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Biodiesel Synthesis with Different Feedstock Oils: Free Fatty Acid Analysis and Fuel Properties Characterization

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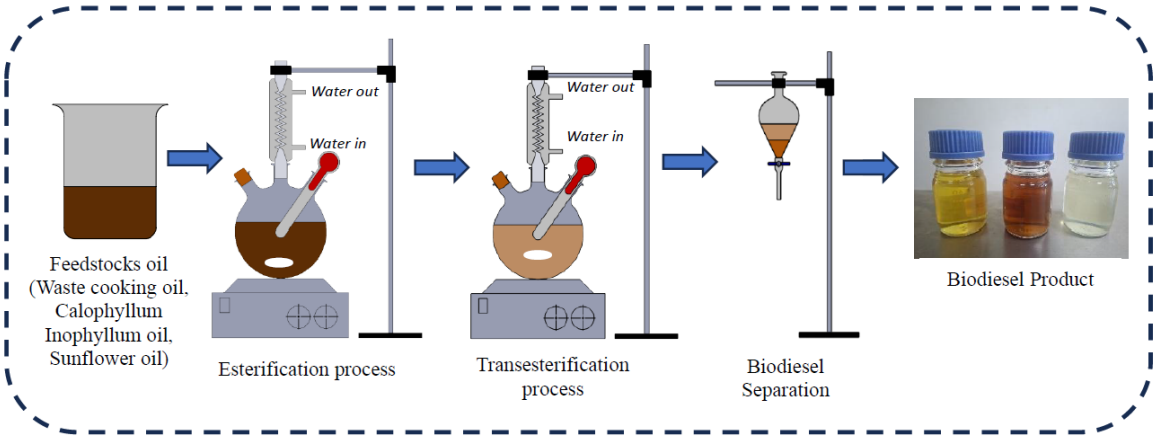
GRAPICHAL ABSTRACT



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ABSTRACT

The present study evaluates the feasibility of non-edible and waste oils as sustainable feedstocks for biodiesel production through a two-step esterification–transesterification process. Waste cooking oil, *Calophyllum inophyllum* oil, and sunflower oil were employed as raw materials, with sodium hydroxide used as the base catalyst during transesterification. The initial free fatty acid content varied significantly among the feedstocks, ranging from 29.38% in *Calophyllum inophyllum* oil to 3.20% in waste cooking oil and 1.28% in sunflower oil. Acid-catalyzed esterification effectively reduced th e free fatty acid levels, enabling efficient conversion during the subsequent transesterification step. Fourier transform infrared spectroscopy confirmed the successful formation of fatty acid methyl esters, indicated by the presence of characteristic ester functional group absorption bands. Methyl oleate is the dominant component in all biodiesel samples, reaching 83.12% in sunflower oil biodiesel. The produced biodiesel exhibited kinematic viscosities of 4.61-5.63 cSt and flash points of 164-175 °C, meeting ASTM D6751 specifications.

1. INTRODUCTION

The continuous rise in global energy demand, along with the gradual depletion of fossil fuel resources and growing environmental concerns, has encouraged significant research into sustainable and renewable energy alternatives [1-2]. Biodiesel has emerged as a promising substitute for petroleum-based diesel among renewable energy options, primarily due to its renewable characteristics, biodegradability, and lower greenhouse gas emissions [3-5]. As an environmentally friendly fuel that is compatible with conventional diesel engines, biodiesel holds great potential to support the transition towards a low-carbon economy [6-8]. However, conventional biodiesel production still heavily depends on edible vegetable oils such as soybean and palm oils. The use of edible feedstocks directly competes with food production, raising ethical and economic concerns about food security, land-use changes, and food price fluctuations [9, 10]. Therefore, the exploration of alternative oil sources that do not compete with human consumption, particularly non-edible and waste oils, has become a critical focus for achieving sustainable biodiesel production.

Non-edible and waste feedstocks such as *Calophyllum inophyllum* (nyamplung oil), sunflower oil, and waste cooking oil (WCO) have received growing attention as promising raw materials for biodiesel synthesis [11, 12]. These feedstocks are abundantly available, relatively inexpensive, and remain underutilized. Nevertheless, each feedstock has distinct chemical characteristics, particularly fatty acid and free fatty acid (FFA) content, which significantly influence transesterification efficiency [13]. A high FFA content, especially in waste cooking oil, can interfere with base-catalyzed transesterification by promoting soap formation (saponification), thereby reducing biodiesel yield [14]. Various technologies have been developed to reduce biodiesel production costs, employing either a one-step or a two-step process [15]. In the one-step method, transesterification is carried out directly and is generally suitable only for low-FFA oils, as excessive FFA may inhibit the reaction. The main drawback of this method lies in the higher requirements for catalyst and alcohol compared to the two-step approach [16]. The two-step method, consisting of an initial esterification followed by transesterification, or even double esterification in some studies, is more effective for oils with high FFA content [17]. When the FFA level exceeds 1%, the initial esterification step becomes crucial to reduce it before transesterification. Therefore, the two-step process is widely regarded as a more efficient approach for converting various vegetable oils into biodiesel [18, 19]. Accurate analysis of FFA content and the application of pre-esterification are thus essential for enhancing biodiesel yield, while understanding FFA variations among feedstocks helps select the appropriate catalyst and reaction parameters.

The transesterification reaction in biodiesel synthesis can utilize homogeneous acid catalysts such as HCl, H_3PO_4, and H_2SO_4, or base catalysts such as NaOH, KOH, and $NaOCH_3$ [20]. Kasirajan et al. reported the production of biodiesel from *Chrysophyllum albidum* seed oil using KOH as a base catalyst, achieving a conversion efficiency of 99.2% under optimized conditions: an oil-to-methanol molar ratio of 1:9, 1 wt% KOH catalyst, stirring speed of 500 rpm, reaction time of 40 minutes, and temperature of 65 °C [19]. Similarly, Efavid et al. investigated the production of biodiesel from Citrullus vulgaris (watermelon) seed oil using NaOH as a catalyst. The influence of NaOH concentration on biodiesel yield was evaluated at 0.13, 0.15, and 0.18 g, with an oil-to-methanol molar ratio of 5:1 under reaction times of 90, 120, and 150 minutes at 60 °C [21]. The highest yield of 70% was obtained at 0.13 g NaOH, compared to 53% and 49% for 0.15 g and 0.18 g, respectively. The physicochemical properties of the produced biodiesel were as follows: density of 0.88 ± 0.02 g/cm^3, kinematic viscosity of 30.80 mm^2/s, acid value of 0.96 ± 0.21 mg KOH/g, and flash point of 141.2 ± 0.1 °C.

In addition to conversion efficiency, the physicochemical properties of biodiesel, such as viscosity, acid value, and flash point, play a crucial role in determining engine performance and compliance with fuel quality standards (ASTM D6751 or EN 14214) [10]. The fatty acid composition of the feedstock oil strongly influences these properties. Therefore, comparative evaluation of the fuel properties obtained from different feedstocks is essential to identify the most suitable raw material for biodiesel production [22]. Another important aspect of biodiesel utilization is its environmental performance, particularly its exhaust emissions [23]. The type of feedstock directly affects the combustion emissions profile in diesel engines. Hence, emission testing for carbon

monoxide (CO), nitrogen monoxide (NO), and nitrogen oxides (NO_x) is necessary to assess environmental impact and ensure compliance with current emission regulations.

The selection of the three oil feedstocks in this study was hypothesis-driven rather than purely empirical. Waste cooking oil (WCO), *Calophyllum inophyllum* oil (CIO), and sunflower oil (SFO) were deliberately chosen to represent a wide range of free fatty acid (FFA) contents, while also reflecting differences in availability, sustainability, and economic implications. Specifically, CIO was selected as a representative non-edible oil with a very high FFA content, which is commonly encountered in second- and third-generation biodiesel feedstocks. It is relatively abundant, particularly in the Madura region, and poses significant technical challenges in transesterification due to its high acidity. WCO is a waste-derived feedstock with moderate FFA levels, is widely available at the local level, cost-effective, and highly sustainable as it valorizes waste streams. In contrast, SFO was chosen as a low-FFA oil with a relatively stable fatty acid composition, thereby serving as a benchmark for comparison with the other feedstocks.

This study offers novelty through a systematic comparative approach involving three oil feedstocks with markedly different free fatty acid (FFA) characteristics, namely waste cooking oil, *Calophyllum inophyllum* oil, and sunflower oil, processed under identical esterification–transesterification conditions. Unlike previous studies that typically focus on a single feedstock, the present work specifically emphasizes the relationships among initial and final FFA values, the resulting biodiesel conversion efficiency, and fuel quality. Furthermore, this study integrates the analysis of FFA reduction, methyl ester composition, and physicochemical properties of the produced biodiesel within a single evaluation framework. This comprehensive approach provides deeper insight into how feedstock characteristics influence biodiesel performance and highlights their potential utilization as sustainable energy sources.

2. EXPERIMENTAL METHODS

2.1. Materials and equipments

The materials and equipment used in this study included a hotplate magnetic stirrer, a three-neck round-bottom flask, beakers, a volumetric pipette, a burette, an Erlenmeyer flask, an analytical balance, a condenser, a diesel engine, and a gas analyzer. The reagents used were methanol and sodium hydroxide (NaOH), all purchased from Merck. Sulfuric acid (H_2SO_4) was obtained from Smart Lab, and phenolphthalein (PP) was used as an indicator. The oil feedstocks consisted of waste cooking oil (WCO), Calophyllum Inophyllum oil (CIO), and sunflower oil (SFO).

2.2. Synthesis of Biodiesel from Waste Cooking Oil, *Calophyllum inophyllum* Oil, and Sunflower Oil

2.2.1. Esterification process

In the present study, the reaction conditions were slightly modified from those applied in our previous work [24]. The esterification stage aimed to reduce the free fatty acid (FFA) content of the waste cooking oil. The reaction was carried out between the oil and methanol using sulfuric acid (H_2SO_4) as the catalyst at a concentration of 2% (v/v) relative to the oil. The molar ratio of oil to methanol was maintained at 1:6. The reaction was conducted at 60 °C with a stirring speed of 600 rpm for 60 minutes. After completion, the mixture was allowed to settle for 24 hours to form two layers, and the upper layer was collected for transesterification. The same procedure was repeated for *Calophyllum inophyllum* and sunflower oils.

The free fatty acid (FFA) content in raw and esterified oils was determined using the acid-base titration method according to AOCS Ca 5a-40 and Suriyanti et al [25]. Approximately 0.1 g of oil sample was placed in an Erlenmeyer flask, followed by the addition of 3 mL of isopropanol. The mixture was heated to 40 °C until completely dissolved. Two drops of phenolphthalein indicator were added, and the solution was titrated with 0.01 N NaOH until a faint pink color appeared. The FFA content was calculated using the following equation [26]:

$$\% \text{ FFA} = \frac{V \times N \times Mr}{m \times 10} \quad (1)$$

where V is the volume of NaOH used (mL), N is the normality of NaOH, M_n is the molecular weight of oleic acid (g/mol), and m is the mass of the oil sample (g).

2.2.2. Transesterification process

In the transesterification process, methanol was first mixed with sodium hydroxide (NaOH) at 0.5 wt% relative to the oil weight, and the mixture was stirred until fully dissolved to form sodium methoxide. This mixture was then added to the pre-esterified oil. The molar ratio of oil to methanol was maintained at 1:9. The reaction was conducted at 60 °C with a stirring speed of 800 rpm for 2 hours. Upon completion, the mixture was allowed to settle in a separating funnel, forming two layers; the upper biodiesel layer was carefully collected. The produced biodiesel was then characterized using Gas Chromatography–Mass Spectrometry (GC–MS) to determine its methyl ester composition and Fourier Transform Infrared Spectroscopy (FTIR) to identify the functional groups. The physicochemical properties of the biodiesel were also evaluated, including acid value, flash point, viscosity, and calorific value. The stages of the biodiesel production process are illustrated in Figure 1. In this study, free fatty acid (FFA) content was determined in triplicate to ensure analytical reliability. In contrast, biodiesel production was conducted once for each feedstock oil, with experimental variation introduced by using different feedstocks.

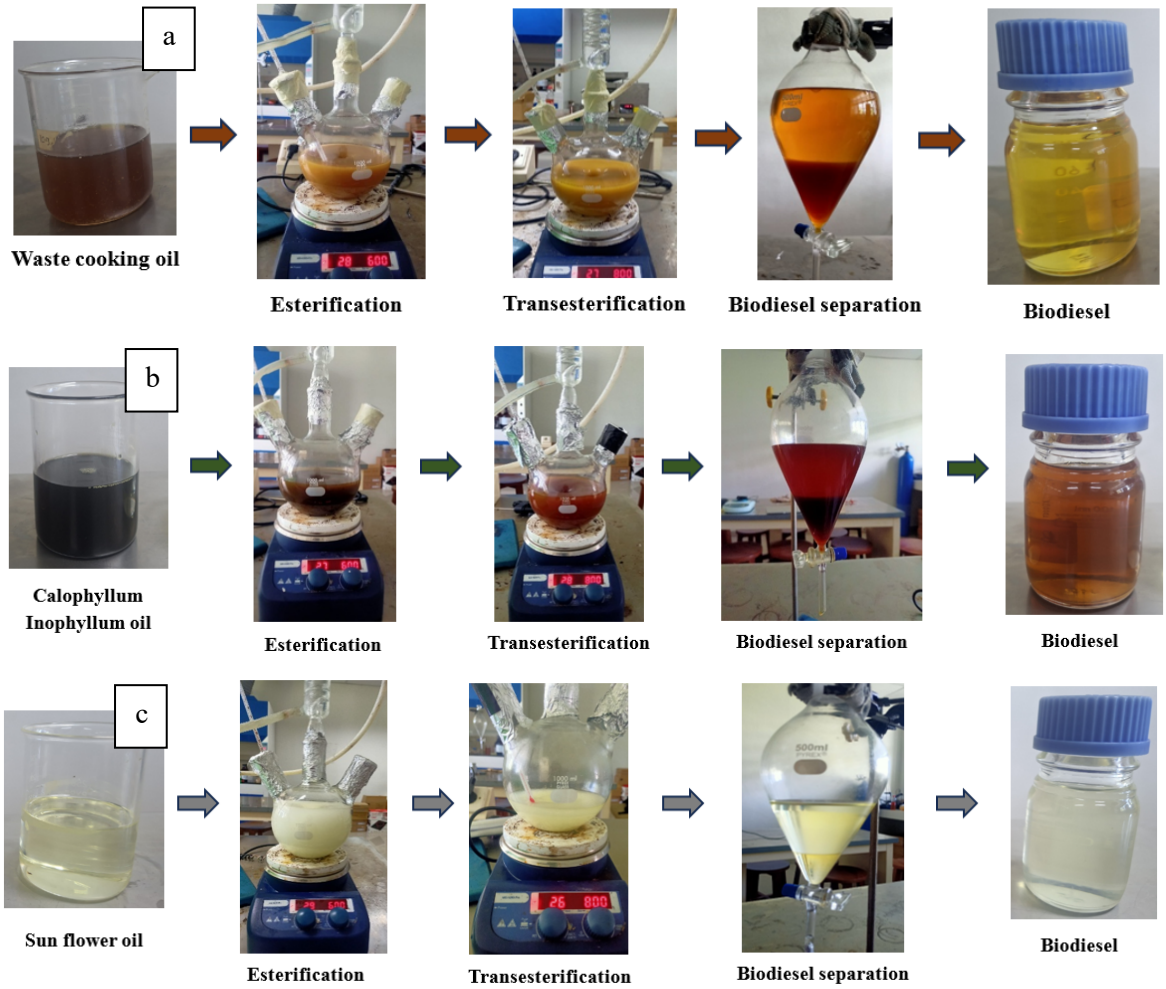

Figure 1. The stages of the biodiesel production process

3. RESULTS AND DISCUSSIONS

Figure 2 illustrates the variation in free fatty acid (FFA) content among different types of vegetable oil feedstocks and the corresponding biodiesel produced. The highest FFA value was observed in *Calophyllum inophyllum* oil (CIO) at 29.38%, followed by waste cooking oil (WCO) at 3.2%, and sunflower oil (SFO) at 1.28%. The high FFA content in CIO indicates a large amount of

free fatty acids present due to oxidation and triglyceride hydrolysis during storage or heating. This condition is commonly observed in non-edible oils, leading to soap formation (saponification) during base-catalyzed transesterification [27, 28]. Therefore, oils with high FFA levels, such as CIO, must undergo a pre-esterification stage using an acid catalyst to reduce the FFA content to below 1%, making them suitable for further transesterification processing [29]. After esterification and transesterification, the FFA content decreased significantly across all feedstocks The FFA value of biodiesel derived from waste cooking oil (WCB) was found to be 0.71%.

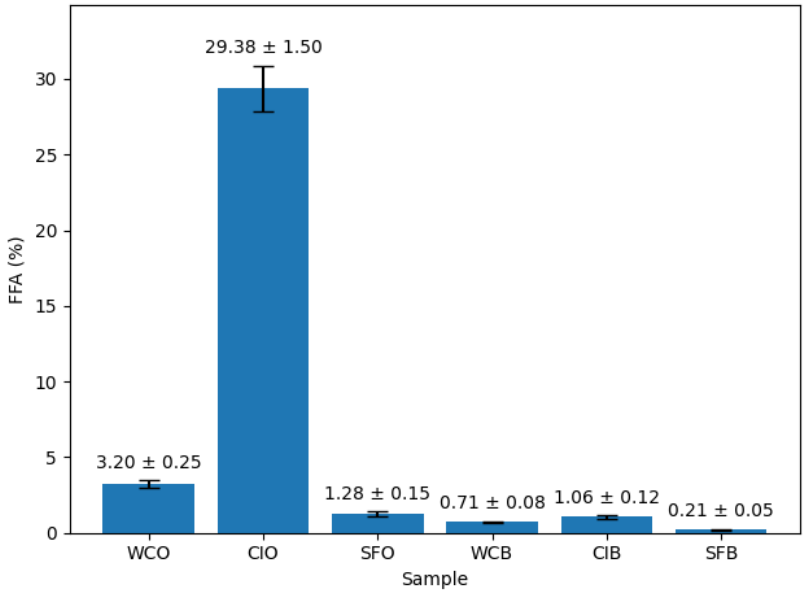

Figure 2. FFA content of feedstock oils and biodiesel

In comparison, those of biodiesel derived from *Calophyllum inophyllum* (CIB) and sunflower oil (SFB) were 1.06% and 0.21%, respectively. This substantial reduction demonstrates the effectiveness of acid-catalyzed esterification in converting free fatty acids into methyl esters, thereby lowering oil acidity and improving the efficiency of the subsequent transesterification reaction. Recent studies have reported that decreasing the FFA content to below 1% is a key indicator of successful pre-esterification, as it prevents soap formation and improves biodiesel yield [30, 31]. These findings are consistent with recent research showing that high FFA conversion to methyl esters can be effectively achieved using heterogeneous catalysts based on metal oxides or solid waste-derived materials [28]. The high FFA content observed in CIO prior to treatment is primarily attributed to the natural hydrolysis of triglycerides occurring during post-harvest handling and storage. The fruit contains endogenous lipases that catalyze the hydrolysis of triglyceride ester bonds into glycerol and free fatty acids, particularly when the fruit tissue is damaged or exposed to elevated moisture levels. Water acts directly as a reactant in the hydrolysis reaction; therefore, humid storage conditions or delays in oil extraction can accelerate the increase in FFA content [32, 33]. In addition, exposure to oxygen, elevated temperature, and light may induce oxidative degradation, further contributing to the formation of free fatty acids. As a non-edible oil, CIO typically exhibits higher initial FFA levels than edible vegetable oils, necessitating pretreatment steps, such as acid esterification, to reduce FFA content prior to transesterification.

Overall, the results reveal a strong correlation between the initial FFA content of the feedstock and the conversion efficiency of the biodiesel process. Feedstocks with high FFA levels, such as *Calophyllum inophyllum* oil, require stronger chemical treatment, such as ZrO_2/Al_2O_3 or $SnCl_2$ catalysts, to achieve optimal conversion. In contrast, low-FFA feedstocks such as sunflower oil require milder processing and can produce high-quality biodiesel with minimal residual FFA. Therefore, controlling the FFA level through an effective pre-esterification step is crucial for ensuring both the efficiency of the transesterification reaction and the final biodiesel quality in compliance with international fuel standards [34].

The presence of free fatty acids (FFA) in biodiesel feedstock plays a critical role in the transesterification reaction mechanism, catalyst performance, soap formation, and the final quality of biodiesel. Mechanistically, FFA are fatty acids generated from triglyceride hydrolysis and oil degradation, characterized by the presence of free carboxylic groups (–COOH). In base-catalyzed transesterification processes (e.g., NaOH or KOH), FFA react with the alkaline catalyst through an acid–base neutralization reaction to form soap (saponification). As a result, active catalyst species are consumed during soap formation, thereby reducing the number of available active sites for the primary transesterification reaction between triglycerides and methanol to produce fatty acid methyl esters (FAME) [35-37]. This side reaction not only decreases catalyst efficiency but also lowers the overall transesterification rate and FAME conversion, as the base catalyst is preferentially consumed by FFA rather than facilitating methoxide ($–OCH_3$) formation for nucleophilic substitution of triglycerides. Consequently, biodiesel produced from high-FFA feedstocks tends to exhibit lower conversion yields than those from low-FFA oils, as widely reported in the literature. Increasing FFA content significantly reduces methyl ester conversion efficiency, biodiesel yield, and fuel quality.

TABLE I. Comparison of FFA test results under different feedstocks and processing methods.

Reference	Feedstock	Initial FFA (%)	Final FFA (%)	Yield / Conversion (%)	Method & Catalyst	Remarks
This study	WCO, CIO, SFO	3.20 – 29.38	0.21 – 1.06	97 - 98	H_2SO_4 esterification + NaOH transesterification	Two-step approach effectively reduced high FFA prior to base-catalyzed transesterification
Abdullah et al. (2021) [38]	Waste cooking oil	4.26	0.14	96.3	Acid esterification + base transesterification	Significant FFA reduction resulted in biodiesel meeting SNI 7182:2015
Roza et al. (2025) [39]	Waste palm oil	~0.75 – 1.00	<0.50	>90	NaOH tranesterification	Reduction of FFA below the biodiesel standard limit after transesterification
Amaliah et al. (2025) [40]	Waste cooking oil	3.81	1.32	-	Esterification + Transesterification (Coconut Fiber catalyst)	Final FFA slightly above 1%, indicating partial limitation of the heterogeneous catalyst
Thein et al. (2019) [41]	Crude Rice Bran Oil	8-10	0.61-0.7	-	Acid esterification (H_2SO_4)	Pretreatment reduced FFA below 1%, making CRBO suitable for biodiesel production
Putra & Abdullah (2025) [42]	Waste cooking oil	3.75	1.74	-	H_2SO_4 esterification (RSM optimization)	Yield decreased when FFA was not reduced below 1%

Overall, Table 1 demonstrates that the success of biodiesel production is strongly influenced by the ability to reduce the initial FFA content to approximately 1% or lower prior to base-catalyzed transesterification. The studies compared consistently indicate that acid esterification pretreatment is effective in lowering high FFA levels and preventing saponification reactions, which can otherwise reduce catalyst efficiency and methyl ester conversion. When the final FFA content is successfully reduced below this critical threshold, biodiesel yield and fuel quality tend to improve. Conversely, insufficient FFA reduction leads to decreased biodiesel yield. Therefore, controlling FFA content is a key parameter for optimizing biodiesel production processes using feedstocks with diverse free fatty acid profiles.

The FTIR spectra in Figure 3 illustrate the characterization results for feedstock oils and biodiesel produced from various feedstocks. The absorption peaks detected within specific

wavenumber ranges correspond to characteristic functional groups associated with the chemical structure of biodiesel (fatty acid methyl ester, FAME). A strong absorption band observed around 1746 cm^{-1} is attributed to the stretching vibration of the ester carbonyl group (C=O) [43], which serves as the primary indicator of biodiesel formation during the transesterification reaction. In contrast, the raw oils, particularly CIO, exhibit weaker and broader peaks with slightly different spectral patterns. Additionally, the bands observed at 2922 cm^{-1} and 2856 cm^{-1} correspond to the asymmetric and symmetric stretching vibrations of the C–H bond in $–CH_2$ groups, derived from the long aliphatic chains of fatty acids. These peaks appear consistently in both biodiesel and raw oil samples, indicating that the hydrocarbon chains remain intact after the transesterification process. Meanwhile, the absorption bands at 1464 cm^{-1} and 1365 cm^{-1} are attributed to the bending vibrations of $–CH_2$ and $–CH_3$ groups, respectively. This observation is expected since the long aliphatic (hydrocarbon) chains persist before and after transesterification [44]. It implies that the transesterification reaction does not alter the hydrocarbon backbone but only converts the glyceride bonds into ester linkages.

Furthermore, the peaks observed around 1169 cm^{-1} and 1017 cm^{-1} are characteristic of C–O stretching vibrations of ester groups, further confirming the formation of methyl esters [45]. In the raw oils, these C–O bands are still present but appear weaker and more complex due to the presence of glycerol structures within triglycerides. The emergence and intensification of these ester-related peaks provide strong evidence for the successful formation of new ester bonds after transesterification. Interestingly, a distinct absorption band at 722 cm^{-1} corresponds to the rocking vibration of the $–CH_2$ group in long hydrocarbon chains [46], which is typically found in compounds containing extended alkyl structures such as biodiesel. In addition, raw oils sometimes exhibit a weak and broad absorption band in the 3200–3500 cm^{-1} region (–OH stretching), particularly in CIO, due to the presence of glycerol or residual moisture [11]. In biodiesel samples, this band either disappears or becomes very weak, as glycerol has been separated as a by-product during the transesterification process. The disappearance of the –OH absorption band provides further confirmation that the crude oil has been successfully converted into biodiesel.

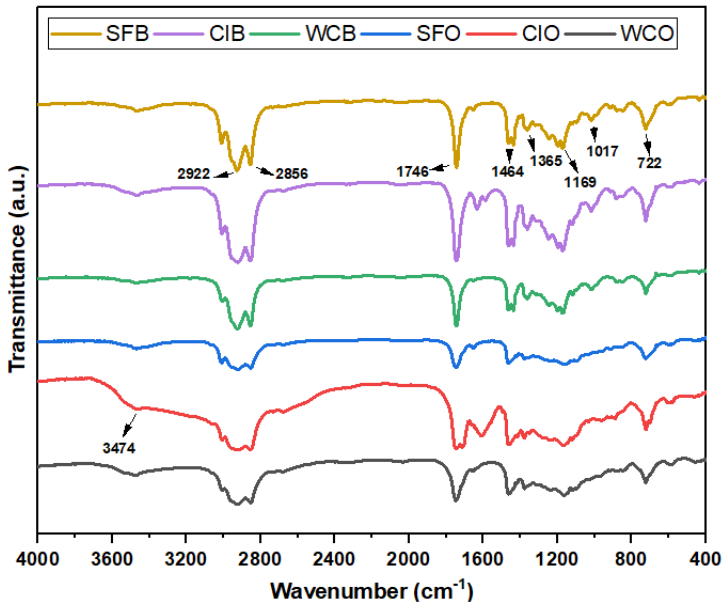

Figure 3. FTIR spectra of SFB (sunflower biodiesel), CIB (*Calophyllum inophyllum* biodiesel), WCB (waste cooking oil biodiesel), SFO (sunflower oil), CIO (*Calophyllum inophyllum* oil), and WCO (waste cooking oil)

The data presented in Table 2 and Figure 4 represent the results of methyl ester compound analysis in biodiesel produced from three different types of feedstock oils using Gas Chromatography–Mass Spectrometry (GC–MS). The GC-MS analysis of biodiesel derived from these three distinct feedstocks revealed variations in methyl ester composition, reflecting the differences in fatty acid profiles of the raw materials. In biodiesel produced from waste cooking oil,

the predominant components were methyl oleate (53.17%) and methyl palmitate (38.36%), accompanied by smaller amounts of methyl stearate (6.64%), methyl myristate (0.68%), and methyl aracate (0.31%). These results are consistent with those reported by Khan *et al. and* Ginting *et al.* [47, 48], who also found that methyl oleate was the dominant component in biodiesel produced from waste cooking oil. This composition suggests that the waste cooking oil used likely originated from palm oil or a mixture of other vegetable oils rich in oleic and palmitic acids. A high concentration of methyl oleate contributes to good oxidative stability, while the relatively high palmitate content enhances thermal stability but may reduce low-temperature performance.

In biodiesel derived from *Calophyllum inophyllum* oil, a different profile was observed, with methyl oleate as the major component (60.4%), followed by methyl palmitate (17.23%) and methyl stearate (18.71%). The relatively high amount of methyl stearate indicates a significant presence of long-chain saturated fatty acids in CI oil. Minor components such as methyl elaidate (0.38%) and methyl aracate (0.8%) reveal the presence of trans-unsaturated and long-chain saturated fatty acids, respectively. A high oleate content in biodiesel from CI oil was also reported by Muderawan *et al.* [49], who found 43.41%. The elevated oleate level contributes to oxidative stability, whereas the high stearate content may raise the biodiesel's melting point.

Meanwhile, biodiesel produced from sunflower oil exhibited a clearly dominant composition of methyl oleate (83.12%), followed by methyl palmitate (8.68%) and methyl stearate (5.88%). The exceptionally high oleate content is characteristic of high-oleic sunflower oil, resulting in biodiesel with excellent oxidative stability, low viscosity, and superior cold flow properties compared to biodiesel with higher saturated fatty acid content. Minor compounds such as methyl behenate (0.88%) and methyl lignocerate (0.28%) indicate the presence of very long-chain fatty acids (C22–C24), which, although present in small quantities, can contribute to an increased flash point. Santoso *et al.* [50] also reported that the dominant component in sunflower biodiesel was methyl oleate, amounting to 46.79%.

TABLE II. GCMS analysis results of biodiesel samples

Biodiesel	Methyl ester compounds	Retention time (min)	Area (%)
WCB	Methyl myristate	11.78	0.68
	Methyl palmitate	14.23	38.36
	Methyl oleate	16.09	53.17
	Methyl stearate	16.19	6.64
	Methyl aracate	17.84	0.31
CIB	Methyl palmitate	14.11	17.23
	Methyl oleate	16.07	60.4
	Methyl stearate	16.23	18.71
	Methyl elaidate	17.63	0.38
	Methyl aracate	17.85	0.8
SFB	Methyl palmitate	14.07	8.68
	Methyl oleate	16.16	83.12
	Methyl stearate	16.23	5.88
	Methyl behenate	19.55	0.88
	Methyl lignocerate	21.11	0.28

Overall, differences in methyl ester composition among biodiesels derived from the three feedstocks are influenced by the oils' intrinsic properties, which, in turn, determine the physicochemical characteristics of the resulting biodiesel. Waste cooking oil exhibits a mixed profile with relatively balanced proportions of saturated and unsaturated fatty acids, yielding biodiesel with moderate oxidative stability and an intermediate melting point [51]. CIO-based biodiesel contains a higher proportion of saturated compounds, enhancing thermal stability but potentially reducing low-temperature performance. Conversely, sunflower oil-based biodiesel is dominated by unsaturated compounds, resulting in good oxidative stability and excellent low-temperature properties, though it is more susceptible to oxidation than biodiesel with higher saturated content [52].

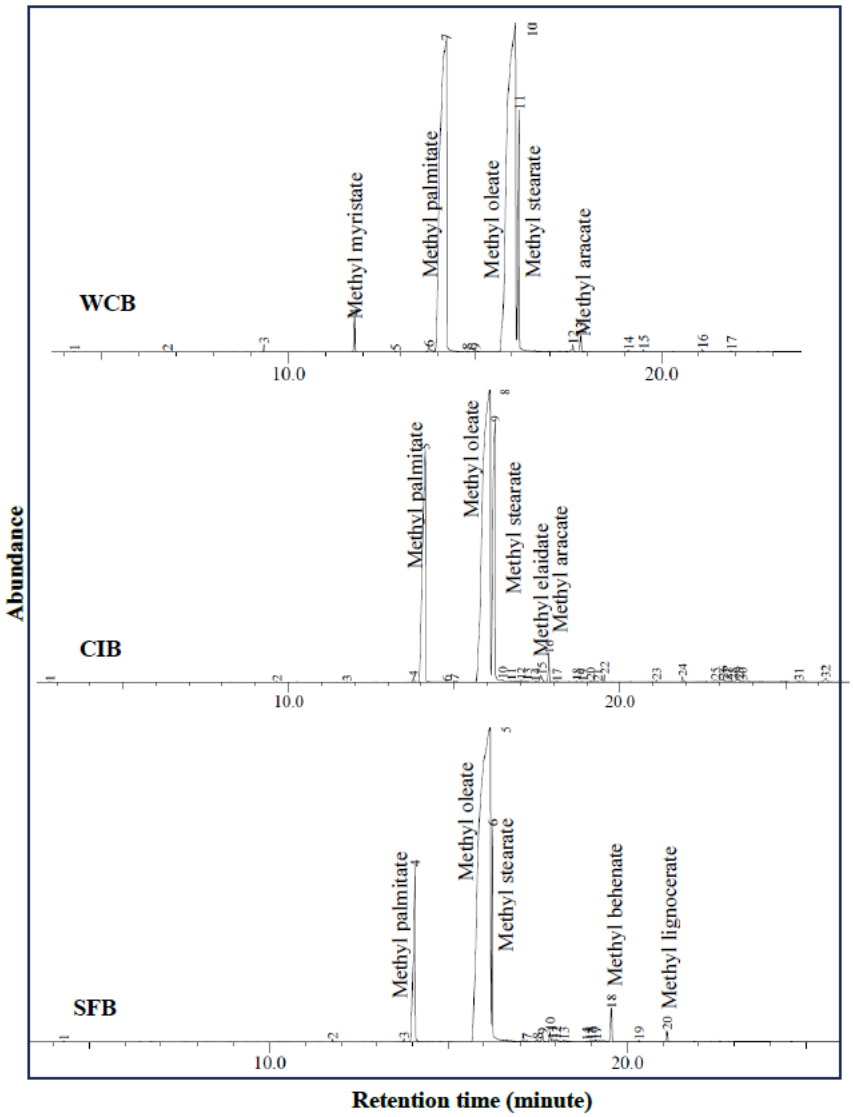

Figure 4. Chromatogram of WCB, CIB and SFB

TABLE III. Properties of Biodiesel

No	Parameter	Result			Unit	Test Method	Standard (ASTM D6751)
		WCB	CIB	SFB			
1	Kinematic Viscosity at 40 °C	5.06	5.63	4.61	cSt	ASTM D445	1.9-6
2	Flash point	164	175	170	°C	ASTM D 92	Min 130
3	Calorific value	9578	9273	9543	Kcal/Kg	Bomb calorimeter	-
4	Acid Number	0.12	1.85	0.083	mg-KOH/g	SNI 7182:2015	Max 0.5

The physicochemical properties of biodiesel presented in Table 3 indicate that all three feedstocks can be successfully converted into biodiesel with characteristics closely matching or meeting the ASTM D6751 standards. The kinematic viscosity at 40 °C for the three biodiesel samples ranged from 4.61 to 5.63 cSt, which falls within the ASTM standard limit of 1.9–6.0 cSt. Viscosity is a crucial parameter as it affects fuel atomization and injection performance in diesel engines. The slightly higher viscosity observed for CIB (5.63 cSt) suggests a greater proportion of long-chain fatty acids compared to SFB (4.61 cSt), while WCB (5.06 cSt) lies between the two. Despite these differences, all samples remain within an acceptable range for efficient combustion without posing risks of injector clogging. The flash points of the three biodiesel samples were relatively high, at 164

°C (WCB), 175 °C (CIB), and 170 °C (SFB). These values are well above the minimum ASTM requirement of 130 °C, indicating that the produced biodiesel is safe for handling and storage due to its low volatility and reduced fire risk at lower temperatures. The high flash points also imply that the residual methanol content in the biodiesel is minimal, as methanol is known to decrease flash point values [53, 54]. Among the samples, CIB exhibited the highest flash point, reflecting superior thermal stability and enhanced safety compared to the others.

The calorific values of the biodiesel samples ranged between 9273 and 9578 kcal kg^{-1}, with the highest value obtained for WCB and the lowest for CIB. These calorific values represent the energy content released during combustion and are directly related to the thermal efficiency of diesel engines. Although slightly lower than that of conventional diesel fuel (approximately 10,000 kcal kg^{-1}), the values remain sufficiently high for practical diesel engine applications. In terms of acid value, WCB and SFB recorded 0.12 mg-KOH g^{-1} and 0.083 mg-KOH g^{-1}, respectively, both well below the ASTM limit of 0.5 mg-KOH g^{-1}, indicating good fuel quality and adequate oxidative stability. However, CIB exhibited a relatively high acid value of 1.85 mg-KOH g^{-1}, exceeding the standard limit. This elevated acidity suggests a higher residual free fatty acid (FFA) content, likely due to incomplete esterification or to the inherent chemical nature of CIO, which is known for its naturally high FFA levels.

4. CONCLUSIONS

This study demonstrates that biodiesel can be successfully produced from waste cooking oil, *Calophyllum inophyllum* oil, and sunflower oil using a two-step esterification–transesterification process. The esterification stage played a crucial role in reducing the high free fatty acid content of the feedstocks, particularly for *Calophyllum inophyllum* oil, where the free fatty acid level decreased from 29.38% to 1.06%. Following transesterification, all biodiesel samples exhibited clear formation of fatty acid methyl esters, as confirmed by FTIR and GC–MS analyses, with methyl oleate identified as the predominant compound in each sample. The physicochemical properties of the biodiesel, including a kinematic viscosity range of 4.61-5.63 cSt and a flash point above 160 °C, met the ASTM D6751 standard requirements. Although biodiesel derived from *Calophyllum inophyllum* oil had a higher acid value than the other samples, the overall results confirm that non-edible and waste oils are viable and sustainable feedstocks for biodiesel production. Among the investigated feedstocks, sunflower oil produced biodiesel with the most favorable fuel characteristics. In contrast, waste cooking oil and *Calophyllum inophyllum* oil offer strong potential for large-scale, low-cost biodiesel applications.

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References

[1] B. Panchal, C. H. Su, C. C. Fu, S. J. Wu, and H. Y. Juan, "Ecofriendly and cost-effective biodiesel production from water containing feedstocks through electrolysis- a review," Oct. 15, 2025, *Elsevier B.V.* doi: 10.1016/j.fuproc.2025.108277.

[2] M. Govindasamy, M. Ezhumalai, A. Munimathan, S. Dixit, S. Singh, and R. Dhairiyasamy, "Lemongrass oil as a renewable additive for enhancing the oxidation and thermal properties of calophyllum inophyllum biodiesel," *Results in Engineering*, vol. 26, Jun. 2025, doi: 10.1016/j.rineng.2025.105102.

[3] S. Brahma *et al.*, "Biodiesel production from mixed oils: A sustainable approach towards industrial biofuel production," *Chemical Engineering Journal Advances*, vol. 10, no. January, p. 100284, 2022, doi: 10.1016/j.ceja.2022.100284.

[4] A. D. Burmana, D. R. Hasibuan, Y. Benguerba, Taslim, and Iriany, "Corrosion effect in safety implication of a leakage steam traps: A case studies in biodiesel storage tank," *Results in Engineering*, vol. 25, Mar. 2025, doi: 10.1016/j.rineng.2025.104508.

[5] Taslim, S. Nova, R. Manurung, Iriany, V. Alexander, and A. D. Burmana, "Sustainable production of CaO rich-indigofera (Indigofera zollingeriana) as heterogeneous catalyst of biodiesel from refined bleached deodorized palm olein using associated transesterification process," *Results in Engineering*, vol. 26, p. 105507, Jun. 2025, doi: 10.1016/j.rineng.2025.105507.

[6] W. V. Reid, M. K. Ali, and C. B. Field, "The future of bioenergy," *Glob. Chang. Biol.*, vol. 26, no. 1, pp. 274–286, Jan. 2020, doi: 10.1111/gcb.14883.

[7] B. Shaik and A. Muni Kumari, "A review on nanoparticles as a catalyst for biodiesel production," Jul. 01, 2025, *Elsevier B.V.* doi: 10.1016/j.rechem.2025.102426.

[8] I. Tariq, M. Zubair, W. Iqbal, A. Badshah, S. M. Abbas, and A. Haider, "Alkaline earth metal oxides supported on WO3@MCM-41; bifunctional catalysts for biodiesel production from corn and waste cooking oil," *Hybrid Advances*, vol. 10, Sep. 2025, doi: 10.1016/j.hybadv.2025.100477.

[9] Rozina *et al.*, "Sustainable and eco-friendly synthesis of biodiesel from novel and non-edible seed oil of Monotheca buxifolia using green nano-catalyst of calcium oxide," *Energy Conversion and Management: X*, vol. 13, Jan. 2022, doi: 10.1016/j.ecmx.2021.100142.

[10] E. Hoque *et al.*, "A review of non-edible indigenous seeds feedstock in Bangladesh for biodiesel: Production, fuel properties and combustions performance," Oct. 01, 2025, *KeAi Communications Co.* doi: 10.1016/j.grets.2025.100240.

[11] M. Fatah, A. Hamid, Z. Rahmawati, Saiful, T. E. Purbaningtias, and A. Jakfar, "Synthesis of sodalite-natural dolomite as novel bifunctional catalyst for biodiesel production: Experimental study of performance and emissions on diesel engine," *International Journal of Renewable Energy Development*, vol. 14, no. 5, pp. 1024–1035, Sep. 2025, doi: 10.61435/ijred.2025.61434.

[12] Q. H. Hassan, N. S. Mohammed Ali, H. A. Alalwan, A. H. Alminshid, and M. M. Mohammed, "The impact of adding nanoparticles to biodiesel fuel prepared from waste sunflower oil on the performance and emission of diesel engines," *Circular Economy*, vol. 4, no. 2, Jun. 2025, doi: 10.1016/j.cec.2025.100138.

[13] J. Jayaprabakar *et al.*, "Development of Free Fatty Acid (FFA) monitoring device for evaluation of oil samples used for biodiesel production," *Heliyon*, vol. 10, no. 17, Sep. 2024, doi: 10.1016/j.heliyon.2024.e37118.

[14] J. B. Tarigan, B. Ginting, S. Perangin-angin, R. N. Sari, P. F. Sianipar, and E. K. Sitepu, "Homogenizer-intensified amidation of free fatty acids in waste cooking oil for biodiesel production," *S. Afr. J. Chem. Eng.*, vol. 46, pp. 271–276, Oct. 2023, doi: 10.1016/j.sajce.2023.08.011.

[15] S. M. Dubey, V. L. Gole, and P. R. Gogate, "Cavitation assisted synthesis of fatty acid methyl esters from sustainable feedstock in presence of heterogeneous catalyst using two step process," *Ultrason. Sonochem.*, vol. 23, pp. 165–173, 2015, doi: 10.1016/j.ultsonch.2014.08.019.

[16] M. L. Pisarello and C. A. Querini, "Catalyst consumption during one and two steps transesterification of crude soybean oils," *Chemical Engineering Journal*, vol. 234, pp. 276–283, Dec. 2013, doi: 10.1016/j.cej.2013.08.109.

[17] S. Photaworn, C. Tongurai, and S. Kungsanunt, "Process development of two-step esterification plus catalyst solution recycling on waste vegetable oil possessing high free fatty acid," *Chemical Engineering and Processing: Process Intensification*, vol. 118, pp. 1–8, 2017, doi: 10.1016/j.cep.2017.04.013.

[18] Z. Z. Cai *et al.*, "A two-step biodiesel production process from waste cooking oil via recycling crude glycerol esterification catalyzed by alkali catalyst," *Fuel Processing Technology*, vol. 137, pp. 186–193, Sep. 2015, doi: 10.1016/j.fuproc.2015.04.017.

[19] R. Kasirajan, "Biodiesel production by two step process from an energy source of Chrysophyllum albidum oil using homogeneous catalyst," *S. Afr. J. Chem. Eng.*, vol. 37, pp. 161–166, Jul. 2021, doi: 10.1016/j.sajce.2021.05.011.

[20] S. T. Al-Humairi, J. G. M. Lee, and A. P. Harvey, "Direct and rapid production of biodiesel from algae foamate using a homogeneous base catalyst as part of an intensified process," *Energy Conversion and Management: X*, vol. 16, Dec. 2022, doi: 10.1016/j.ecmx.2022.100284.

[21] J. K. Efavi *et al.*, "The effect of NaOH catalyst concentration and extraction time on the yield and properties of Citrullus vulgaris seed oil as a potential biodiesel feed stock," *S. Afr. J. Chem. Eng.*, vol. 25, pp. 98–102, Jun. 2018, doi: 10.1016/j.sajce.2018.03.002.

[22] K. M. Muhammad *et al.*, "Biodiesel production in Africa from non-edible sources: Sources, production, properties and policies," Mar. 01, 2025, *Elsevier B.V.* doi: 10.1016/j.scenv.2024.100201.

[23] P. Moonsin *et al.*, "Development of liquid biofuel properties through the blending of biodiesel from used cooking oil and pyrolysis oil from low-quality rubber waste," *Case Studies in Chemical and Environmental Engineering*, vol. 11, Jun. 2025, doi: 10.1016/j.cscee.2025.101230.

[24] A. Hamid *et al.*, "Waste Cooking Oil Biodiesel via a Sodium Hydroxide-Catalyzed Transesterification Process: Effects on Diesel Engine Performance and Emissions," 2025. [Online]. Available: http://e-jurnal.pnl.ac.id/polimesin

[25] L. Suriyanti, T. Usman, and W. Rahmalia, "Reducing Free Fatty Acids in Crude Palm Oil Using Diethylene Glycol and Zinc(II) Chloride Based Deep Eutectic Solvent," *Indonesian Journal of Chemistry*, vol. 24, no. 3, pp. 691–700, 2024, doi: 10.22146/ijc.85980.

[26] A. Selemani and G. G. Kombe, "Glycerolysis of high free fatty acid oil by heterogeneous catalyst for biodiesel production," *Results in Engineering*, vol. 16, Dec. 2022, doi: 10.1016/j.rineng.2022.100602.

[27] R. D. Kusumaningtyas, H. Prasetiawan, N. D. Anggraeni, E. D. N. Anisa, and D. Hartanto, "Conversion of Free Fatty Acid in Calophyllum inophyllum Oil to Fatty Acid Ester as Precursor of Bio-Based Epoxy Plasticizer via SnCl2–Catalyzed Esterification," *Polymers (Basel).*, vol. 15, no. 1, Jan. 2023, doi: 10.3390/polym15010123.

[28] M. Supeno, J. P. Sihotang, Y. V. Panjaitan, D. S. Y. Damanik, J. B. Tarigan, and E. K. Sitepu, "Room temperature esterification of high-free fatty acid feedstock into biodiesel," *RSC Adv.*, vol. 13, no. 47, pp. 33107–33113, Nov. 2023, doi: 10.1039/d3ra06912e.

[29] Darwin, M. Thifal, M. Alwi, Z. Murizal, A. Pratama, and M. Rizal, "The synthesis of biodiesel from palm oil and waste cooking oil via electrolysis by various electrodes," *Case Studies in Chemical and Environmental Engineering*, vol. 8, no. July, p. 100512, 2023, doi: 10.1016/j.cscee.2023.100512.

[30] F. N. Rahma and A. Hidayat, "Biodiesel Production from Free Fatty Acid using ZrO2/Bagasse Fly Ash Catalyst," *International Journal of Technology*, vol. 14, no. 1, pp. 206–218, 2023, doi: 10.14716/ijtech.v14i1.4873.

[31] S. R. Putri Primandari, A. Arafat, and H. Veny, "Optimization of Waste Cooking Oil's FFA as Biodiesel Feedstock," *Teknomekanik*, vol. 4, no. 1, pp. 14–21, May 2021, doi: 10.24036/teknomekanik.v4i1.9072.

[32] B. Uçar, Z. Gholami, K. Svobodová, I. Hradecká, and V. Hönig, "A Comprehensive Study for Determination of Free Fatty Acids in Selected Biological Materials: A Review," Jun. 01, 2024, *Multidisciplinary Digital Publishing Institute (MDPI)*. doi: 10.3390/foods13121891.

[33] W. Z. Ng *et al.*, "Unveiling the role of mechanical process intensifications and chemical additives in boosting lipase-catalyzed hydrolysis of vegetable oil for fatty acid production: A comprehensive review," Jan. 01, 2025, *Elsevier B.V.* doi: 10.1016/j.ijbiomac.2024.138144.

[34] F. Salsabillah Maulidinoor and R. T. Wisnu Broto, "Effect of Water Content on Free Fatty Acid Value Reduction in Nyamplung Crude Oil (Calophyllum Inophyllum L.) Extracted by N-Hexane Solvent and Using Factorial Design Experiment," *Waste Technology*, vol. 11, no. 2, pp. 97–101, 2023, doi: 10.14710/wastech.11.2.97-101.

[35] H. H. Naseef and R. H. Tulaimat, "Transesterification and esterification for biodiesel production: A comprehensive review of catalysts and palm oil feedstocks," Apr. 01, 2025, *Elsevier Ltd*. doi: 10.1016/j.ecmx.2025.100931.

[36] V. S. Shanthini, D. Chitra, and G. Moorthy, "Biodiesel: A comprehensive review of properties, catalyst types, and feedstock sources," Nov. 01, 2025, *Elsevier B.V.* doi: 10.1016/j.rechem.2025.102678.

[37] M. Yaghi, S. Chidiac, S. Awad, Y. El Rayess, and N. Zgheib, "An Overview of Biodiesel Production via Heterogeneous Catalysts: Synthesis, Current Advances, and Challenges," Sep. 01, 2025, *Multidisciplinary Digital Publishing Institute (MDPI)*. doi: 10.3390/cleantechnol7030062.

[38] M. H. Abdullah *et al.*, "Optimization of Esterification and Transesterification Process for Biodiesel Production fom Used Cooking Oil," *Journal of Research and Technology*, vol. 7, no. 2, pp. 207–216, 2021.

[39] F. N. Roza, S. R. Widyaningrum, L. R. Alvita, and A. S. Rezki, "Study Of Reaction Temperature Effects On The Yield And Quality Of Biodiesel Synthesis From Waste Palm Oil," *Indonesian Journal of Pure and Applied Chemistry*, vol. 8, no. 2, pp. 45–53, Aug. 2025, doi: 10.26418/indonesian.v8i2.96780.

[40] K. Biodiesel Yang Dibuat Dari Minyak Jelantah Menggunakan Katalis Abu Sabut Kelapa and D. Eka Pratiwi, "Biodiesel characterization made of recycled frying oil with coconut fiber (C. nucifera L.) catalyst," 2024.

[41] T. M. L. Thein, V. K. Jindal, R. Jindal, and N. Yoswathana, "Acid-catalyzed esterification pretreatment of high free fatty acid crude rice bran oil for biodiesel production," *Environ. Nat. Resour. J.*, vol. 17, no. 3, pp. 68–79, Jul. 2019, doi: 10.32526/ennrj.17.3.2019.24.

[42] G. Firmansyah Putra and M. Hasan Abdullah, "Prosiding Seminar Nasional & Call for Paper," *Inovasi Inklusif Gender dalam Sociopreneurship*, vol. 12, no. 1, 2025.

[43] S. M. M. Hasnain *et al.*, "Performance, Emission, and Spectroscopic Analysis of Diesel Engine Fuelled with Ternary Biofuel Blends," *Sustainability (Switzerland)*, vol. 15, no. 9, May 2023, doi: 10.3390/su15097415.

[44] M. Shahidul Islam, C. Robin Hart, and D. Casadonte, "Ultrasound-assisted solid Lewis acid-catalyzed transesterification of Lesquerella fendleri oil for biodiesel synthesis," *Ultrason. Sonochem.*, vol. 88, Aug. 2022, doi: 10.1016/j.ultsonch.2022.106082.

[45] A. Mulula, T. N. Manoka, E. B. Bayindu, and A. D. Bouzina, "Fourier Transform Infrared (FTIR) Analysis of Fatty Acid Methyl Ester from Congolese Non-Edible Afzelia bella Seeds Oil," *Asian Journal of Applied Chemistry Research*, pp. 15–24, Sep. 2022, doi: 10.9734/ajacr/2022/v11i430262.

[46] N. Saifuddin and H. Refal, "Spectroscopic analysis of structural transformation in biodiesel degradation," *Research Journal of Applied Sciences, Engineering and Technology*, vol. 8, no. 9, pp. 1149–1159, 2014, doi: 10.19026/rjaset.8.1079.

[47] N. Khan *et al.*, "Locally Sustainable Biodiesel Production from Waste Cooking Oil and Grease Using a Deep Eutectic Solvent: Characterization, Thermal Properties, and Blend Performance," *ACS Omega*, vol. 6, no. 13, pp. 9204–9212, Apr. 2021, doi: 10.1021/acsomega.1c00556.

[48] Z. Ginting, R. Mulyawan, M. Meriatna, T. Tirani, A. Asnadia, and L. M. A. Haryono, "Characteristic Study of Biodiesel from Used Cooking Oil using Nipah Skin Ash as a Heterogeneous Catalyst," *Indonesian Journal of Fundamental and Applied Chemistry*, vol. 8, no. 1, pp. 34–39, Feb. 2023, doi: 10.24845/ijfac.v8.i1.34.

[49] W. Muderawan, N. Ketut, and P. Daiwataningsih, *Pembuatan Biodiesel Dari Minyak Nyamplung (Calophyllum Inophylum L.) Dan Analisis Metil Esternya Dengan GC-MS*. 2016.

[50] A. Santoso *et al.*, "Synthesis of methyl esters from palm oil, candlenut oil, and sunflower seed oil and their corrosion phenomena on iron nail," *AIMS Mater. Sci.*, vol. 9, no. 5, pp. 719–732, 2022, doi: 10.3934/matersci.2022044.

[51] Monika, S. Banga, and V. V. Pathak, "Biodiesel production from waste cooking oil: A comprehensive review on the application of heterogenous catalysts," *Energy Nexus*, vol. 10, no. March, p. 100209, 2023, doi: 10.1016/j.nexus.2023.100209.

[52] K. A. V. Miyuranga, U. S. P. R. Arachchige, D. Thilakarathne, R. A. Jayasinghe, and N. A. Weerasekara, "Impact of the Chemical Composition of Oil for Biodiesel Production to Reduce Environmental Pollution," *Nature Environment and Pollution Technology*, vol. 21, no. 4, pp. 1681–1687, Dec. 2022, doi: 10.46488/NEPT.2022.v21i04.021.

[53] J. H. F. Boog, E. L. C. Silveira, L. B. De Caland, and M. Tubino, "Determining the residual alcohol in biodiesel through its flash point," *Fuel*, vol. 90, no. 2, pp. 905–907, Feb. 2011, doi: 10.1016/j.fuel.2010.10.020.

[54] E. G. O. Lechuga, M. R. Zúñiga, and K. A. Niño, "Efficiency evaluation on the influence of washing methods for biodiesel produced from high free fatty acid waste vegetable oils through selected quality parameters," *Energies (Basel).*, vol. 13, no. 23, Dec. 2020, doi: 10.3390/en13236328.