

Effect of sodium hydroxide (NaOH) catalyst variation on the transesterification results of used cooking oil

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ABSTRACT

Used cooking oil is one of the potential raw materials for producing biodiesel in order to reduce the impact of environmental pollution and diseases caused by its repeated consumption. This research aims to analyze the effect of variations in the concentration of the alkaline catalyst Sodium Hydroxide (NaOH) of 0.1% and 0.2% on the characteristics of the methyl ester resulting from the transesterification of used cooking oil, with test parameters covering Free Fatty Acid (FFA) content, acid number, density, pH, and yield. The method used was esterification with a 1% H₂SO₄ catalyst to reduce the initial FFA content of the used cooking oil from 3.6% to 0.7%, followed by a transesterification process using the reflux method at a temperature of 60°C–65°C for 30 minutes. The test results show that the 0.1% NaOH catalyst variation produced an FFA content of 0.4%, an acid number of 0.05 mg NaOH/g, a density of 875.5 kg/m³, a pH of 7, and a yield of 69%. Meanwhile, the 0.2% NaOH catalyst variation produced an FFA content of 0.6%, an acid number of 0.09 mg NaOH/g, a density of 871.5 kg/m³, a pH of 7, and a yield of 69%. Both variations have met the SNI 7182:2015 quality standard for the density and pH parameters, with the 0.2% catalyst variation providing optimal conversion efficiency.



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INTRODUCTION

The increasing consumption of vegetable cooking oil, particularly palm oil, in household activities as well as in the food industry significantly generates waste frying oil, or used cooking oil. The repeated use of used cooking oil for processing foodstuffs can endanger human health because of the formation of saturated fatty acids and toxic compounds that have the potential to trigger cancer (Nabiilah, 2024). On the other hand, discharging used cooking oil waste directly into the environment without prior treatment causes pollution of soil and water sources (Kusumaningtyas et al., 2018). Therefore, innovation is needed in utilizing used cooking oil waste into a product with high added value, one of which is as a raw material for producing biodiesel (Efendi et al., 2018). Biodiesel is a renewable, environmentally friendly fuel that has physicochemical properties similar to those of fossil-based diesel fuel (Atadashi et al., 2013). Used cooking oil generally contains a fairly high content of *Free Fatty Acid* (FFA) as a result of oxidation and hydrolysis processes during frying (Nabiilah, 2024). A high FFA content in the raw material can trigger a side reaction in the form of saponification if it is directly reacted through alkaline-catalyzed transesterification, which results in decreased efficiency and interferes with the methyl ester purification process (Andalia & Pratiwi, 2018). To overcome this obstacle, the conversion of used cooking oil into biodiesel requires a combination of two reaction stages, namely an esterification reaction with an acid catalyst to reduce the FFA content to below 2%, followed by a transesterification reaction with an alkaline catalyst to convert triglycerides into methyl ester (Siskayanti et al., 2019). The use of a sodium hydroxide (NaOH) catalyst in the transesterification stage is highly preferred because it has a high level of reactivity and a relatively fast reaction rate compared with other alkaline catalysts (Mukminin et al., 2023). Previous studies have examined variations in reaction time and the purification of used cooking oil in order to increase the methyl ester yield, such as the research by Militya (2022) and Rezablina et al. (2023)[cite: 1]. Nevertheless, the specific relationship between the use of low-dose NaOH catalyst concentrations (0.1% and 0.2% v/v) and the technical quality characteristics of the resulting biodiesel—covering FFA value, acid number, density, and *yield* still requires comprehensive analysis (Mukminin et al., 2022). The novelty of this research

lies in the evaluation of the effectiveness of using highly efficient NaOH catalyst concentration variations (0.1% and 0.2%) with a constant reaction time of 30 minutes at a temperature of 60°C–65°C in achieving the biodiesel quality standard based on SNI 7182:2015. This research aims to analyze the effect of variations in NaOH catalyst concentration (0.1% and 0.2%) on the transesterification results of used cooking oil and to test the physicochemical characteristic parameters of the resulting methyl ester, covering FFA content, acid number, density, pH value, and *yield*. The benefit of this research is to provide a scientific and technical contribution regarding the determination of the optimal alkaline catalyst concentration in the processing of used cooking oil, while also supporting efforts to convert waste frying oil into an environmentally friendly renewable energy source.

RESEARCH METHODS

Type of Research

This research is a laboratory experimental study that aims to analyze the effect of variations in the concentration of the Sodium Hydroxide (NaOH) catalyst of 0.1% and 0.2% in the transesterification process of used cooking oil on the characteristics of the resulting methyl ester (biodiesel).

Research Population and Sample

1. Research Population
The population in this research is waste frying oil (used cooking oil) from household/culinary use collected in the Balikpapan area.
2. Research Sample
The sample used is used cooking oil that has been filtered with filter paper to separate residual impurities/physical particulates. The research subject was focused on testing the characteristics of methyl ester from used cooking oil after passing through the esterification and transesterification stages, with test parameters covering *Free Fatty Acid* (FFA), acid number, density, *yield*, and pH.

Research Variables

1. Independent Variable
The variable that was varied in this research is the concentration of the NaOH catalyst in the transesterification reaction, namely 0.1% and 0.2% of the oil volume.
2. Dependent Variables
The dependent variables comprise the characteristic parameters of the biodiesel resulting from transesterification, namely:
 - Content of *Free Fatty Acid* (%FFA)
 - Acid Number (mg NaOH/g)
 - Density (kg/m³)
 - Yield Percentage (Yield) (%)
 - Degree of Acidity (pH)
3. Controlled Variables
The controlled variables kept constant during the research comprise:
 - Raw material: Used cooking oil
 - Solvent: Methanol with an oil sample to methanol ratio of 5:2
 - Reaction temperature: 60°C – 65°C
 - Transesterification reaction time: 30 minutes

Research Equipment and Materials

1. Research Equipment
The equipment used in this research includes:
 - *Hotplate* and *magnetic stirrer*
 - *Heating mantle*
 - Three-neck flask and condenser (a set of *reflux* apparatus)

- Thermometer
- Separating funnel, glass funnel, and beaker (*beaker glass*)
- Erlenmeyer flask, measuring cylinder, and dropping pipette
- Digital balance
- Pycnometer
- A set of titration equipment (burette and stand)

Universal pH paper

2. Research Materials

The materials used in this research include:

- Used cooking oil sample
- Methanol solvent (CH_3OH)
- Sodium Hydroxide catalyst (NaOH)
- Sulfuric Acid catalyst (H_2SO_4)
- Indicator *Phenolphthalein* (PP)
- Distilled water

Research Procedure

1. Research Flow

- This literature study began by reviewing journals, books, and final projects related to the topic of this research. This literature study is useful for obtaining the theoretical basis that can be used to solve the problem under study.
- Sample preparation in this research is useful for identifying the characteristics of the sample used in this research, which serves to determine the next step in the research.
- Esterification is the process of reacting the sample with methanol assisted by an H_2SO_4 catalyst, in which the product of this reaction will form an ester. The main purpose of the esterification process is to reduce the FFA content of the sample.
- After esterification, the process continues with transesterification, in which the ester from the esterification is reacted with methanol assisted by an NaOH catalyst, and the reaction product will form methyl ester.
- Once the methyl ester (biodiesel) has been obtained, the research results can be analyzed and conclusions can be drawn.

2. Sample Preparation

- The sample to be tested was prepared
- pH paper was dipped into the sample
- The color change on the pH paper was observed
- The pH test result was obtained

3. Esterification Process

- The equipment and materials were prepared.
- The equipment to be used was assembled.
- The oil sample that had been FFA-tested was placed into the three-neck flask.
- Methanol was added.
- The sample mixed with methanol was refluxed for ± 120 minutes at a temperature of 60°C - 65°C .
- H_2SO_4 was added gradually in an amount of 1% of the sample mass.
- The esterification product was then placed into a separating funnel and left to stand for 24 hours.

Separating the ester product by opening the separating funnel valve little by little.

4. Transesterification Process

- The equipment and materials were prepared.
- The equipment used was assembled.
- The ester was placed into the three-neck flask.
- Dissolve the NaOH catalyst in an amount of 0.1% of the oil volume in methanol
- in an amount of 29 ml.
- Add the mixture of catalyst and methanol into the three-neck flask containing the ester.
- The mixed sample was refluxed for 30 minutes at a temperature of 60°C - 65°C.
- The transesterification product was placed into a separating funnel and left to stand for ± 24 hours.
- Separating the transesterification product by opening the separating funnel valve little by little.
- The equipment and materials were prepared.
- The equipment to be used was assembled.
- The ester was placed into the three-neck flask
- Dissolve the NaOH catalyst in an amount of 0.2% of the oil volume into 29 ml of methanol.
- Add the mixture of catalyst and methanol into the three-neck flask containing the ester.
- The mixed sample was refluxed for 30 minutes at a temperature of 60°C -65°C.
- The transesterification product was placed into a separating funnel and left to stand for ± 24 hours.
- Separating the transesterification product by opening the separating funnel valve little by little.

RESULTS AND DISCUSSION

The results of the initial characteristic testing of the used cooking oil before the esterification process are presented in Table 1. The average FFA content of the initial used cooking oil sample was found to be 3.6% with a pH of 7. Because the FFA content exceeds the 2% threshold, the used cooking oil first requires an esterification stage.

Table 1 FFA Test Results of Used Cooking Oil

Sample	FFA Test 1 (%)	FFA Test 2 (%)	FFA Test 3 (%)	Average FFA (%)	Average pH
Used Cooking Oil	3.4	3.7	3.7	3.6	7

The esterification process using a 1% H₂SO₄ catalyst for 120 minutes successfully converted the free fatty acids into methyl ester and reduced the FFA content from 3.6% to 0.7% (Table 2). This reduction of the FFA content to below 1% meets the requirement for proceeding to the alkaline transesterification process.

Table 2 Esterification Analysis Results of the Oil Sample

Sample	Volume of Used Cooking Oil (ml)	Volume Methanol (ml)	Volume NaOH (ml)	Volume of Esterificati on Product (ml)	Average pH	Average FFA (%)
Used Cooking Oil	214	86	3	210	3	0.7

The results of the transesterification reaction with NaOH catalyst concentration variations of 0.1% and 0.2% are presented in Table 3 and Table 4.

Table 3 Transesterification Reaction Analysis Results of the Used Cooking Oil Sample

Catalyst Variation NaOH (%)	Volume of Oil from Esterification (ml)	Volume Methanol (ml)	FFA (%)	Volume of Transesterification Product (ml)
0.1	71	29	0.4	69
0.2	71	29	0.6	69

Table 4 Analysis Results of Methyl Ester (Biodiesel) Characteristics

Catalyst Variation NaOH(%)	pH	FFA (%)	Average Acid Number (mg NaOH/g)	Density (kg/m ³)	Yield (%)
0.1	7	0.4	0.05	875.5	69%
0.2	7	0.6	0.09	871.5	69%

The pH value of the biodiesel for both variations was at a neutral figure (pH 7). FFA content testing produced a value of 0.4% for the 0.1% catalyst and 0.6% for the 0.2% catalyst, with acid numbers of 0.05 mg NaOH/g and 0.09 mg NaOH/g respectively. According to SNI 7182:2015, the maximum threshold for the acid number of biodiesel is 0.4 mg KOH/g, so that the results obtained meet the safe qualitative limit. The density of the methyl ester obtained (875.5 kg/m³ and 871.5 kg/m³) also meets the specification range of SNI 7182:2015 (850–890 kg/m³). The biodiesel yield obtained was constant at 69%.

CONCLUSION

Variations in NaOH catalyst concentration of 0.1% and 0.2% affect the characteristics of the methyl ester resulting from the transesterification of used cooking oil. The use of a 0.2% NaOH catalyst concentration provides a lower density characteristic (871.5 kg/m³) that is closer to the optimum value of the SNI 7182:2015 specification, with an acid number of 0.09 mg NaOH/g, a pH of 7, and a yield of 69%.

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