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Impact of Temperature Variation on Yield and Quality of Biodiesel from Waste Cooking Oil Catalysed by Sulfonated Rubber Seed Shell Activated Carbon

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Abstract:
The energy crisis and environmental issues have increased interest in biodiesel as a sustainable fuel. This study examined the effect of reaction temperatures, namely 50°C, 55°C, 60°C, 65°C, and 70°C on the yield and quality of biodiesel produced from a simultaneous esterification transesterification reaction using a 2 %wt catalyst and a mass ratio of methanol to oil, 9:1 for 1 hour. The results showed that temperature has a crucial role in the yield and quality of the resulting biodiesel. The highest yield is 72.62%, was achieved at a temperature of 65°C, producing biodiesel that suitable to SNI 7182:2015 with a density of 875.60 kg/m³, a viscosity of 2.86 cSt, and an acid number of 0.0065 mg-KOH/g. Temperatures above 65°C decreased the yield and quality due to methanol evaporation and incomplete conversion.

Keywords: Biodiesel, Waste Cooking Oil, Temperature of Transesterification, Sulfonated Activated Carbon;

1. Introduction

The energy crisis and growing awareness of the environmental impacts of fossil fuel use have prompted the search for environmentally friendly alternative energy sources. The production of diesel fuel and other petroleum-based products is so high that petroleum resources are severely depleted. Based on the statement Brahma et al (2022), increasing industrialization, transportation, urbanization, and rapid population growth create enormous energy needs.

Biodiesel is an alternative that provides the most hope to deal with the problems that arise compared to conventional fossil fuels. Biodiesel production is through 2 (two) chemical reactions, namely esterification reaction and transesterification reaction. In the study, Monika et al (2023) stated that biodiesel is a Fatty Acid Methyl Ester (FAME) produced from fatty acid esters produced from vegetable oils or animal fats by meeting specified quality standards. Biodiesel can be used in its pure form as a diesel fuel or mixed with diesel oil in a wide variety of comparisons. In the biodiesel production process, transesterification is a crucial stage that is influenced by various factors, one of which is the reaction temperature. Optimal temperature is very important to increase reaction efficiency and obtain maximum biodiesel yield.

The process of esterification is a chemical reaction used to convert free fatty acids (Free Fatty Acids/FFA) becomes an ester through a reaction with alcohol, such as methanol or ethanol with the help of a catalyst. According to Megawati et al (2021), esterification is carried out when the level of free fatty acids (Free Fatty Acids/ FFA) in oil exceeds 2%, as high FFA levels can cause a soaking reaction if directly processed with an alkaline catalyst in transesterification. The transesterification process is the main method in converting vegetable oil or animal fat into biodiesel, where the oil reacts with alcohol, such as methanol or ethanol with the help of a catalyst. This reaction aims to break down the triglyceride molecules in the oil into short-chain esters, known as methyl esters or ethyl esters, depending on the type of alcohol used. Biodiesel can be made from vegetable oil, animal fat, or waste cooking oil. The increase in waste cooking oil production is an environmental problem that can cause problems for households and industries (Joseph et al, 2024).

Therefore, one of the potential raw materials for biodiesel production is waste cooking oil, whose use can reduce environmental pollution and reduce biodiesel production costs.

The use of catalysts, both homogeneous such as sodium hydroxide (NaOH) and potassium hydroxide (KOH) and heterogeneous such as sulfonated activated carbon, plays an important role in accelerating reactions and improving conversion efficiency. In addition, the use of catalysts also plays an important role in increasing the efficiency of transesterification reactions. Heterogeneous catalysts such as sulfonated activated carbon derived from biomass waste are a promising option because they are reusable and have a smaller environmental impact than homogeneous catalysts. According to Haryono et al (2024), the sulfonation process further adds a sulfonate bond ($-SO_3H$), which increases the acidity of the catalyst and makes it more effective in accelerating transesterification reactions. The main advantages of this catalyst are that they are reusable, easy to separate from the final product, and are more environmentally friendly than homogeneous catalysts. In addition, the use of rubber seed shell waste as a catalyst also supports the principle of the circular economy by reducing agricultural waste and creating added value in more sustainable biodiesel production.

Activated carbon catalysts are rated as heterogeneous catalysts that are widely used in reaction processes using acid catalysts, especially during biomass conversion. Sulfonated activated carbon catalysts are also able to be an alternative to acid solution catalysts because they have high acidity and are easy to recycle, so sulfonated activated carbon catalysts are quite promising in the biodiesel manufacturing process. Based on research Mirzayanti et al (2020), the sulfonation process using H2SO4 solution as carbon activation has a high strength of catalytic activity.

2. Material and Methods

Materials

This research used rubber seed shells were obtained from local rubber plantation and used cooking oil were obtained from households. The chemicals were of analytical grades.

Preparation of Sulfonated Rubber Seed Shell Activated Carbon Catalyst

The rubber seed shells are washed first, then, dried in a tray dryer, pounded, and put back in the oven at 110°C for 1 hour. Rubber seed shell powder in the previous stage was soaked in a KOH solution with a ratio of 1:1 (10 grams of rubber seed shell powder : 10 grams of KOH pellet) in 100 ml of aquadest at room temperature for 24 hours. Next, it is carbonized using a furnace with a temperature of 500°C for 3 hours. The activated carbon sulfonation process begins with mixing between activated carbon and sulfuric acid agent (H_2SO_4) 98% with a concentration ratio of 1:20 (1 gram of activated carbon: 20 ml H_2SO_4). Then, heat using a hot plate at 120°C for 6 hours. After that, the mixture is cooled to 25°C, the mixture is filtered through filter paper. Then, wash using aquadest until the pH of the washing water becomes neutral and dry using an oven with a temperature of 110°C for 1 hour.

FFA (Free Fatty Acid) of Waste Cooking Oil

Analyzing FFA content was carried out before and after the process of simultaneous esterification of transesterification using method described by Hartono et al (2023). The titration was carried out using the PP (Phenolphlatein) indicator and titrated using a solution of NaOH 0.1 N. The FFA content can be calculated using the following formula:

$$\text{FFA content (\%)} = \frac{\text{M x V x T}}{10.m} \text{ x } 100\%$$

Where:

M = Molecular weight of fatty acids (gr/mol)
T = Normality of NaOH (N)
m = Mass of biodiesel (gr)
V = Volume of NaOH required for titration (ml)

Simultaneous Esterification of Transesterification of Waste Cooking Oil into Biodiesel

At this stage, 2 (two) experiments were carried out, namely using a catalyst and without using a catalyst, as follows:

1. Simultaneous Esterification of Transesterification of Waste Cooking Oil into Biodiesel with Catalyst

Simultaneous esterification of transesterification is carried out inside a three-neck flask equipped with a reflux condenser. Then, add methanol with a ratio of waste cooking oil mass to methanol, which is 1:9 and add a sulfonated rubber seed shell activated carbon catalyst of 2 %wt. The mixture is refluxed by varying temperatures of 50°C, 55°C, 60°C, 65°C, and 70°C with a time of 1 hour and a constant stirring speed of 600 rpm. Next, the mixture is left for 24 hours until it separates into two (2) phases, biodiesel at the top and glycerol at the bottom. Then, the resulting biodiesel is washed using aquadest heated to 50°C.

2. Simultaneous Esterification of Transesterification of Waste Cooking Oil into Biodiesel Without Catalyst

Repeat the same steps as above by taking the most optimal operating conditions, i.e. at a temperature of 65°C for 1 hour and without the addition of sulforated activated carbon catalysts.

Product Analysis and Evaluation

The biodiesel produced was analyzed for its quantity (% yield) and its quality (density, viscosity, and acid number) using equations below (Permana et al (2020); Setiorini et al (2023); Aprilian & Purwaningtyas (2025)). Chemical composition analysis was carried out using Gas Chromatography-Mass Spectrometry (GC-MS).

$$\text{Yield (\%)} = \frac{\text{mass of biodiesel (g)}}{\text{mass of waste cooking oil (g)}} \text{ x } 100\%$$

$$\text{Density} = \rho \text{ (gr/ml)} = \frac{(\text{mass of biodiesel} + \text{mass of pycnometer}) - \text{mass of empty pycnometer}}{\text{volume of pycnometer}}$$

$$\text{M = Viscosity = viscosity of aquadest x } \frac{\text{flow time ofbiodiesel x density of biodiesel}}{\text{flow time of aquadest x density of aquadest}}$$

$$\text{Acid Number (mg-KOH/gr)} = \frac{\text{volume of KOH (ml)x normality of KOH (N) x 56,1}}{\text{weight of sample (mg)}}$$

3. Results and Discussion

Characteristics of Biodiesel from Used Cooking Oil with Activated Carbon Catalyst Sulfonated Rubber Seed Shell

In this study, the characteristics produced were influenced by the temperature of the simultaneous esterification transesterification reaction of biodiesel, namely at temperatures of 50°C, 55°C, 60°C, 65°C, and 70°C within 1 hour using an activated carbon catalyst of sulfonated rubber seed shells. For a deeper understanding of the influence of temperature on each variation in transesterified temperature, the content in biodiesel through GC-MS analysis, %yield, and biodiesel quality parameters are calculated to analyze and evaluate how optimal the waste cooking oil biodiesel produced is in accordance with existing quality standards, namely SNI 7182:2015.

Based on Table 1 which shows the results of biodiesel quality with temperature variations based on SNI 7182:2015 on the parameters analyzed and calculated, such as yield, density, viscosity, and acid number. At a temperature of 65°C, the quality of biodiesel in terms of %yield is the highest, which is 72,62%. The resulting density is 875,60 kg/m^3 which falls into the density range in accordance with SNI 7182:2015, which is 850 – 890 kg/m^3. The viscosity obtained is 2,86 cSt, which is in the range of 2,3 – 6,0 and the acid rate obtained at a temperature of 65°C is 0,0065 mg-KOH/gr. Therefore, the quality results of biodiesel at a temperature of 65°C with parameters, such as density, viscosity, and acid number fulfill the SNI 7182:2015 standard.

Table 1. Biodiesel quality and the reference (SNI 7182:2015)

Temperature (°C)	**Volume (mL)**	***Yield* (%)**	**Density (kg/m^3)**	**Viscosity (cSt)**	**Acid Number (mg-KOH/gr)**
Reference	-	-	850 – 890	2.3 – 6.0	0.5 (max)
50	27	53,59	837.60	2.09	0,0094
55	30	60,84	855.80	2.21	0,0080
60	33	66,83	854.60	2.67	0,0075
65	35	72,62	875.60	2.86	0,0065
70	25	50,25	848.20	2.25	0,0056

Initially yield biodiesel has increased, but, when the optimal temperature has been reached, yield biodiesel is decreasing. %yield *b*iodiesel will experience a decrease at too high temperatures, according to a statement Cahyo et al (2024) that temperatures passing 65°C will increase the volatility of methanol causing some of the reactants to evaporate before the simultaneous esterification transesterification reaction is complete and may accelerate the saponification reaction which can decrease %yield biodiesel. This study shows that temperatures above 65°C are ineffective and can reduce %yield biodiesel produced.

Based on the results of the biodiesel density test that has been carried out in this study, that at each temperature there is an increase in density but there is a decrease in the temperature that has passed the optimal temperature, which is 65°C. The increase in biodiesel density is influenced by the process of converting used cooking oil into biodiesel which is decreasing at high temperatures (Muarif et al., 2024). Viscosity values that are not in accordance with SNI 7182:2015 indicate that the simultaneous esterification reaction of transesterification does not take place perfectly, so that there are still triglycerides or other heavy compounds that are not converted into Fatty Acid Methyl Ester (FAME). In a statement submitted by Lin & Ma (2021) that the structure of compounds in biodiesel affects the viscosity of the biodiesel produced, where the high content of fatty acids tends to increase the viscosity of the fuel. Low acid levels in biodiesel indicate that the process of simultaneous esterification of transesterification is going well and free fatty acids (FFA) in waste cooking oil has been converted into Fatty Acid Methyl Ester (FAME). This is reinforced by the studies conducted Monde et al (2022), temperature can affect the formation of larger methyl ester fatty acids and a small amount of free fatty acids.

In this study, biodiesel quality parameters, such as density, viscosity, and acid number have increased but at temperatures that have exceeded 65°C they have decreased and do not enter the quality standard in accordance with SNI 7182:2015, based on the statement Haryono et al (2024) that transeterified temperatures that have passed 65°C will experience significant methanol evaporation and that emulsion formation can occur that makes it difficult to separate biodiesel and glycerol.

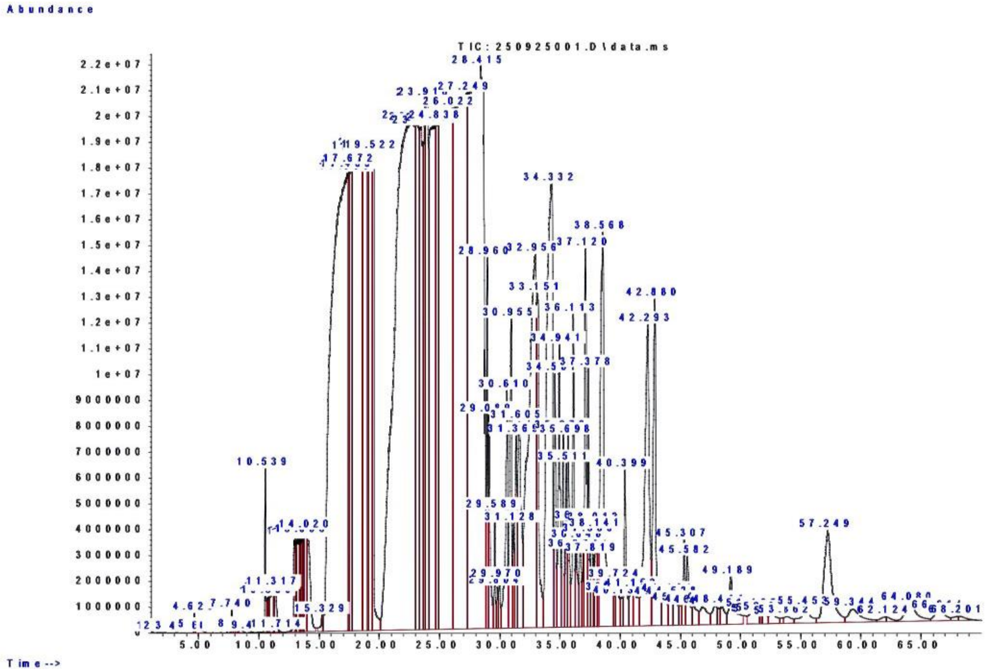
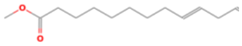
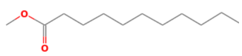
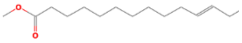
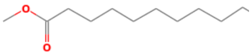
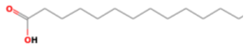
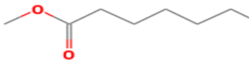


Figure 1. GC-MS biodiesel from used cooking oil with activated carbon catalyst sulfonated rubber seed shell

Based on the results of the GC-MS analysis in Figure 1, where the X axis is the retention time and the Y axis is the intensity which proves that the compound with the most %area is the compound 11-octadecenoic acid, methyl ester, which is 49,77%. The results of this GC-MS analysis show that methyl ester compounds dominate the content in biodiesel by 85,24% and the remaining 14,76% which are other compounds that are not converted to methyl esters, where there are several factors that occur, such as operating conditions during the simultaneous esterification process of transesterification and unstable laboratory electrical conditions. In the research conducted by Shah et al (2022) which produces a similar 78% content of methyl ester fatty acids, such as oleic acid, palmitic acid, myritic acid, lauric acid whose data are not explicitly mentioned. The 7 (seven) methyl ester compounds that dominate in biodiesel can be seen in Table 2 below.

Table 2. Methyl Ester Compounds from GC-MS biodiesel from used cooking oil with activated carbon catalyst sulfonated rubber seed shell

Senyawa	Retention Time	Rumus Molecule	Structure Molecule	Density (kg/m3)	%Area
11-Octadecenoic acid, methyl ester (oleic acid)	22,608	$C_{19}H_{36}O_2$		872,3	49,77
Hexadecanoic acid, methyl ester (palmitic acid)	17,356	$C_{17}H_{34}O_2$		853,0	21,73
11-Eicosenoic acid, methyl ester	32,956	$C_{21}H_{40}O_2$		871,0	6,83
Methyl Tetradecanoate, methyl ester (myristic acid)	12,971	$C_{15}H_{30}O_2$		862,0	3,27
Dodecanoic acid, methyl ester (lauric acid)	10,539	$C_{13}H_{26}O_2$		874,4	2,72
Nonadecanoic acid, methyl ester	29,589	$C_{19}H_{38}O_2$		886,0	0,83
Octanoic acid, methyl ester	4,629	$C_9H_{18}O_2$		900,0	0,09
Total					**85,24**

GC-MS analysis prove that the process of simultaneous esterification transesterification reaction of biodiesel from waste cooking oil with an activated carbon catalyst of sulfoned rubber seed shells at a temperature of 65°C has not met the standard because it only contains 85,24% methyl ester and biodiesel quality based on SNI 7182:2015 where the content of methyl ester components is at least 96,5%. With the content of methyl ester fatty acids that do not meet standards and quality parameters, such as density, viscosity, and acid number that meet SNI 7182:2015, this biodiesel is physically good, but chemically it does not meet the standards.

Biodiesel Characteristics of Used Cooking Oil without Activated Carbon Catalyst Sulfonated Rubber Seed Shell

In the research conducted, the manufacture of biodiesel without a catalyst cannot produce biodiesel because the manufacture of biodiesel without a catalyst requires supercritical operating conditions. Basically, the process of simultaneous esterification of transesterification is carried out by reacting oil, alcohol, and also catalysts. The reaction process without using a catalyst is carried out using the optimal conditions that have been obtained from the process of using a catalyst, namely at a temperature of 65°C with a time of 1 hour, but biodiesel is not successfully formed.

Based on the research that has been carried out, it is very possible that the cause of biodiesel is not formed due to too low temperatures and too short reaction times, this experiment requires supercritical operating conditions. According to a statement written by Ahmed et al (2023), the reaction occurs in supercritical conditions, namely at a temperature of 200°C – 300°C and at a pressure of 100 – 200 bar because in supercritical conditions it is able to break down triglyceride molecules and can trigger a constant transesterification reaction.

4. Conclusion

Temperature variations in the simultaneous esterification of transesterification reaction greatly affect the yield and quality of the biodiesel produced. The temperature of 65°C produces biodiesel with the best quality, this can be seen from the %yield of 72,62%, biodiesel density of 875,60 kg/m3, viscosity of 2,86 cSt and acid number of 0,0065 mg-KOH/gr. Therefore, all parameters that have been analyzed and calculated are within the quality range of biodiesel based on SNI 7182:2015. It is necessary to carry out further testing of the quality of biodiesel based on its quality standards, such as flash point testing, iodine number, cetane number, and other quality standard parameters. The aim of this further testing is to further strengthen the understanding of the variation in reaction temperature that is crucial to the quality of the biodiesel produced.

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