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Research Article

EFFECT OF MICROFILTRATION AND TRANSESTERIFICATION ON VISCOUSITY AND COMPOSITION OF METHYL ESTER IN BIODIESEL

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ABSTRACT

This study aims to determine the effect of microfiltration and transesterification treatment of floured sardine used frying oil on the acid number, viscousity and composition of methyl ester on the biodiesel produced. There are 2 variables in this study, the first variable is the size of filter paper with 2 levels of treatment, while the second variable is the percentage of NaOH in sodium methoxid used in transesterification with 3 levels of treatment. The study was conducted with 3 repetitions, so that 18 treatments were obtained. Acid number and viscousity values refer to SNI Biodiesel no. 04-7182-2015. From the results of the study, the acid number values of all treatments were included in the limits poured in SNI Biodiesel no. 04-7182-2015 namely Max acid value 0.5 mg-KOH / gr but in this study acid ranged numbers were obtained from 0.26 to 0.33 mg-KOH / gr,viscousity based on SNI is 2.3 - 6.0 mm2/s in this study obtained biodiesel viscousity with ranged from 3.5 mm2/s up to 16 mm2/s. The highest methyl ester content was obtained in the treatment of 16 µm microfiltration and 1.5% NaOH concentration in the transesterification treatment, producing methyl ester of 100%, with the composition of methyl ester including Octanoic acid 10.497%, Decanoic acid 7.393%, Nonanoic acid, 9-oxo- 1.468%, Dodecanoic acid 12.241%, Tridecanoic acid, 12-methyl- 18.630%, Pentadecanoic acid 13,835%, Hexadecatrienoic acid 1,846%, 9-hexadecenoic acid 33,180% and Hexadecanoic acid 0.910%.

INTRODUCTION

Diesel fuel is one of the backbone of the transportation, industrial and electricity generation sectors. Limited oil resources and capacity of oil refineries in the country causes 50% of the fulfillment of national diesel fuel needs to be carried out through imports. This is a serious problem and must be addressed immediately. Efforts that can be taken are to immediately substitute diesel oil for renewable alternative fuels, namely biodiesel, which many raw materials can be obtained domestically (Darmanto, 2006).

Biodiesel is an alkyl-ester compound which results from the transesterification of vegetable oils or animal fats. Biodiesel has the same physical properties as diesel oil so that it can be used as an alternative fuel for diesel-engined vehicles. Biodiesel has a minimum heating value of 37 MJ / kg so that it can be used as fuel. While fossil fuels have a heating value of around 42.7 MJ / kg. Biodiesel is different from diesel oil in terms of chemical composition.

In general, diesel oil consists of 30-35% aromatic hydrocarbon compounds and 65-70% paraffin accompanied by a little olefin. Unlike diesel oil, biodiesel consists of C16-C18 fatty acid methyl ester with 1-3 double bonds in each molecule. The advantages of biodiesel compared to diesel oil are in exhaust emissions, sulfur content, sethane numbers, decomposition and stability, as well as engine lubrication and cleaning. The acids value and viscousity is a very influential parameter in the use of biodiesel. SNI Biodiesel Value no. 04-7182-2015 explains that the acid number must not exceed 0.5 mg-KOH / g and kinematic viscosity ranges from 2.3-6.0 mm2 / s. The value of viscosity that is too high can make the atomization of fuel and air become less good, namely in the form of poorer evaporation so that combustion becomes imperfect (Siti and Bambang, 2015). The value of acid numbers and viscousity that is too high can damage engine performance. So it is necessary to study the acid number and viscousity of floured sardine used frying oil.

Based on the results of the feasibility evaluation of several biodiesel feedstocks, Ruhyat and Firdaus (2006) have

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determined that the type of vegetable oil that is most suitable for use as biodiesel feedstock is used cooking oil. The main reason for finding alternative sources of diesel engine fuel is because of the high price of oil products (Chetri, 2008). Processing biodiesel from fried oil is an effective way to reduce the selling price of biodiesel because of the low cost of raw materials (Zhang, *et al*, 2003). The use of used cooking oil as a biodiesel feedstock can also overcome the problem of oil waste disposal which has an impact on public health. Hambali, *et al*. (2008) suggested that used cooking oil has a fairly high free fatty acid. To reduce the acid number in general, two stages of treatment are carried out, namely pretreatment and transesterification to convert the used cooking oil into biodiesel. Transesterification is the conversion stage of triglycerides to alkyl esters, by reacting with alcohol and producing byproducts, namely glycerol. Among monohydric alcohols which are source candidates or suppliers of alkyl groups, methanol is the most commonly used, the price is relatively cheap and the highest reactivity is a separate calculation material in choosing methanol as a source of alkyl groups. So, in most biodiesel production it is practically identical to the fatty acid methyl ester (FAME). According to Gerpen (2005), the right pretreatment process must be carried out before the transesterification stage to produce high-quality biodiesel. Microfiltration technique was chosen for the pretreatment of used cooking oil because it was easy, simple and inexpensive. Microfiltration aims to reduce or eliminate suspended solids and organic compounds such as proteins, carbohydrates and free fatty acids (Nasir *et al*, 2002). After the microfiltration process is carried out, proceed with the transesterification process.

This study aims to determine the effect of microfiltration and transesterification treatment of floured sardine used frying oil on the acid number, viscosity and composition of methyl ester on the biodiesel produced.

MATERIALS AND METHOD

Location and Duration of Study

Bioethanol production is carried out in the Agricultural Products Technology laboratory - Majapahit Islamic University, Mojokerto and the Genetics and Biomolecular laboratory - Maulana Malik Ibrahim Islamic State University, Malang. While the tests were carried out at the Genetics and Biomolecular laboratory - Maulana Malik Ibrahim State Islamic University, Malang and the MIPA Faculty laboratory, Udayana University, Denpasar. This research was conducted for 2 months starting from the manufacture to analysis of the research results.

Materials and tools

The materials used in this study include: floured sardine used frying oil, crystal NaOH, 96% methanol solution, acid periodic, Kl, hydrochloric acid, distilled water, bleaching earth, ethanol, phenolphthalein, KOH.

The tools used include: pore size 11 µm, and 16 µm of filter paper, analytic balance, hot plate, stirring, vacuum pump, thermometer, centrifuge, 500 ml erlenmeyer, 500 ml beaker glass.

Method

This research was conducted in three stages, including microfiltration, transesterification and purification. There are 2 variables in this study including the size of filter paper with 2 levels of treatment, 11 µm and 16 µm in the microfiltration process, while the second variable is the percentage of NaOH in sodium methoxid used in transesterification with 3 treatment levels namely 0.5%, 1% and 1.5 %. The study was conducted with 3 repetitions, so that 18 treatments were obtained.

The stirring temperature when the transesterification process is 50 ^oC and the stirring time during the transesterification process is 90 minutes. Purification was carried out by adding 1% bleaching earth adsorbent and then filtration.

The analysis process was carried out on floured sardine used frying oil which included acid number and viscosity referring to SNI Biodiesel no. 04-7182-2015. As well as qualitatively identifying the results of biodiesel synthesis using Gas Chromatography - Mass Spectroscopy (GS - MS).

Research Procedure

Microfiltration

Microfiltration is a pretreatment process of floured sardine used frying oil. The used frying oil of sardine flour was removed by filtration by filtration using variations in filter size, namely 11 µm and 16 µm. Then the content of FFA (Free Fatty Acid) was tested for used frying oil of sardine fish meal before and after microfiltration. Microfiltration results with the lowest FFA content are used for the next stage.

Transesterification

The Transesterification process is carried out in the following stages:

1. Sodium methoxid solution was prepared first by mixing NaOH and methanol evenly with a variation of 0.5%, 1.0% and 1.5% NaOH concentrations of the total weight of floured sardine used frying oil and methanol (w/w).
2. The filtered oil in the pretreatment process was then poured into a beaker glass and heated to a temperature of 50 ^oC. After the temperature is reached, then the prepared sodium methoxid solution is mixed into the filetered oil while stirring. Heating and stirring are carried out evenly for 90 minutes.
3. After the heating and mixing process, the mixture is settled for 12 hours. The sediment and filtrate separation process was carried out using centrifuge 1100 rpm for 10 minutes. Separation begins by taking the bottom layer (sediment) which is glycerol and then the liquid above it which is biodiesel.

Purification

Biodiesel produced from the process of microfiltration and transesterification of floured sardine used frying oil by the process stages is carried out including earth bleaching adsorbent 1% of the volume of floured sardine used frying oil weighed. Then mixed into biodiesel. The solution is stirred for 15 minutes at 55 ^oC. Then biodiesel and adsorbents are separated by filtration.

RESULTS

Free Fat Acid (FFA) Percentage

Table 1 Free Fat Acid Percentage Before and After Microfiltration

Pore Size (µm)	FFA (%)	Reduction (%)
Control	0.24	-
11	0.20	16.67
16	0.18	25.00

From the results of the research above, it can be seen that the highest percentage reduction of Free Fat Acid is 25% in the microfiltration process using pore-sized filter paper 16 µm with the Free Fat Acid percentage of 0.18%. The filtrated oil which being use for the next step of the experiment is the filtered oil using filter paper with pore size of 16 µm.

Acid Value

Table 2 The acid value conducted from the research

NaOH Concentration (%)	Acid Value (mg-KOH/gr)
0.5	0.33
1	0.33
1.5	0.26

From the data above showed that the lowest acid value was obtained in the transesterification process using NaOH catalyst of 1.5% by 0.26 mg-KOH/gr. There is no difference in the acids value either from transesterification using NaOH catalysts of 0.5% or 1%, but between 0.5% and 1% to 1.5% there is a difference in the decrease in acid numbers by 0.07 mg-KOH/gr.

Viscousity

Table 3 Viscousity of biodiesel

Konsentrasi NaOH (%)	Viscousity (mm^2/s)
0.5	16
1	11.93
1.5	3.5

From the research data above, it can be seen that the higher the concentration of NaOH in sodium methoxid used in transesterification, the lower the viscousity of the biodiesel obtained.

Methyl Esther Compound

Table 4 The composition of methyl ester from transesterification using sodium methoxid with 0.5% NaOH concentration.

No.	Name	Persentase	Methyl Ester Percentage
1.	Decanoic acid, methyl ester	0,633	0,633
2.	Dodecanoic acid, methyl ester	7,010	7,010
3.	Methyl tetradecanoate	30,532	-
4.	Pentadecanoic acid, methyl ester	1,228	1,228
5.	7-hexadecenoic acid, methyl ester	0,373	0,373
6.	9-hexadecenoic acid, methyl ester	3,504	3,504
7.	Hexadecanoic acid, methyl ester	16,310	16,310
8.	Hexadecanoic acid, methyl ester	1,099	1,099
9.	Hexadecanoic acid, methyl ester	9,059	9,059
10.	Cyclopropaneoctanoic acid, 2-hexyl-, methyl ester	0,372	0,372
11.	Heptadecanoic acid, methyl ester	2,236	2,236
12.	9,12-Octadecadienoic acid, methyl ester	21,447	21,447
13.	9-Octadecenoic acid, methyl ester	6,198	6,198
	Total	100,000	69,468

Table 5 The composition of methyl ester from transesterification using sodium methoxid with 1% NaOH concentration.

No.	Name	Persentase	Methyl Ester Percentage
1.	1,2,3-Propanetriol (CAS) glycerol	12,962	-
2.	Octanoic acid, methyl ester	5,363	5,363
3.	Decanoic acid, methyl ester	3,523	3,523
4.	Nonanoic acid, 9-oxo-, methyl ester	0,884	0,884
5.	Dodecanoic acid, methyl ester	13,066	13,066
6.	Stannane, chlorotrimethyl-	0,028	-
7.	Tridecanoic acid, 12-methyl-, methyl ester	22,237	22,237
8.	Xenon dioxide difluoride	0,028	-
9.	Pentadecanoic acid, methyl ester	6,288	6,288
10.	Hexadecatrienoic acid, methyl ester	0,914	0,914
11.	7-hexadecenoic acid, methyl ester	1,410	1,410
12.	9-hexadecenoic acid, methyl ester	15,124	15,124
13.	9-hexadecenoic acid, methyl ester	0,866	0,866
14.	Hexadecanoic acid, methyl ester	5,060	5,060
15.	Hexadecanoic acid, methyl ester	12,248	12,248
	Total	100,000	86,982

Table 6 The composition of methyl ester from transesterification using sodium methoxid with 1.5% NaOH concentration.

No.	Name	Persentase	Methyl Ester Percentage
1.	Octanoic acid, methyl ester	10,497	10,497
2.	Decanoic acid, methyl ester	7,393	7,393
3.	Nonanoic acid, 9-oxo-, methyl ester	1,468	1,468
4.	Dodecanoic acid, methyl ester	12,241	12,241
5.	Tridecanoic acid, 12-methyl-, methyl ester	18,629	18,629
6.	Pentadecanoic acid, methyl ester	13,835	13,835
7.	Hexadecatrienoic acid, methyl ester	1,846	1,846
8.	9-hexadecenoic acid, methyl ester	33,180	33,180
9.	Hexadecanoic acid, methyl ester	0,910	0,910
	Total	100,000	100,000

DISCUSSION

Acid Value

The acid number is one of the most influential parameters in biodiesel. Based on research conducted by Agus *et al* (2015), biodiesel produced from transesterification of used cooking oil with the help of microwave waves has acid numbers of 2.98-4.20 mg-KOH/gr. While the SNI value of acid number biodiesel cannot exceed 0.5 mg-KOH/gr. An acid value that is too high can damage engine performance. In the biodiesel research conducted by Putri *et al*. (2018), the acid number was 0.27 mg-KOH/gr using household cooking oil with 1% NaOH catalyst, in a reaction time of 60 minutes and a temperature of 60 ^oC. In this study, ranged acid numbers were obtained from 0.26 to 0.33 mg-KOH/gr, so that all acid numbers obtained fulfilled the biodiesel SNI value no. 04-7182-2015. The lowest acid number was obtained by microfiltration treatment using 16 µm filter paper, with the transesterification process using a NaOH catalyst of 1.5% with an acid number of 0.26mg-KOH/gr.

Viscousity

Based on Siti (2017), the viscosity of biodiesel must meet SNI values ranging from 2.3 - 6.0 mm^2/s. The value of viscosity that is too high can make the atomization of fuel and the air condition become less good, this is due to poorer evaporation so that combustion becomes imperfect. In a study conducted by Diah *et al*. (2013), making biodieselfloured sardine used frying oil using esterification-transesterification using 1.5% NaOH catalyst produced biodiesel with a viscousity of 4.1133 mm^2/s. In this study, the viscousity that fulfilled the SNI value was 3.5 mm^2/s in the microfiltration treatment with a 16 µm pore size with the transesterification process using NaOH catalyst of 1.5%.

Methyl Esther Compound

Based on research conducted by Apriani *et al* (2015), the transesterification of used cooking oil with H_2SO_4 catalyst used a temperature of 60 °C for 3 hours using esterification as a pretreatment process, producing methyl ester as much as 70.35%. Whereas Diah *et al* (2013), making biodiesel using used sardine frying oil with esterification-transesterification method using 1.5% NaOH catalyst produced 80.96% methyl ester.The amount of methyl ester produced shows the efficiency of the transesterification reaction. Based on the quality standards of SNI, a biodiesel that is good for fuel must have a minimum methyl ester level of 96.5%. As based on research conducted by Lia and Listiana (2017), transesterification of Crude Palm Oil (CPO) without pretreatment, with a temperature of 65 °C for 2 hours resulted in 99% of the methyl ester content of biodiesel.In this study the lowest levels of methyl ester were 69.468% and the highest was 100% in the pretreatment treatment with 16 µm filter paper pore size with the transesterification process using NaOH catalyst as much as 1.5% for 90 minutes at 50 °C.

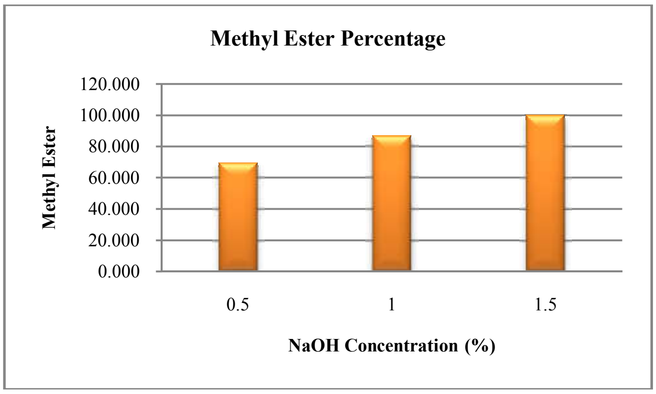

Figure 1 Percentage of methyl ester compound

With methyl ester composition:Octanoic acid 10.497%, Decanoic acid 7.393%, Nonanoic acid, 9-oxo- 1.468%, Dodecanoic acid 12.241%, Tridecanoic acid, 12-methyl- 18.630%, Pentadecanoic acid 13,835%, Hexadecatrienoic acid 1,846%, 9-hexadecenoic acid 33,180% and Hexadecanoic acid 0.910%.

CONCLUSION

From this study, it can be concluded that the sardine used frying oil is suitable as a biodiesel feedstock with microfiltration - transesterification treatment with acid numbers and viscousities that meet the requirements of biodiesel SNI no. 04-7182-2015, by using 16 µm pore-sized filter paper, and transesterification using NaOH concentrated 1.5% as a catalyst for 90 minutes at 50 °C.

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