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Analysis of biodiesel process from waste cooking oil using heterogeneous catalyst field snail shell (*Pilaampullacea*)

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Abstract

Biodiesel is an alternative fuel in the form of Fatty Acid Methyl Ester (FAME) which can be renewed using vegetable oils and animal oils through esterification or transesterification processes. This study used waste cooking oil as a raw material for biodiesel with a CaO catalyst from the shells of field snails (Pilaampullacea) which were calcined for 4 hours at a temperature of 700°C. This experiment aims to investigate the effect of temperature (55°C, 60°C, 65°C) and catalyst weight (4%, 6%, 8%) on 2 hours of transesterification process and 1:9 of the molar ratio oil to methanol. Crude biodiesel from the transesterification process washed using dry washing method with activated coconut shell charcoal which has been activated using 1M H₃PO₄. Based on this research, the optimum yields about 91.5%-volume, were obtained in A2T2 with a temperature of 60°C and a catalyst weight of 6% with a biodiesel yield of 91.5%. The characteristics of the biodiesel produced were kinematic viscosity 3.32 cSt, density 863 Kg/m3, acid number 0.543%, iodine number 14.3%-mass, methyl ester content 169.12% and cetane number 43.28%-mass.

Keywords:

Biodiesel, field snail shells, heterogeneous base catalyst, transesterification, waste cooking oil.

1 Introduction

The increasing of global population needs to be balanced with an adequate supply of fossil fuels to prevent energy crises. Fossil fuels, such as petroleum, require a significant amount of time for their formation process [1]. Alternative fuels to replace fossil fuels are needed as it is estimated that the reserves of fossil fuels will be depleted in the coming years. Fuel derived from vegetable oil is an alternative and renewable fuel source that is considered suitable for replacing fossil fuels [2].

Biodiesel is a renewable fuel as its feedstock can be cultivated by humans. The benefits of the use of biodiesel are that biodiesel has high energy efficiency and low emissions, so it may reduce environmental damage due to air pollution [3]. The most suitable raw material for use as biodiesel is used cooking oil or waste cooking oil [4]. The potential or capacity of used cooking oil based on press release No. 388.Pers/04/SJI/2020 by Directorate General of Renewable Energy and Energy Conversion, Ministry of Energy and Mineral Resources, the initial study by the National Team for the Acceleration of Poverty Reduction (TNP2K) and Traction Energy Asia stated in 2019, national consumption of palm cooking oil is 16.2 million kilo liters. From these information, the average waste cooking oil produced is 40%-60% or 5.46-9.72 million kiloliters. Waste cooking oil is a waste product from frying that can be converted into biodiesel through several chemical processes due to its high content of fatty acids [5]. Free fatty acid content in waste cooking oil exceeding 2% requires an esterification process to reduce the free fatty acid level and to achieve a more optimal biodiesel conversion [6].

The production of biodiesel requires the assistance of a catalyst to accelerate the reaction. The utilization of heterogeneous base catalysts is currently being developed to replace homogeneous catalysts because heterogeneous base catalysts are solid catalysts that can be separated through filtration and do not require a significant amount of water in the separation process[7]. One example of a heterogeneous base catalyst that can be utilized is the compound Calcium Oxide (CaO) found in field snail shells. Field snails are available in nature, especially during the rice planting season because rice field snails are pests of rice plants. So far, field snail meat has been used as additional protein for some people, while the shell is thrown away and becomes waste for the environment [8].

Snail shells contain 71.2% Calcium Carbonate (CaCO3) compound which will decompose into Calcium Oxide (CaO) at a temperature of 700°C[9]. Calcium oxide possesses with high alkalinity level, enabling its utilization as a catalyst in the transesterification process. Additionally, it can be easily separated due to its solid form and non-corrosive nature, making it environmentally friendly[10].

The waste cooking oil that has been converted into crude biodiesel through the transesterification process requires purification to remove residual methanol, catalyst remnants, soap, and glycerol. The purification process conducted is known as the dry washing method. Dry-washing a purification method employed to remove impurities from biodiesel through the process of adsorption. Dry washing method is more effective than water washing due to its faster washing process, resulting in higher quality fuel as a consequence of minimal water content in the biodiesel[11].

Nopriansyahet al, (2016) have researched that snail shells contain around 28.75% calcium carbonate, there are 23 grams of CaCO3 in 80 grams of dried snail shells [12]. Research by Delvita, et al (2015) stated that the calcination process of rice field snail shells (*Pilaampullaceae*) at temperatures of 380°C, 410°C, 440°C, 470°C, and 500°C for 2 hours produced calcite particle sizes of 118,983nm, 139,882nm, 137,067nm, 140,047nm, and 63,533 nm respectively after the drying process in an oven at 110°C for 24 hours to remove the water content [13].

Based on the aforementioned background, a study was conducted on biodiesel production from waste cooking oil using a field snail shell catalyst (*Pilaampullacea*). This research aims to analyze the effect of catalyst and temperature interaction on the biodiesel yield of the biodiesel process and the quality of biodiesel according to the biodiesel standard SNI 7182-2015.

2 Research Methods

2.1 Characterization of Waste Cooking Oil

The waste cooking oil underwent filtration using filter paper to remove debris from frying residues. Subsequently, the waste cooking oil underwent a homogenization process due to being sourced from various fried food vendors, resulting in varying characteristics of waste cooking oil. The homogenization process was performed on a total of 2 liters of waste cooking oil. After homogenization, heating was carried out using an oven at 110°C to eliminate any remaining water content in the waste cooking oil. The characterization process then continued with testing the waste cooking oil for Free Fatty Acid (FFA) content, density, and kinematic viscosity. Fig. 1 shows the waste cooking oil pretreatment process.



Fig. 1. Pre-treatment of waste cooking oil.

2.2 Calcination of Field Snail Shell

The shells separated from the field snail meat were then dried in an oven memmert UN30 at 110°C for 4 hours. Once dried, the shells were crushed and sieved to a size over 200 mesh. A calcination process was carried out at 700°C for 3 hours in a furnace muffler, and the calcined product was placed in a vacuum container to maintain its dryness[14]. The CaO catalyst is presented in Fig. 2.



Fig. 2. Snail shell catalyst.

2.3 Activation of Coconut Shell Charcoal

Coconut shell charcoal was finely ground and sieved using two stacked mesh screens of sizes 60 and 80. The charcoal retained by the 80 mesh size was subsequently activated. The activation process involved the addition of 1M phosphoric acid (H_3PO_4) for 24 hours in erlenmeyer 500 mL to remove the impurities in the pores of the charcoal and activate the pore as an adsorbent. Then it was followed by washing with distilled water until reaching a neutral pH (pH = 7), and then drying in an oven at 110°C for 3 hours. The activated charcoal is shown in Fig. 3.



Fig. 3. Activated coconut shell charcoal.

2.4 Transesterification

The waste cooking oil that was used in this research is about 100 ml per sample. The transesterification process was conducted with a methanol-to-oil molar ratio of 9:1 for 120 minutes in hotplate with waste cooking oil about 100 ml and methanol 120 ml. After the transesterification process, separation was carried out in a separatory funnel for approximately 8 hours, resulting in the formation of two layers consisting of biodiesel and glycerol. The transesterification process employed temperature variations of 55°C, 60°C, and 65°C. These temperature settings were selected based on the previous research that the best temperature of transesterification using methanol is about 55°C-65°C (the boiling temperature of methanol) [15]. The weight variations of CaO catalyst at 4%, 6%, and 8%.completely randomize design in this experiment as shown in Table 1.

Table 1.2-factor Completely Randomized Design (CRD) experiment

Temmeneture	CaOcatalyst concentration				
Temperature	A1(4%)	A2(6%)	A3(8%)		
T1 (55°C)	T1A1	T1A2	T1A3		
T2 (60°C)	T2A1	T2A2	T2A3		
T3 (65°C)	T3A1	T3A2	T3A3		

2.5 Dry Washing Process

The washing process was carried out using the *dry washing* method, utilizing activated charcoal from coconut shells. The activated charcoal contains 5%-15% moisture, 2%-3% ash, and therest is carbon. [16]. The active charcoal used was 5% w/w of oil at a temperature of 45° C-60°C with a stirring time of 20 minutes, as it represents the optimal stirring time[17]. The drywashing process is shown in Fig. 4.



Fig. 4. Washing of crudebio diesel.

In this research, tests were carried out to analyze the ability of heterogeneous base catalysts and the effect of temperature in increasing biodiesel yield efficiency and biodiesel quality which includes density, kinematic viscosity, acid value, iodine number, and cetane number.

3 Results and Discussion

3.1 Testing of Waste Cooking Oil

The Free Fatty Acid (FFA) content testing is conducted using the AOCS Ca. 5a-40 method, involving the addition of 50 ml of 95% ethanol to 5 grams of waste cooking oil and heating the mixture to a temperature of 40°C. Titrations are performed using 0.1 N KOH with the addition of phenolphthalein indicator. The results of the initial FFA content testing in the waste cooking oil areused to determine the bio diesel production process. If the FFA value is below 2%, the bio diesel production does not require the esterification process[6].

The density value of waste cooking oil is determined through testing using the AOAC 920.212 and AOAC 920.213 methods using a pycnometer. Meanwhile, the kinematic viscosity value is determined using the ASTM D445 method, employing a viscometer at a temperature of 40°C. The results of used cooking oil characterization as shown in Table 2.

Table 2.	Waste	cooking	oil	characteristics
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Characteristics	Value	Unit
FFA Content	1.32	%
Density	911	Kg/m ³
Viscosity	5.06	cSt

3.2 Biodiesel Yield

The calculation of bio diesel yield is performed after the washing and evaporation processes on the crude bio diesel. Bio diesel yield is the ratio of the final mass to the initial mass of biodiesel. Bio diesel yield is determined using Eq. 1 and the result of transesterification is presented in Table 3.

$$\frac{\text{Mass of purified biodiesel }(g)}{\text{Mass of waste cooking oil }(g)} \times 100\%$$
(1)

Table 3.Biodieselyield

Tomporatura	CaO	Yi	Mean	
Temperature	Catalyst	Replication 1	Replication 2	Mean
	4%(A1)	88	86	87
55°C	6%(A2)	88	90	89
	8%(A3)	79	81	80
	4%(A1)	85	85	85
60°C	6%(A2)	93	90	91.5
	8%(A3)	84	87	85.5
65°C	4%(A1)	70	73	71.5
	6%(A2)	72	70	71
	8%(A3)	82	83	82.5

From Table 3, it can be be be very that sample T2A2 with a temperature treatment of 60° C and a catalyst concentration of 6% produces the high estyield, which is 91.5%. Meanwhile, the lowest yield is in the T3A2 variation, with a temperature treatment of 55°C and 6% catalyst, resulting in a yield of 71%.

3.3 Effect of Temperature

Theuse of higher temperatures in bio diesel production can affect the resulting biodiesel yield result. Molecules can move more rapidly as the reaction energy increases with temperature, making it easier to breakthe carbon bonds in triglycerides with the aid of catalyst and alcohol [18]. The results of the effect of temperature are presented in Fig. 5.

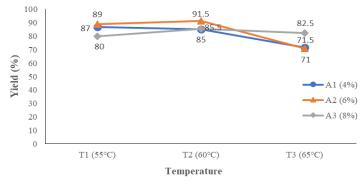


Fig. 5. Effect of temperature on bio diesel yield.

Based on Fig. 5, it can be observed that raising the temperature from 55°C to 60°C increases the bio diesel yield. However, increasing the temperature from 60°C to 65°C yield sthelowe staverage bio diesel production. This is due to 65°C being a high temperature, surpassing the boiling point of methanol at 64.7°C, causing methanol to evaporate and resulting in lower bio diesel yields.

3.4 Effect of Catalyst Concentration

Catalyst serves to accelerate the reaction rate in the formation of methylesters by reducing activation energy. The addition of catalyst also influences the yield out come in biodiesel production. The results of the effect of catalyst concentration are presented in Fig. 6.

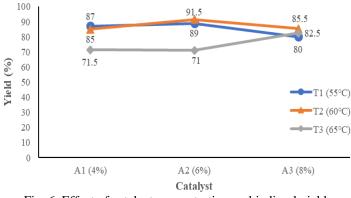


Fig. 6. Effect of catalyst concentration on biodiesel yield.

The addition of catalyst from 4% to 6% w/w of oil can enhance the bio diesel yield. This occurs due to the increase in the number of colliding molecules and the acceleration of the reaction rate when the catalyst amount is raised. However, the utilization of 8% w/w of oil catalyst results in the low estaverage yield due to the excess catalyst causing accumulation in the glass reactor and affecting the reaction equilibrium[18].

3.5 ANOVA Test

The bio diesel yield was furtheranalyzed using a 2-factor 2replication factorial design in the transesterification stage. The first factor was the temperature variation (T), consisting of 3 levels: 55°C, 60°C, and 65°C. Meanwhile, the second factor was the catalyst usage (A) with 3 levels: 4%, 6%, and 8%. The result of ANOVA test as shown in Table 4.

Table 4. Analysis of Variance (ANOVA)	Table	4. Anal	vsis of	Variance	(ANOVA)
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SK	DB	JK	VТ	Ehitung	FΤ	abel	- Notasi
SK	DB	JK	КI	F hitung -	5%	1%	Notasi
Т	2	525.78	262.889	107.545	4.256	8.021	**
А	2	21.33	10.722	4.386	4.256	8.021	*
TA	8	289.22	36.153	14.789	3.229	5.467	**
Error	9	22	2.444				
Total	17	858.44					

Explanation:

* = statistically significant

** = highly statistically significant

Based on Table 4, it is evident that the interaction between the two variations has a highly significant impact on each treatment in terms of bio diesel yield, as well as the temperature (T) factor. These results were further subjected to post hoc testing using the Duncan Multiple Range Test (DMRT).

3.6 Post Hoc Duncan Multiple Range Test (DMRT)

The highest yield does not necessarily indicate the best treatment in this study. The results obtained from the ANOVA test were further subjected to the Duncan Multiple Range Test (DMRT) to determine the differences in each effect within each treatment and to identify the best treatment for producing bio diesel yield. The result of Duncan Multiple Range Test (DMRT) for Temperature (T) as shown in Table 5 and for TxA treatment presented in Table 6.

Table 5. Results of Duncan Multiple Range Test (DMRT) for Temperature (T) treatment

Variation of temperature treatment	Meanbio diesel yield
T2	87.333ª
T1	85.333 ^b
T3	75°

Explanation: numbers followed by the same letter (notation) in thesame column indicate no significant effect in the 5% Duncan Multiple Range Test (DMRT).

Based on the post hoc DMRT results in Table 5, the T2 variation treatment significantly affects T1, where as T1 also significantly affects T3. The T2 treatment variation is the most effective treatment in producing bio diesel yield.

Table 6. Results of Duncan Multiple Range Test (DMRT) for TxA treatment

Treatment T×A	Mean bio diesel yield
T2A2	91.5ª
T1A2	89 ^b
T1A1	87°
T2A3	85.5 ^{cd}
T2A1	85 ^{cd}
T3A3	82.5 ^d
T1A3	80 ^e
T3A1	71.5 ^f
T3A2	71 ^{fg}

Note: numbers followed by the same letter (notation) in the same column indicate no significant effect in the 5% Duncan Multiple Range Test (DMRT).

The $T \times A$ treatment represents the interaction between temperature and catalyst in transesterification. The occurring interactiont ends to have a significant impact in each case. However, significant result sare observed in specific treatments. Based on these advanced test results, it can be observed that the T2A2 variation is the most effective in yielding bio diesel.

This indicates that the increase in temperature and catalyst concentration does not result in an increase in bio diesel yield. An increase in temperature beyond the boiling point of methanol causes methanol to evaporate, leading to lower bio diesel yield. Meanwhile, an excess of catalyst can disrupt the reaction equilibrium and yield excessive by-products [18].

3.7 Biodiesel Characteristics

The highest yield obtained in bio diesel production is from sample T2A2. This yield is subsequently subjected to testing for bio diesel characteristics, encompassing various parameters as shown in Tabel 7.

Table 7. Bio diesel characteristic

Parameter	Unit	Biodiesel standard	Biodiesel waste cooking oil	Information
Density	Kg/m ³	850-890	863	Appropriate
Kinematic viscocity at 20°C	cSt	2.3-6.0	3.32	Appropriate
Acid value	Mg KOH/gr	Max 0.5	0.4	Appropriate
Iodine number	%-massa	Max 115	14.3	Appropriate
Cetane number	-	Min 51	43.28	Not Appropriate
FAME	%	Min 96.5	108.79	Appropriate

3.8 Density

Density, or densitas, is the ratio of mass to volume in bio diesel (Eq. 2). Density testing is carried out using the AOAC 920:212 method. The testing is conducted using a pycnometer and water as a reference fluid, with a temperature of 20° C.

$$\rho \ biodiesel = \frac{m \ biodiesel}{m \ water} \rho \ water \tag{2}$$

Explanation: ρ = density (kg/m³) m = mass (kg)

Based on the testing conducted following the AOAC 920:213 method with temperature correction, the density value of the biodiesel is 863 kg/m³. This value complies with the quality standard for bio diesel, as specified by SNI 7182-2015. A lower density will affect the fuel capability on high oil and a high density will affect the low fuel capability [19].

3.9 Kinematic Viscosity

Viscosity values influence combustion within the engine combustion chamber. Lower viscosity facilitates the easy ignition of the fuel [20]. The method for testing kinematic viscosity employs the ASTM D445 method, utilizing a viscometer at a temperature of 40°C. The formula used to determine the viscosity value is on Eq. 3.

$$v = C \cdot t \tag{3}$$

Explanation:

v = kinematic viscosity (cSt)

C = viscometer constant (cSt/s)

t = flow time (s)

Based on the conducted testing, the kinematic viscosity of bio diesel at 40° C is 3.32 cSt. This value complies with the quality

standard for bio diesel according to SNI 7182-2015, which falls within the range of 2.3 to 6.0. Fuel with a low viscosity will be easier to inject and will easily catch fire and complete combustion will occur. The high viscosity number makes fogging difficult and causes incomplete combustion. This combustion will leave combustion residue and damage the combustion chamber [20].

3.10 Saponification Number

The saponification number is expressed in the amount of milligrams of KOH required to saponify 1 gram of oil. Thevalue of the saponification number depends on themolecular weight of the oil [20]. Calculation of the saponification number is carried out using the Eq. 4 according to AOAC 920.160.

$$Saponification \, Value = \frac{28.05(B-S)}{Sample \, weight(g)} \tag{4}$$

Explanation:

B = volume of titrant in the blank (ml)

S = volume of titrant in the sample (ml)

The result of the saponification value testing using the method in AOAC 920.160 is 27.4 mg/g. A lower saponification value indicates that the carbon chains in bio diese larefewer, and the content of free fatty acids is lower[21].

3.11 Iodine Value

The iodine value represents the quantity of double bonds in the fatty acids composing bio diesel. The iodine value testing method follows AOAC 920.159, involving the addition of the Wijsreagent during the testing process. The formula determining the iodine value is as Eq. 5.

$$I/Cl = \frac{2X}{3B - 2X} \tag{5}$$

Explanation:

X = volume of 0.1 N Na₂S₂O₃ in solution I (ml)

B = volume of 0.1 N Na₂S₂O₃ required forhalogen content (ml)

Based on the conducted testing, the iodine value of the bio diesel is 14.3% by mass. This value complies with the bio diesel standard SNI 7182-2015. If the iodine number is higher than the standart, it can cause scale to form in the combustion chamber [22].

3.12 Cetane Number

The cetane number is associated with the fuel quality. A higher cetane number indicates that the fuel ignites easily at lower temperatures[23].Cetane number testing is conducted using the cetane index approach. The formula employed for this approach is in Eq. 6.

Cetane Number =
$$46.3 + \left[\frac{5.458}{saponification}\right] (0.225 \times Iod)$$
 (6)

Based on the cetane number test results of the bio diesel, a value of 43.28 was obtained. This out come does not meet the requirement of SNI 7182-2015, which specifies a minimum cetane number of 51. A high cetane number indicates that the fuel can ignite easily even at low temperatures. Combustible fuels at low temperatures can reduce detonation [23].

3.13 Acid Value

The acid value is a parameter that identifies the content of free fatty acids in bio diesel. The determination of Free Fatty Acid (FFA) content is carried out using the AOCS Ca. 5a-40 method, involving the addition of 50 ml of 95% ethanol to 5 grams of waste cooking oil and heating the mixture to a temperature of 40°C. The titration was conducted using 0.1 N KOH with the addition of phenolphthalein indicator. The acid value was

determined through testing using the AOCS Ca. 5a-40 test method. The testing procedure involved adding 50 ml of 95% ethanol to 5 grams of bio diesel and heating it to a temperature of 40°C.Subsequently, this solution was titrated using 0.1 N KOH along with 1% phenolphthalein indicator. The titration with KOH was performed until a pinkish-red color was observed.

$$Acid value = \frac{A \times N \times 56.1}{G}$$
(7)

Explanation:

A = volume of KOH in the titration (ml) N = normality of the KOH solution

G = weight of the sample (g)

56.1 =molecular weight of KOH

The conducted testing yielded an acid value of 0.5. This result is in accordance with the SNI 7182-2015 standard. A high acid value can cause deposits to form in the combustion chamber because ash is formed during the combustion process and can increase the corrosiveness of the fuel [23].

4 Conclusion

The optimal interaction of temperature and catalyst for bio diesel yield was observed in sample T2A2, with a temperature treatment of 60 degrees Celsius and a catalyst concentration of 6%, resulting in a biodiese lyield of 91.5%. The characterization of the bio diesel aligned with the SNI 7182-2015 standard, yielding a density of 863 kg/m3, viscosity of 3.32 cSt, acid value of 0.533 mgKOH/g, iodine value of 14.3%, and a FAME content of 108.79%.

As for recommendations for further research, they include the production of biodiesel using different heterogeneous catalysts and the implementation of dry washing with alternative substances that exhibit potential as adsorbents. The cetane number testing should ideally be conducted directly within a laboratory setting.

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