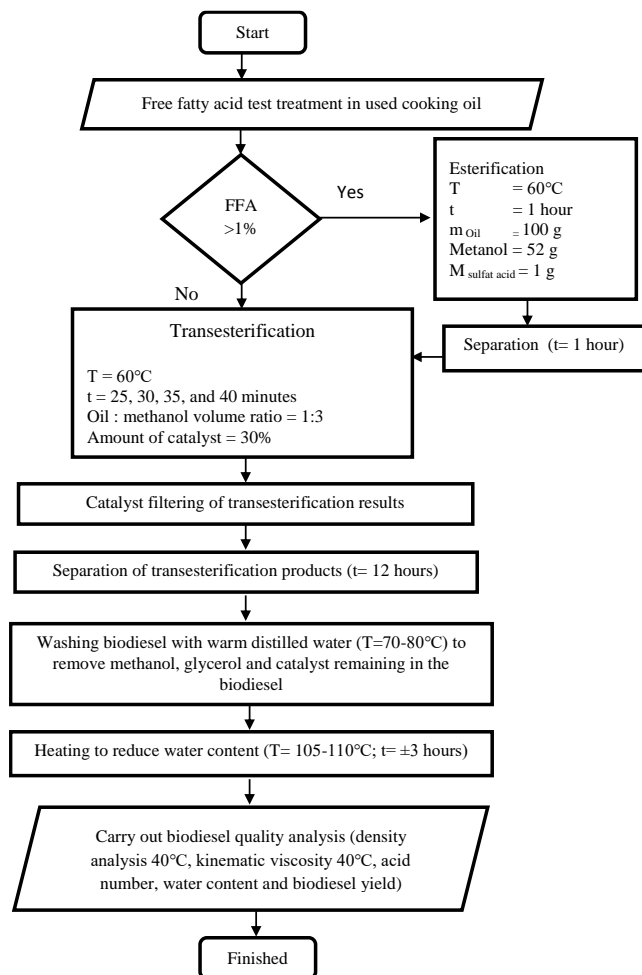


Fig. 2. Biodiesel Manufacturing Flow Diagram

Work procedures

□ Production of low-rank coal as a catalyst

1. Prepare low-rank coal.
2. Analyze coal samples with the parameters of water content, ash content, volatile matter, fixed carbon, total sulfur, and heating value.
3. Grind the coal using a blender, then sift the coal to 200 mesh.

□ Production of Biodiesel

1. Collect used cooking oil.
2. Analyze used cooking oil, including water content, density, kinematic viscosity, FFA content, and acid number.
3. If the FFA (Free Fatty Acid) level exceeds 1%, an esterification reaction must be carried out [5]
4. Separate the esterification results in a separating funnel for 1 hour, then take the bottom layer to be used in the transesterification reaction [11]
5. Carry out a transesterification reaction by reacting the esterified oil with a catalyst-methanol mixture at a temperature of 60-70°C for 25 minutes, 30 minutes, 35 minutes, and 40 minutes where 2 grams of catalyst are added with the help of ultrasonic waves to speed up the rate. transesterification reaction. The ratio used for oil: ethanol is 1:2 v/v [12]
6. Filter the catalyst along with separating the transesterification results in a separating funnel for 12 hours [12]
7. Take the top layer then heat it on a hot plate, at a temperature of 60-70°C for ± 1 hour to remove the remaining methanol.
8. Wash the biodiesel using distilled water at a temperature of 70-80°C until the water becomes clear.
9. Put the biodiesel in the oven for ±3 hours to reduce the water content in the biodiesel.
10. Save the biodiesel results that have been obtained.

III. RESEARCH RESULTS AND DISCUSSION

Research result

TABLE 1. Results of Analysis of Used Cooking Oil Characteristics

| Parameter | Analysis results |
|---------------------------------|-------------------------|
| Free Fatty Acid Content (%) | 1.51 |
| Acid number, mgKOH/g | 2.9986 |
| Density 40°C, Kg/m ³ | 898.5 |
| Kinematic Viscosity 40°C, cSt | 35.4 |
| Color | Abnormal (Yellow Brown) |

TABLE 2. Results of Biodiesel Analysis

| Parameter | Results of Biodiesel Analysis of Transesterification Time Variations | | | | SNI 7182 :2015 |
|---------------------------------|--|------------|------------|------------|----------------|
| | 25 Minutes | 30 Minutes | 35 Minutes | 40 Minutes | |
| Density 40°C, kg/m ³ | 851.6 | 855.0 | 859.5 | 855.5 | 850-890 |
| Kinematic Viscosity 40°C, cSt | 9.20 | 7.56 | 4.01 | 4.58 | 2.3-6.0 |
| Water Content, % (v/v) | 0.22 | 0,327 | 0,026 | 0.17 | 0.05 |
| Acid number, mgKOH/g | 1.33 | 1.45 | 0.44 | 2.76 | 0.5 |
| Yield, % | 54.53 | 66.68 | 77.78 | 49.90 | - |
| Free Fatty Acids (%) | 0.67 | 0.73 | 0.24 | 0.87 | - |

Discussion

In this research, biodiesel was made from used cooking oil using a low-rank coal catalyst, with varying transesterification times of 25 minutes, 30 minutes, 35 minutes, and 40 minutes to determine the effect of transesterification time on the methyl esters in the oil.

The initial stage of making biodiesel is that the oil is analyzed using the parameters of free fatty acid content, acid

number, density at 40°, kinematic viscosity at 40°, and color. This analysis is to determine the condition of used cooking oil before it is used as raw material, as well as to determine the reaction stages used in making biodiesel.

In Table 2 above, the parameter results for the free fatty acid content in used cooking oil are 1.51%, where according to [5], if the free fatty acid content exceeds 1%, biodiesel production is carried out in two reaction stages, namely esterification. and transesterification reactions.

The esterification reaction is the process of converting fatty acids into esters, namely by reacting fatty acids with alcohol using a strong acid catalyst [1]. This reaction aims to reduce the level of free fatty acids in the oil, where the lower the fatty acids, the lower the possibility of soap formation so that the biodiesel yield will be greater. The esterification reaction in this research was carried out based on research [11], where in this research alcohol was used with methanol to which the H₂SO₄ catalyst was added. Methanol is used because it is cheap, more reactive, and able to react at lower temperatures compared to other types of alcohol [8]. According [14] this H₂SO₄ catalyst can accelerate the rate of the esterification reaction, thereby increasing the conversion of fatty acids into esters.

Meanwhile, the transesterification reaction is a reaction between triglycerides and alcohol with the help of a catalyst. This reaction produces the main product in the form of alkyl ester/biodiesel and a side product in the form of glycerol [7]. In this reaction, an oil: methanol ratio of 1:2 is used, which refers to research [12]. The purpose of adding large amounts of methanol is to shift the reaction towards the product so that biodiesel yield can increase [13]. Increasing biodiesel yield can also be done by adding a catalyst to the reaction, where the catalyst used is a low-rank coal catalyst. The resulting catalyst is then used in the transesterification reaction which lasts for 25 minutes, 30 minutes, 35 minutes, and 40 minutes. The transesterification results obtained are then purified through separation, washing, and drying stages to obtain the desired results.

The separation and purification stage begins with filtering the catalyst and separating the glycerol in a separating funnel for 12 hours [12]. The top layer obtained is then heated for ± 1 hour at a temperature of 60-70°C to remove the remaining methanol. After that, the process continues by washing the biodiesel using distilled water at a temperature of 70-80°C until the water becomes clear [7] The washed biodiesel is then heated in an oven at a temperature of 105-110°C for 3 hours to reduce the water content of the biodiesel [7]. In this way, biodiesel products with high purity can be obtained.

Effect of Transesterification Time on Biodiesel Yield

Varying transesterification times, namely 25 minutes, 30 minutes, 35 minutes and 40 minutes in the transesterification reaction, have a real influence on the amount of oil produced as shown in Fig. 1:

Based on Figure 1, the addition of time in the transesterification reaction shows an increase in the yield produced, where the highest yield was obtained at 35 minutes,

namely 77.78%. By varying the transesterification time, you can produce an optimal reaction time so you can get a higher yield. However, at 30 and 40 minutes the yield of biodiesel produced decreased due to the formation of soap which occurred through the saponification reaction, where the formation of soap can hinder the conversion of oil into methyl ester [8]. According to [2], in producing high biodiesel yields, the washing process and water separation after washing are also very influential. Due to the biodiesel washing process, the water content produced exceeds SNI 7182:2015. The high value of kinematic viscosity is also caused by the presence of impurities in biodiesel that do not react, therefore it is necessary to carry out a purification process first.

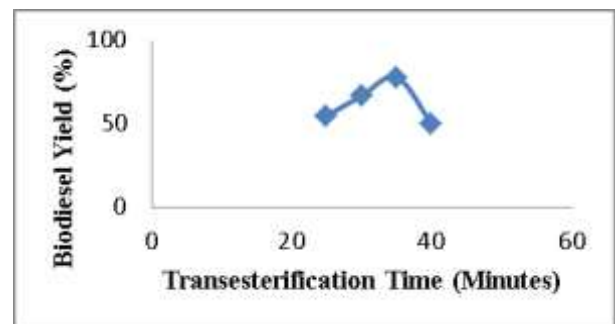


Figure 3. Effect of Transesterification Reaction Time on Biodiesel Yield

Effect of Transesterification Time on the Characteristics of the Biodiesel Produced

The quality of the biodiesel produced can be seen in Table 2 with analysis parameters including density at 40°C, kinematic viscosity at 40°C, water content, and acid number. To vary the transesterification time with the highest yield (35 minutes), additional analysis was carried out in the form of methyl ester content to determine the level of purity of the biodiesel produced.

Based on Table 2, the density of biodiesel obtained ranges from 851.6-859.5 kg/m³. These results have met the density standards in SNI 7182:2015, namely between 850-890 kg/m³. This is caused by an increase in the conversion rate of triglycerides into methyl esters so that the density of biodiesel will decrease. Biodiesel with a density according to SNI is capable of producing perfect combustion [5]. Meanwhile, the viscosity produced experienced a significant increase due to low and long heating, so the viscosity of biodiesel did not decrease [16] and according to [15] the high viscosity was caused by used cooking oil, not A purification process is carried out where the higher the viscosity, the thicker it is and the more difficult it is for the material to flow. High viscosity will cause the fuel to atomize into larger droplets and tend to collide with the relatively cold tank [16]. The water content parameters of biodiesel products are still above the permitted limits. High water content can be caused by an incomplete evaporation process [6]. The water contained in the product is caused by residual water from product purification which will hinder the distribution of fuel to the piston [6]. Meanwhile,

according to the acid number parameter [17] the high acid number in biodiesel indicates the presence of remaining free fatty acids. High acid numbers can cause corrosion in diesel engine fuel tanks [4]. The methyl ester content parameters were only reviewed for biodiesel with the highest yield, namely 77.78% from a variation of transesterification time of 35 minutes. Methyl ester analysis was carried out using an FT-IR instrument, with the resulting IR spectrum being as follows:

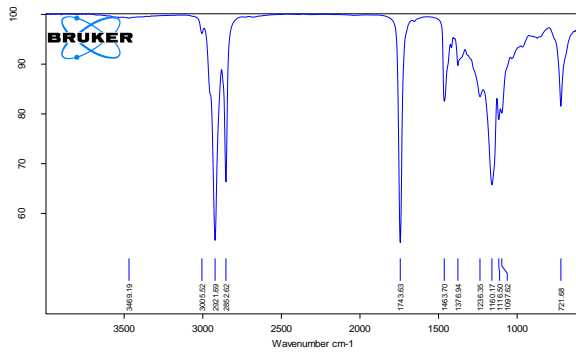


Figure 4. The IR spectrum of Biodiesel

In the spectrum above, there is a sharp absorption at the wavelength 1097.62; 1116.50; 1160.17; 1236.35; 1376.94; 1463.70; 1743.63; 2852.62; and 2921.69 cm⁻¹. Based on the book [3], the absorption wave numbers are 1097.62; 1116.50; 1160.17; and 1236.35 cm⁻¹ indicating the presence of C-O bonds (1300-1000 cm⁻¹). Meanwhile, the wave number absorption of 1376.94 and 1463.70 cm⁻¹ indicates the presence of a bending C-H bond (1474-1300), then the absorption of 1743.63 indicates the presence of an ester group, namely C=O (1900-1650 cm⁻¹). And the absorptions of 2852.62 cm⁻¹ and 2921.69 cm⁻¹ indicate the presence of C-H bonds (3000-2700 cm⁻¹). The C-O, C=O, and C-H bonds in biodiesel prove the presence of methyl ester compounds contained in them.

IV. CONCLUSION

From the research that has been carried out, several things can be concluded, including:

1. Based on this research, it is known that the function of coal as a catalyst is not effective in producing biodiesel according to SNI 7182:2015.
2. The density parameter of 40°C in the biodiesel produced meets SNI 7182:2015. Meanwhile, the kinematic viscosity parameters at 40°C, water content, and acid number do not meet SNI 7182:2015

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REFERENCES

- [1]. Ardhanay, S. ., & Lamsiyah. (2018). Level of Knowledge of Tent Stall Traders on Jalan Yos Sudarso Palangkaraya About the Dangers of Using Used Cooking Oil for Health. 06(1), 68–72.
- [2]. Busyairi, M., Muttaqin, A. Z., Meicahyanti, I., & Saryadi, S. (2020). The Potential of Used Cooking Oil as Biodiesel and the Effect of Catalysts and Reaction Time on Biodiesel Quality Through the Transesterification Process. *Serambi Engineering Journal*, 5(2), 933–940. <https://doi.org/10.32672/jse.v5i2.1920>
- [3]. Dachriyanus. (2004). Nalisi structure n a w a r g a n i c t o t r o s c o p i s.
- [4]. Efendi, R., Aulia, H., Faiz, N., & Firdaus, E. R. (2018). Production of Used Cooking Oil Biodiesel Using Esterification-Transesterification Methods Based on Amount of Used Cooking Oil Biodiesel Production From Waste Cooking Oil By Esterification-Transesterification Methods Based on Amount of Used Cooking Oil. *Industrial Research*, 7182, 2.4.
- [5]. Hadrah, H., Kasman, M., & Sari, F. M. (2018). Analysis of Used Cooking Oil as Biodiesel Fuel using the Transesterification Process. *Journal of Environmental Cycling*, 1(1), 16. <https://doi.org/10.33087/daurling.v1i1.4>
- [6]. Kasumaningtyas, R. D., & Bachtiar, A. (2013). Synthesis of Biodiesel from Rubber Seed Oil with Varying Temperature and Koh Concentration for the Transesterification Stage. *Journal of Renewable Natural Materials*, 1(2), 74683.
- [7]. Khoiruummah, D., Sundari, N., Zamhari, M., Yuniar, & Yuliati, S. (2020). Aplikasi Katalis Berbasis Karbon Aktif Dari Kayu Akasia (Acacia Mangium) Diimpregnasi Basa Pada Sintesis Biodiesel. *Politeknik Negeri Surabaya, Prosiding Seminar Mahasiswa Teknik Kimia*, 1(1), 20–28. <https://www.jurnal.polsri.ac.id/index.php/psmtk/article/view/2685>
- [8]. Kurniasih, E., & Pardi. (2017). Performance of NaOH and Zeolite/NaOH Base Catalysts in Biodiesel Synthesis as an Alternative Energy Source. *National Seminar on Science and Technology 2017*, November, 1–7
- [9]. Maisarah, Q. H., & Hidayati, N. (2019). Transesterification of Used Cooking Oil into Biodiesel Using Ultrasonic Waves and Na₂O/Fly Ash Catalyst. *Proceedings of the National Seminar on Chemical Engineering "Kejuangan."* 4(2019: PROCIDING SNTKK 2019), E3. <http://jurnal.upnyk.ac.id/index.php/kejuangan/article/view/2833>
- [10]. Moulita, R. N., Rusdianasari, R., & Kalsum, L. (2020). Biodiesel Production from Waste Cooking Oil using Induction Heating Technology. *Indonesian Journal of Fundamental and Applied Chemistry*, 5(1), 13–17. <https://doi.org/10.24845/ijfac.v5.i1.13>
- [11]. Oko, S., Kurniawan, A., & Rahmatina, J. (2021). Effect of Mass Comparison of Ca and C on the NaOH / CaO / C Catalyst in Biodiesel Synthesis Using Used Cooking Oil. *Proceedings of the 12th Industrial Research Workshop and National Seminar Bandung*, 1–6.
- [12]. Pasae, Y., Leste, J., Bulu, L., Tandiseno, T., & Tikupadang, K. (2019). Biodiesel production from waste cooking oil with catalysts from clamshell. *ARPN Journal of Engineering and Applied Sciences*, 14(3), 596–599.
- [13]. Prihanto, A., & Irawan, T. A. B. (2018). Effect of Temperature, Catalyst Concentration and Methanol-Oil Molar Ratio on Biodiesel Yield from Used Cooking Oil Through the Neutralization-Transesterification Process. *Methane*, 13(1), 30. <https://doi.org/10.14710/metana.v13i1.11340>
- [14]. Sutapa, I. W., A. B., Rosmawaty, & Mahulau, N. (2014). the Utilitation of Beef Tallow Into Biodiesel With Heterogenous Catalyst Pengolahan Lemak Sapi Menjadi Biodiesel Dengan Katalis Heterogen. *Ind. J. Chem. Res.*, 166–170.
- [15]. Wahyuni, S., Ramli, & Mahrizal. (2015). The Effect of Process Temperature and Deposition Time on the Quality of Biodiesel from Student Used Cooking Oil
- [16]. Yunsari, S., Husaini, A., & Rusdianasari, R. (2019). Effect of Variation of Catalyst Concentration in the Production of Biodiesel from Crude Palm Oil using Induction Heater. *AJARCODE | Asian Journal of Applied Research for Community Development and Empowerment*, 3(1), 24–27. <https://doi.org/10.29165/ajarcde.v3i1.19>
- [17]. Zamhari, M., Junaidi, R., Rachmatica, N., Oktarina, A., Srijaya, J., Bukit, N., Palembang, B., & Selatan, S. (2021). Preparation of Activated Carbon Based Catalyst From Coconut Shell (Cocos Nucifera) Impregnated with Koh in the Transesterification Reaction.