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Research Article

Use of Sulfuric Acid-Impregnated Biochar Catalyst in Making of Biodiesel from Waste Cooking Oil via Leaching Method

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Abstract

The biodiesel synthesis of waste cooking oil (WCO) over a impregnated biochar catalyst was systematically studied. This research aimed to prepare Biochar-based material that comes from coconut coir, activate it, and apply it as a catalyst to the esterification reaction of high-FFA waste cooking oil. Activation of the catalyst was done by impregnation H₂SO₄ solution in Biochar. The obtained catalyst was characterized by FTIR, XRF, XRD, surface area analyzer, and SEM-EDS. The esterification process was conducted by varying the catalyst weight (5, 7, and 10 wt%) and the reaction temperature (55 and 60 °C). The obtained liquid yields were characterized by GC-MS. The study found that the esterification process worked best with 10 wt% catalysts, a 1:76 mole ratio of oil to alcohol, and a reaction temperature of 60 °C. The waste cooking oil was successfully converted into biodiesel, reaching 84.50% of yield and 77.30% of purity (methyl ester content). Meanwhile, testing using national biodiesel standards with parameter limits of density, viscosity, iodine number, and acid number shows results that meet the requirements.

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Keywords: biochar; solid-acid catalyst; high-FFA waste cooking oil, biodiesel synthesis; esterification

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1. Introduction

The use of heterogeneous catalysts for biodiesel applications has been supported by various parties currently as an alternative to homogeneous catalysts, which are generally effective in reaction processes but have adverse effects such as waste and do not support the issue

of sustainability, one of which is the issue of ease handling and material cvcle Heterogeneous catalysts based on biomass raw materials have recently gained a place in several studies [4–6], because it is cheap, easy to obtain, and relatively easy to process into a porous material with further activation as a catalyst material, according to the desired application. Researchers also used that material instead of the others (e.g. conventional chemical reagents, magnetic materials, ionic liquids, etc.) to

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accommodate the principles of green chemistry, where several important points are less hazardous chemical synthesis and design of fewer chemical reagents, based on several previous reviews and research [7–9].

Biochar is a porous material that can be used as a support material where its structure can be treatment. modified by chemical Biochar generally comes from biomass, which amorphous and consists of various constituent oxides that have a non-uniform pore structure [10]. The crystal phase growth can be increased with certain treatments such as impregnation and encapsulation to support its catalytic performance [11]. Based on various studies [10,12], purified biochar as a silica solid acid catalyst can be promising to produce biodiesel from oil with high Free Fatty Acid (FFA) content. Compared with heterogeneous acid catalysts, advantages of silica are its excellent proton donation ability and simple preparation steps [13].

However, the best data on synthesizing biodiesel using heterogeneous catalysts, both acidic and basic types, has not been found from purified biochar raw materials. Therefore, several other researchers also carried out experiences using biochar catalysts without purification [14,15], using quite a large ratio of alcohol and a long enough time, i.e. 6 h. Apart from that, the methods, materials, and equipment used are also more complex, so it is concluded that they will require higher costs, such as the use of N₂ gas, the use of activated carbon instead of biochar, and others. Thus, using a biochar catalyst without purification is exciting for further exploration. This research tries to produce a biochar catalyst without purification and directly to the activation process by sulfuric acid. Both are processed with a low-cost and straightforward technique. Then, the performance was tested in esterification, starting the leaching technique: extracting compounds from the methanol and catalyst reaction, which is continued with reacting with waste cooking oil. This technique is quite

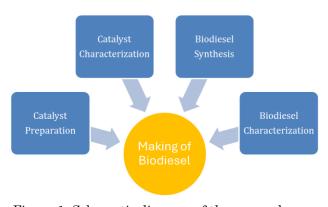


Figure 1. Schematic diagram of the research.

reasonable, where in several studies [16,17], it has successfully produced methyl ester with requirements such as the catalyst concentration must be above 1 wt% and using special equipment.

2. Materials and Method

2.1 Materials

Coconut fiber is obtained from suppliers in local markets. Waste cooking oil was taken from a restaurant in the Serpong area, South Tangerang. The chemicals, sulfuric acid (H₂SO₄, purity 96%), methanol (CH₃OH, purity 99.8%), and ethanol (C₂H₆O, purity 96%) came from Supelco (Merck). Aquades (H₂O) is obtained from the distilled water installation at the Chemical Research Center laboratory, BRIN.

2.2 Method

This biodiesel research method consists of four main activities, as shown in the diagram in Figure 1. The sequence of activities is in the direction of the diagram from left to right. We included a description of each activity in a separate section (see Supplementary Document).

The catalyst obtained was characterized using Fourier Transform Infrared Spectroscopy X-Ray Fluorescence (XRF), X-Ray (FTIR), Diffraction (XRD), Brunauer-Emmett-Teller (BET), and Scanning Electron Microscope (SEM-EDS) instruments. Each characterization determines and analyses the functional group, composition, phase, surface area, and morphology, respectively. All laboratory and characterization activities were conducted at several places: Surfactant Laboratory & The Advanced Characterization Laboratory-Research Center for Chemistry, IEBE Test Laboratory-Nuclear Fuel Technology, and Fuel & Design Engineering

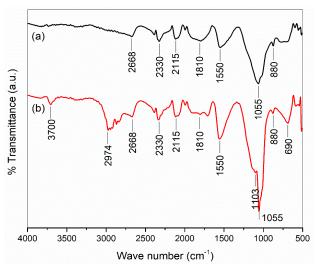


Figure 2. Functional group analysis of (a) Biochar and (b) Impregnated biochar.

Laboratory, all under the National Research and Innovation Agency (BRIN).

3. Result and Discussion

3.1 Functional Group Analysis

First, in the analysis of biochar based on functional groups (Figure 2), the characteristic found [18] was the C-H (alkyne) bending and aliphatic compound vibration at 690 cm⁻¹ and 2974 cm⁻¹, respectively. Followed by the double bond region (1500-2000 cm⁻¹) in C=C stretching vibrations at 1550, and 1810 cm⁻¹. Next, Carbonto-carbon vibrations are found in the triple bond region (2000-2500) at 2115 and 2330 cm⁻¹. It shows the organic characteristics of carbon-rich biochar. Second, the addition of sulfuric acid in the impregnation causes the formation of S-O (sulfonic) functional groups, shown in the wave number 1103 cm⁻¹ [19,20]. Thus, it is concluded that impregnation of sulfuric acid on the biochar catalyst increases the acidity of the catalyst by the appearance of sulfonate functional groups.

Third, the characteristics of silica material or silica oxy compounds are also found, where Si-O stretching from Si-OH [20] and Si-O-Si stretching vibrations [18] occurred at wavenumber 880 and 1055 cm⁻¹, contained in Impregnated Biochar and Biochar. It indicates that before and after impregnation treatment, the silica compounds in the material still exist. This is also reinforced by the absorption at the wave number 2668 cm⁻¹ [21] and 3700 cm⁻¹ [22], where they are crystalline silica and silanol phases (Si-OH), respectively.

3.2 Composition Analysis

The Characterization using XRF shown in Table 1 explains that the sulfuric acid

Table 1. Elemental composition in biochar and impregnated biochar expressed as oxide.

Compound -	Content (wt%)		
	Biochar	Impregnated Biochar	
SiO_2	12.63	14.30	
SO_3	2.92	63.60	
P_2O_5	4.71	5.40	
CaO	41.08	8.80	
MgO	5.61	2.40	
Fe_2O_3	3.67	0.80	
K_2O	21.80	1.70	
Cl	4.17	3.00	
Na_2O	1.80	-	
$\mathrm{Sc_2O_3}$	0.34	-	
Cr_2O_3	0.12	-	
MnO	0.36	-	
SrO	0.73		

impregnation of biochar resulted in a decrease in the content of alkaline oxide and alkaline earth compounds such as CaO, K_2O , MgO, and Na_2O due to the reaction between hydrogen ions (H⁺) from sulfuric acid with alkali and alkaline earth ions contained in biochar. This impregnation process forms compounds that are more soluble in water such as sulfate salts (SO_4^{2-}) and water (H₂O), reducing the concentration of alkaline oxides and alkaline earth in biochar impregnated to become a catalyst material. As for traces of carbon and other light atomic weight compounds that cannot be detected in XRF, they can be found in FTIR and EDS.

Meanwhile, the sulfur trioxide (SO₃) content increases due to added sulfuric acid. This makes the catalyst tend to be acidic. Meanwhile in our previous studies [5,6], Pyrolysis of coconut fiber into biochar at temperatures of 300 and 450 °C increases the levels of alkaline and alkaline earth oxides so that it can be classified as a solid base catalyst [4]. Based on that phenomenon, it can be concluded that the pyrolysis or calcination process (in oxygen-poor conditions) of biomass material can increase the alkalinity of the catalyst. Meanwhile, to increase the acidity, it is obtained by further treatment, such as acid impregnation.

3.3 Phase Analysis

Phase studies show changes in crystal structure after being impregnated with sulfuric acid, as seen from the differences in diffraction peak positions shown in Figure 3. This image compares the diffraction patterns between samples of coconut fiber biochar and biochar impregnated with sulfuric acid, showing that the biochar remains in amorphous form. before and after impregnation. In previous research [5,12,23] states that the broad peak shape with the peak

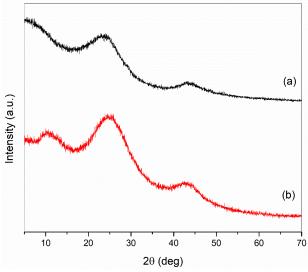


Figure 3. Diffraction pattern of (a) biochar and (b) impregnated biochar.

center around $2\theta = 22-27^{\circ}$ indicates that the peak contains silica phase. This is found in both materials, both Biochar and impregnated Biochar.

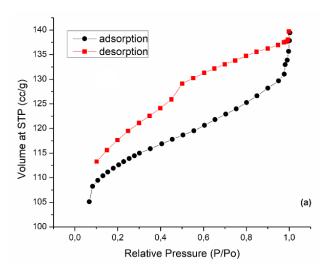
Furthermore, the calculation of the degree of crystallinity in Table 2 shows that the biochar before and after impregnation experienced a phenomenon of increasing crystallinity by 15%. It is known that increasing crystallinity in porous support materials such as biochar correlates with adsorption activity in its role as a catalyst in esterification and transesterification reactions [6].

3.4 Surface Area Analysis

The specific surface area of the obtained biochar has been calculated using the BET isothermal nitrogen (N₂) adsorption-desorption technique. The average pore diameter was calculated using the BJH method. The isotherms for the biochar are shown in Figure 4, and they are all classified as type IV by the IUPAC. The Type IV isotherm is distinguished by its hysteresis loop, which corresponds to capillary condensation occurring in mesopores. The impregnated biochar catalysts displayed a hysteresis loop that corresponded to H4, indicating that it was related to a narrow slit-like pore [24]. Additionally, the

information in Table 3 shows that the mesoporous catalyst formed, with an average pore diameter of 2 to 50 nm. The isotherms in Figure 3 also indicate that before and after the biochar was modified with H_2SO_4 , there was no discernible difference in the isotherm form.

Table 3 displays the specific surface area, total pore volume, and average pore diameter of the proposed catalysts material. Impregnated Biochar has a considerably lower specific surface area than biochar. The addition of sulfuric acid to biochar reduce the surface area of biochar due to the sulfonic groups are located and deposited onto the silica surface [25]. Adeyinka et al. [26] also reported that the sulfonating substance impregnated on the biochar caused a partial blockage of some pores as a result of the sulfonation. The decrease in the total pore volume is also possible due to the presence of sulphate, which blocks the catalyst pores. However, according to them [15,26],this decrease phenomenon is not related to the catalytic performance. The enlargement of catalyst pores during the sulphate-impregnating process can also contribute to the increase in the average pore diameter.



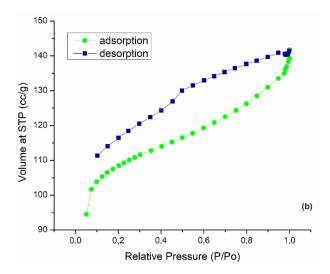


Figure 4. Nitrogen adsorption-desorption isotherms of (a) biochar and (b) impregnated biochar.

Table 2. Degree of crystallinity of biochar before and after impregnation.

Material	$A_{ m c}$	$A_{ m t}$	Cr.I (%)
Biochar	8661.3	14470	59.9
Impregnated biochar	12895	18190	70.9

Table 3. Textural properties of material.

Properties	Biochar	Impregnated biochar
Specific surface area (m²/g)	342.36	337.00
Total pore volume (cm³/g)	0.26	0.22
Average pore diameter (nm)	2.52	2.60

3.5 Morphology Analysis

The following figures represent the characterization results using SEM-EDS for biochar and impregnated biochar. In Figure 5(a) and (b), the white round shape represents silica compounds, where we can see an increase in silica compounds from before to after impregnation. It aligns with our composition analysis, where silica increased from 12.63 to 14.3% mass.

Then, magnification was carried out on the silica particles, and a phenomenon was found, as shown in Figure 5(c) and (d), which might explain the shrinkage of the surface area from biochar to impregnated biochar, as in Table 3. It looks like the silica particles have been exposed. We also found this phenomenon in other silica particles.

Meanwhile, Figure 6 shows the energy spectrum on the surface of biochar and

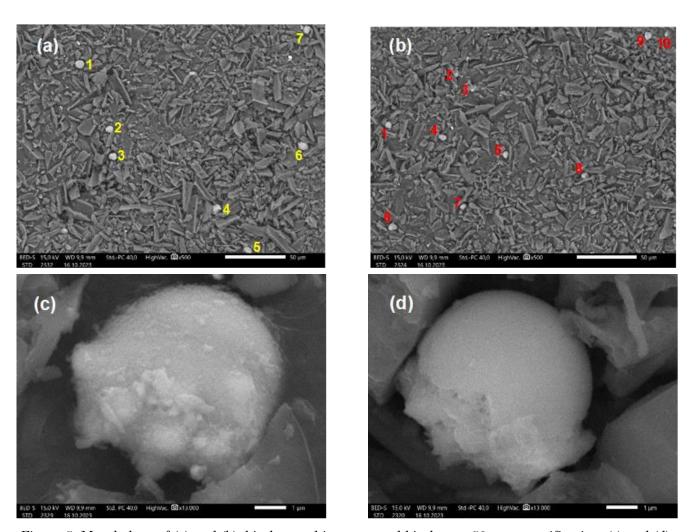


Figure 5. Morphology of (a) and (b): biochar and impregnated biochar at 50 μ m magnification; (c) and (d): Silica particle of biochar and impregnated biochar at 1 μ m magnification

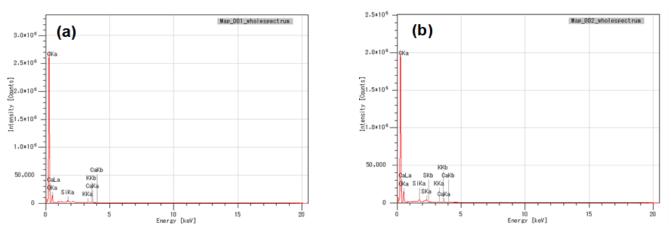


Figure 6. Energy spectra in the surface of (a) biochar, and (b) impregnated biochar.

impregnated biochar, where the results identify the constituent atoms present. In biochar, Si, alkali and alkaline earth metal atoms: K and Ca, were detected well. Meanwhile, the results are about the same with impregnated biochar, but there is a difference: the appearance of sulfur (S) atoms. Our composition analysis shows that the sulfuric acid impregnation process causes the energy spectrum of the S atom to appear in EDS. Both materials generally have the same dominant carbon (C) and oxygen (O) energy spectrum because both result from biomass pyrolysis, producing carbon and metal oxides. Thus, between these two feature (electron microscope and energy spectra), the results mutually support each other.

3.6 Biodiesel Synthesis

3.6.1 Oil Analysis

Table 4 data shows that the Waste Cooking Oil (WCO) is dominated by fatty acids with twelve carbon bonds, namely lauric acid, with a percentage of 45.16%. Followed successively by myristic acid, palmitic acid, and stearic acid. It was slightly different from our previous study [6], where stearic acid was in third place before palmitic acid. It was quite natural because the waste cooking oil used differs in terms of frying time, type of food being fried, *etc*. The hydrolyzed carbon bonds are also different.

3.6.2 Proposed Reaction Mechanism

The reaction mechanism that occurs in biodiesel synthesis using heterogeneous acid catalysts generally involves proton donors from the solid acid. For example, silica sulfuric acid (SSA) is reacted in batches with fatty acids and methanol, donating protons to the carbonyl functional group thereby allowing nucleophilic attack on fatty acids which then form protonated esters [13].

However, in the leaching method, this is different because the compounds in impregnated biochar do not react directly with fatty acids but react with methanol, where the filtrate is then processed in batches with fatty acid/oil reagents. Referring to the XRF results where SO₃ compounds are the most significant fraction in biochar, there are two possible reaction mechanisms related to this. The first is the possibility of SO₃ evaporating and reacting with water vapor to produce sulfuric acid in the gas phase, as in the following reaction:

$$SO_3(g) + H_2O(g) \rightarrow H_2SO_4(g)$$

From this scheme, sulfuric acid then dissociates so that its H⁺ cation becomes a proton donor on the carbonyl functional group on the fatty acid along with the nucleophilic attack of methanol. However, according to another study [27] the activation energy to carry out this

$$H_{3}C-OH + SO_{3} \longrightarrow CH_{3}OSO_{3}H$$

$$(3) \quad R-C-O H$$

$$(3) \quad R-C-O H$$

$$(4) \quad R-C-O H$$

$$(4) \quad R-C-O H$$

$$(5) \quad R-C-O H$$

$$(6) \quad R-C-O H$$

$$(7) \quad R-C-O H$$

$$(8) \quad R-C-O H$$

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$$(1) \quad R-C-O H$$

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$$(4) \quad R-C-O H$$

$$(5) \quad R-C-O H$$

$$(7) \quad R-C-O H$$

$$(8) \quad R-C-O H$$

$$(9) \quad$$

Figure 7. Esterification reaction mechanism with sulfuric acid impregnated biochar catalyst.

Table 4. Fatty acid composition of WCO.

Fatty Acid	Distribution (%)	Structure
Caprylic Acid (8:0)	5.83	$\mathrm{C_8H_{16}O_2}$
Capric Acid (10:0)	5.17	$\mathrm{C}_{10}\mathrm{H}_{20}\mathrm{O}_2$
Lauric Acid (12:0)	45.16	$\mathrm{C}_{12}\mathrm{H}_{24}\mathrm{O}_2$
Myristic Acid (14:0)	18.52	$\mathrm{C}_{14}\mathrm{H}_{28}\mathrm{O}_2$
Palmitic Acid (16:0)	14.80	$\mathrm{C}_{16}\mathrm{H}_{32}\mathrm{O}_{2}$
Stearic Acid (18:0)	10.51	$\mathrm{C}_{18}\mathrm{H}_{36}\mathrm{O}_{2}$

reaction is enormous, and the possibility of H₂O molecules in the gas phase in the reaction flask is minimal, so this possibility is ignored.

The second possibility is to use a leaching process where methanol is reacted with biochar so that the SO₃ compound reacts with methanol to form methyl sulfate, then the methyl sulfate donates its H⁺ to the carbonyl functional group. Because this reaction is endothermic and reversible, not all of the reacting reagents change into methyl sulfate so that methanol which does not change into methyl sulfate still plays a role in esterification as in the reaction mechanism proposed (Figure 7).

After the nucleophilic attack, the electrophilic fatty acid tetrahedral intermediate forms a proton donor to the hydroxyl group and then forms a phi bond to obtain the formation of methyl ester and H₂O, respectively, as a product and a by product. As for the contribution of silica, the influence of phase, with its mesopore size, causes it to dissolve in methanol to form SiOCH₃. However, it has a weaker acid strength [28] than methyl sulfate, so the catalytic activity might be less or negligible in this esterification mechanism. Similar

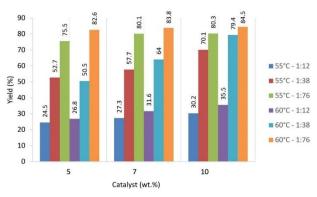


Figure 8. Effect of catalyst weight on biodiesel yield with variations in ratio and temperature.

considerations apply to alkali and alkaline earth oxides, as they are bases and can participate in transesterification reaction; however, this tendency is disregarded throughout the leaching process.

3.6.3 Biodiesel Yield

The biodiesel yield obtained by varying catalyst weight and reaction temperature is shown in Figure 8. The results show that catalyst weight, mole ratio, and temperature in the biodiesel reaction influence the reaction rate and product conversion. At a catalyst weight of 5 and 7%, at a reaction temperature of 55 °C, an increase in the mole ratio from 1:12 to 1:38 increased the yield by around 50-52%. At a reaction temperature of 60 °C, similar results were reported. Meanwhile, at a catalyst weight of 10%, an increase of approximately 55% was reported, because the addition of a catalyst automatically increases the concentration of methyl sulfate in the leaching process, when it reacts with oil, it will result in the formation of more methyl ester, which has an impact on the increasing yield trend. So, it can be concluded that the measured addition of alcohol, and balanced with the addition of a catalyst, can improve the product conversion process. Furthermore, increasing the mole ratio to 1:76 increased the product yield until it reached its best at 60 °C with a catalyst weight of 10%.

Temperature affects the reaction rate, conversion, and product quality in biodiesel synthesis. A temperature that is too low can inhibit the reaction process. At the same time, a too-high temperature can affect the catalyst's performance and make the alcohol volatile, inhibiting the reaction process. The study results show the largest biodiesel yield of 84.5% at a temperature of 60 °C. This is because increasing temperature theoretically increases the reaction

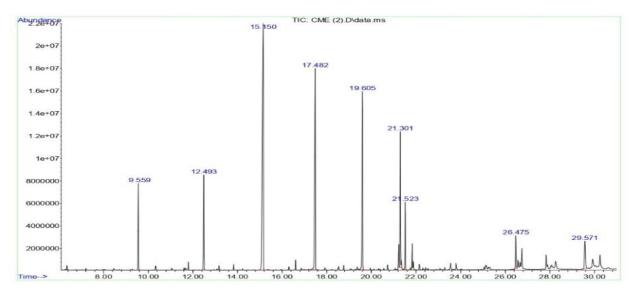


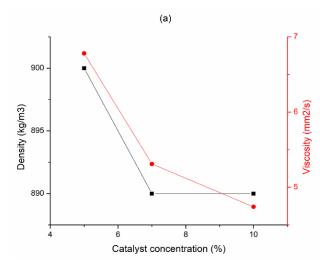
Figure 9. GC-MS chromatogram of biodiesel (10 wt%, 60 °C).

rate, and at this temperature the potential for methanol evaporation can still be controlled [11,29].

3.7 Biodiesel Analysis

GC-MS analysis produced the chromatogram in Figure 9, where the mass spectrometer can detect compounds that appear in the form of peaks. The results show that biodiesel with a catalyst weight of 10 wt% at a temperature of 60 °C contains a methyl ester content of 77.30%, which consists of the compounds methyl caproate, methyl caprylate, methyl caprinate, methyl laurate, methyl myristate, methyl palmitate,

methyl linoleate, methyl oleate, and methyl stearate, as shown in Table 5. Methyl laurate was detected as the most prominent methyl ester, with a content of 25.63%. Followed by methyl myristate, methyl palmitate, and methyl oleate at 15.33, 12.24%, and 8.20%, respectively. It was suitable by Table 4, where the methyl ester corresponds to the initial constituent components: lauric acid, myristate acid, palmitic acid, and stearic acid. The difference between the stearate and oleate hydrocarbon chains is straight bonds and -cis bonds, so a bit different from straight to cis bonds in methyl ester is possible in endothermic reactions involving catalysts.



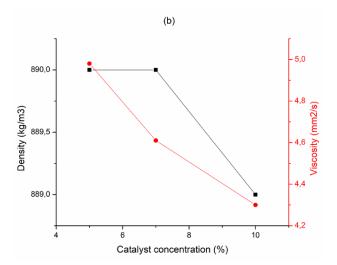


Figure 10. The effect of catalyst concentration on density and viscosity on the reaction temperature: (a) 55 °C and (b) 60 °C

Table 5. Methyl ester content, retention time series based on GC-MS chromatogram.

Retention time (min)	Methyl ester	Structure	Concentration (%)
6.364	Methyl caproate	$\mathrm{C_7H_{14}O_2}$	0.28
9.559	Methyl caprylate	$\mathrm{C_9H_{18}O_2}$	4.29
11.053	Methyl caprate	$C_{11}H_{22}O_2$	0.11
12.493	Methyl caprinate	$C_{11}H_{22}O_2$	4.52
13.825	Methyl undecanoate	$C_{12}H_{24}O_2$	0.24
15.150	Methyl laurate	$C_{13}H_{26}O_{2}$	25.63
16.308	Methyl tridecanoate	$C_{14}H_{28}O_2$	0.22
17.482	Methyl myristate	$C_{15}H_{30}O_{2}$	15.33
18.539	Methyl palmitate	$C_{17}H_{34}O_{2}$	0.28
19.371	Methyl palmitoleate	$C_{17}H_{32}O_{2}$	0.24
19.605	Methyl palmitate	$C_{17}H_{34}O_2$	12.24
20.329	Methyl cis-10-heptadecanoate	$C_{17}H_{32}O_{2}$	0.08
20.568	Methyl palmitate	$C_{17}H_{34}O_{2}$	0.11
21.072	Methyl stearate	$C_{19}H_{38}O_{2}$	0.16
21.223	Methyl linoleate	$C_{19}H_{34}O_{2}$	1.26
21.301	Methyl oleate	$C_{19}H_{36}O_{2}$	8.20
21.523	Methyl stearate	$C_{19}H_{38}O_{2}$	3.64
23.076	Metil cis-11-eicosenoate	$C_{21}H_{40}O_2$	0.15
23.290	Methyl 18-methyl nonadecanoate	$C_{21}H_{42}O_2$	0.16
25.244	Lauryl laurate, dodecyl ester	$\mathrm{C}_{24}\mathrm{H}_{48}\mathrm{O}_2$	0.16

3.7.1 Effect of Catalyst on Density and Viscosity of Biodiesel

Figure 10 shows the effect of adding a catalyst on the density and viscosity of biodiesel at reaction temperatures of 55 and 60 °C. Adding a catalyst reduces density and viscosity with a linear trend at both reaction temperatures. At a reaction temperature of 55 °C, a significant decrease in density occurred at a concentration of 5% to 7 wt%. However, at a reaction temperature of 60 °C, a significant decrease in density occurred at a concentration of 7% to 10 wt%. It shows conformity with the theory [30] that increasing temperature causes an increase in collisions between reactant particles, which causes molecular interactions with the catalyst to become stronger, making it easier to form products.

3.7.2 Characteristics of Biodiesel

Biodiesel characterization, including viscosity, density, oxidation stability, and acid number, was carried out at the mole ratio 1:76, which obtained the best results (Table 6). The results show viscosity and density parameters that meet the national standard range [31]. Biodiesel viscosity has an important role in the fuel injection process. The highest viscosity was achieved with a 5 wt% catalyst of 4.98 mm²/s, while the lowest viscosity was achieved with a 10

wt% catalyst, of 4.3 mm²/s. In applications, viscosity and density affect the flow of fuel pumped by the system, which then flows to the engine injector into the combustion chamber [32] so that the value that meets the standard range is the value that has been determined based on experience in the field.

Furthermore, the iodine number illustrates the fuel's ability to withstand oxidation processes that damage quality. The higher the value, the more double bonds in the ester react with I2, providing an opportunity for accelerated biodiesel oxidation. The results show value that meet the standards. The acid number is an illustration of the success of the biodiesel reaction. The decreased acid value of waste cooking oil to methyl ester proves this. Before processing, waste cooking oil has a high acid number of 3.91 mg KOH/g. After esterification, the acid number decreased drastically to 0.43 mg KOH/g. An 89% reduction in acid number was obtained in this study. The esterification reaction reduces the acid content in the final product, making biodiesel more stable, and corrosion-resistant.

Our biodiesel is then compared with other researchers in a comparative analysis (Table 7). They used catalyst types and raw materials that were primarily comparable to our study's. In the density parameters, the results showed slight variation from one another; however, in the

Table 6. Characteristics of biodiesel with a mole ratio of oil: methanol of 1:76.

Parameter	Catalyst weight (%)	Temperature (°C)	Results	SNI Biodiesel (7182-2015)
Density at 40 °C (kg/m³)	5		900	- 850-890
	7	55	890	
	10		890	
	5		890	
	7	60	890	
	10		889	
Kinematic Viscosity at 40 °C (mm²/s)	5		6.78	- 2.3-6.0
	7	55	5.31	
	10		4.74	
	5		4.98	
	7	60	4.61	
	10		4.30	
Iodine number (g I ₂ / 100 g	10	60	17.17	Max. 115
Acid number (mg KOH/g)	10	60	0.43	Max. 0.5

Table 7. Comparison of related studies on biodiesel

Parameter	SNI Biodiesel (7182-2015)	Our work	Shah <i>et al</i> . [13]	Bhatia <i>et al</i> . [15]	Latebo <i>et al</i> . [23]
Density at 40 °C (kg/m³)	850-890	889	873	870	890
Kinematic viscosity at 40 °C (mm²/s)	2.3-6.0	4.30	3.21	3.90	0.47
Iodine number (g I ₂ /100g)	Max. 115	17.17	44	-	124
Acid number (mg KOH/g)	Max. 0.5	0.43	0.38	-	1.20

kinematic viscosity parameters, the findings in Latebo *et al.* [23] were significantly different. It is unexpected, as kinematic viscosity typically does not deviate much from the standard range.

We also see varying results from our work and other researchers in the parameters of iodine and acid numbers. However, these variations are still within the standard range, except in ref. [23]. It may be caused by the feedstock, which is acid oil with an acid number and a much higher percentage of free fatty acids than ours.

In biodiesel, research generally suggests a two-stage process: esterification transesterification to obtain high biodiesel yield Additionally, washing after and purity. transesterification and рΗ checking recommended to validate the final biodiesel However, in this study, methyl product [33]. had been formed without esters transesterification process and non-standardized purity. If we refer to an experience, a study mixed methyl ester (biodiesel) with non-standardized purity, blended with kerosene with six variations in a ratio [34]. The results provide density, acid number, iodine number, and saponification number improvement information. Therefore, this valuable information should be considered for future research on biodiesel blends with pure diesel fuel. However, it is essential to take note of certain factors, such as using the feedstock in the FFA value range of 1-2 % and maintaining an acid number below 4 mg KOH/g. Additionally, larger equipment should be utilized for reproduction to ensure accurate results.

4. Conclusion

Biodiesel synthesis has been done through the esterification reaction of waste cooking oil (WCO) and methanol using sulfuric acidimpregnated biochar catalyst via leaching method. The most significant biodiesel yield was 84.5%, achieved at 10 wt% catalysts, temperature 60 °C, mole ratio 1:76, for 3 h. Biodiesel characterization shows that the purity of biodiesel is 77.30%. The catalyst with this method in question complies with national biodiesel standards regarding density, viscosity, iodine number, and acid number. As such, it holds significant promise for further development.

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CRediT Author Statement

M.I. Sofyan: Conceptualization, Methodology, Resources. Investigation, Characterization Analysis, Writing, Review, Editing, Supervision; P.J. Mailani and A.W. Setyawati: Experimental, Draft Preparation, Data Curation; S. Sulistia and F. Suciati: Resources, Biomass Preparation, Data Curation; L. Hauli, R.A. Putri, and S.T.C.L. Ndruru: Characterization Analysis, Writing; R.S. Mawarni, Y. Meliana: Resources, Investigation, Validating, Draft Preparation; N. Nurhayati and J. Joelianingsih: Review, Editing, and Supervision. All authors have read and agreed to the published version of the manuscript.

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