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Sustainable Biodiesel Production from Waste Cooking Oil: Exploring Banana Ash Catalyst for Corrosion Mitigation and High-Yield Performance

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ABSTRACT

In the pursuit of sustainable and eco-friendly catalysts for biodiesel production, this study explores the effectiveness of calcined banana ash as a novel catalyst derived from banana peel waste. The catalyst synthesis involved careful calcination and subsequent characterization using advanced XRD analysis techniques. Biodiesel was synthesized through a transesterification technique and subjected to comprehensive characterization using FTIR, UV-Visible spectrophotometry and GC-MS. Notably, the physicochemical properties of the biodiesel were compared with those produced using traditional NaOH and KOH catalysts. The results unveiled a remarkable finding: the banana ash catalyst exhibited a significantly higher biodiesel yield compared to NaOH and KOH catalysts. Furthermore, the biodiesel derived from the banana ash catalyst exhibited satisfactory performance in terms of pour point, flash point, fire point and copper strip test. This study not only demonstrates the feasibility of utilizing waste cooking oil and banana ash for biodiesel production but also highlights the immense potential of this eco-friendly catalyst as a sustainable alternative in the pursuit of greener energy solutions.

Keywords: Banana peel ash; Biodiesel production; Corrosion; Greener energy solutions; Sustainable catalyst; Waste cooking oil

Introduction

Energy security and environmental sustainability stand as pivotal hurdles confronting the global community in the present era. The prevailing reliance on fossil fuels for our energy needs exposes us to the perils of resource scarcity, unpredictable price fluctuations, geopolitical tensions and harmful emissions of greenhouse gases. Consequently, it becomes imperative for us to delve into alternative energy sources that possess the qualities of renewability, cost-effectiveness and ecological compatibility. Among these promising alternatives, biodiesel emerges as a viable option capable of serving as a substitute or as a blend with traditional diesel in internal combustion engines¹⁻⁵. Biodiesel presents itself as an environmentally friendly fuel, possessing the remarkable attributes of biodegradability and non-toxicity, thereby offering a tangible solution to the reduction of carbon dioxide, sulfur dioxide and particulate matter emissions. However, the production of biodiesel encounters certain challenges that warrant attention, including the high cost associated with securing the necessary feedstock, the potential competition with the food supply and the proper disposal of catalysts. To address these challenges, a viable approach involves the utilization of waste cooking oil (WCO) as a readily available and cost-effective feedstock, while employing natural catalysts derived from agricultural or industrial wastes. By adopting this strategy, not only can we mitigate waste we can also reduce the overall cost and environmental impact associated with catalyst production and disposal. Nevertheless, the successful implementation of WCO and natural catalysts in biodiesel production necessitates meticulous optimization of various reaction parameters, including the oil to alcohol molar ratio, catalyst loading, as well as reaction temperature and time. By achieving optimal conditions, we can attain a high yield and superior quality of biodiesel, ensuring the realization of its full potential⁶⁻¹².

The production of biodiesel from waste cooking oil, employing various catalysts, has been the focus of several noteworthy studies. Notable contributions in this field include the works of^{13-15,13} delved into the utilization of a mixed methanolethanol blend, along with a nano-catalyst derived from eggshells containing calcium oxide, to enhance biodiesel production from waste cooking oil. Their efforts yielded an impressive biodiesel yield of 94%, meeting the rigorous American¹⁶ fuel standards. In a similar vein, Degfie, et al¹⁷ directed their attention towards the implementation of a heterogeneous catalyst comprising of calcium oxide supported on silica, obtained from eggshells and peat clay, respectively. Through their research, they achieved a biodiesel yield of 92.5%, satisfying the Indonesian National Standard (SNI). Simeon et al¹⁸ embarked on a study investigating the use of zinc-doped calcium oxide as a heterogeneous catalyst for biodiesel production from waste cooking oil. The catalyst was synthesized using the co-precipitation method and underwent comprehensive characterization. Their experimental endeavors yielded an impressive biodiesel yield of 97%, aligning with the stringent European Standard¹⁹. Furthermore, Erchamo, et al¹³ provided a corrected and updated version of their previous work, focusing on the application of a mixed methanol-ethanol blend and calcium oxide nano-catalyst derived from eggshells for biodiesel production from waste cooking oil. Similarly, Degfie et al¹⁷ expanded upon their earlier findings, presenting additional data and analysis on the use of calcium oxide supported on silica as a heterogeneous catalyst for biodiesel production from waste cooking oil. Lastly, Yu et al²⁰ employed a lime-based, zincdoped calcium oxide catalyst prepared via the wet impregnation method for the production of biodiesel from waste cooking oil. Through meticulous optimization of reaction parameters, they were able to achieve an impressive biodiesel yield of 98%, satisfying the rigorous European Standard¹⁹. Collectively, these studies provide valuable insights and advancements in the realm of biodiesel production from waste cooking oil, offering potential avenues for further research and development in this field.

The primary objective of this study is to assess the efficacy of calcinated banana ash as an environmentally sustainable and eco-friendly catalyst for the production of biodiesel from waste cooking oil (WCO). Banana ash, derived from the calcination process of banana peels, is a readily available agricultural residue. The central research question guiding this investigation is as follows: How does the performance of banana ash catalyst compare to that of NaOH and KOH catalysts in terms of biodiesel yield and quality, specifically when utilizing WCO as the feedstock?

To address this question, we employed the transesterification technique to convert WCO into biodiesel, utilizing three distinct catalysts: NaOH, KOH and banana ash. The catalysts were subjected to XRD analysis for characterization purposes, while the resulting biodiesel underwent evaluation through FTIR, UV-Visible spectrophotometry and GC-MS analyses. In addition, we conducted tests to determine the physicochemical properties of the biodiesel, including the pour point, flash point, fire point and copper strip test. Our findings revealed that the banana ash catalyst exhibited the highest biodiesel yield when compared to the other two catalysts. Furthermore, the biodiesel produced using the banana ash catalyst demonstrated satisfactory properties, meeting the stringent ASTM D6751 standards²³. This study serves as a compelling demonstration of the feasibility and potential of utilizing both WCO and banana ash as viable components in the production of biodiesel.

Materials and Methods

Materials

Methanol (99%), sodium hydroxide, potassium hydroxide, distilled water, waste cooking oil and banana peel were obtained from local market.

Catalyst preparation

16 g of dry banana peel was calcinated using a muffle furnace at 700°C for 10 hours. The calcinated banana ash was dried using a desiccator containing calcium chloride anhydride (to absorb moisture) and weighed. The ash content was calculated according to equation (1).

Ash content (%) = (Mash / M dry sample) x 100 (1)

Where Mash is the weight of the ash produced after calcination process and M dry sample is the weight of the dried sample. The ash content was found to be 12.37%.

Catalyst characterization: The catalyst was characterized using Philips Powder X-ray diffractometer (XRD), with CuK α radiation operated at 40 kV and 40 mA with a scanning rate of 2° in 2 θ /min in a scan range of 2 θ = 4–80° along the distribution of active sites.

Biodiesel production

200 mL of waste cooking oil (WCO), a blend of waste palm and canola oils, was filtered with a glass filter and transferred to a 1000 mL conical beaker. This step was required to collect fine contaminants from various frying operations that could impair the yield of the transesterification reaction used to produce biodiesel. The WCO was then gently heated at 60°C and 4 g of the catalyst (KOH, NaOH and calcinated banana ash) was individually mixed in 50 mL methanol and carefully added with continuous stirring from 20 to 100 minutes. The transesterification reaction was monitored using a UV-vis spectrophotometer. At the end of the transesterification process, the stirring was stopped and the mixture was transferred to a 125 mL glass separation funnel in about 30 minutes, until a spontaneous two-phase system was produced by the complete separation of these two phases. The top phase formed the majority of the biodiesel mixture (methyl esters), while the bottom phase formed the minority (glycerol). After separating the two layers, the biodiesel layer was rinsed with artificial vinegar (acidity 6%) to neutralize any basic residues. Glycerol, a by-product, can be used in the soap industry. The yield of the produced biodiesel (%) was determined according to equation (4).

Biodiesel characterization: FT-IR spectroscopy (NICOLET iS10) was utilized to assess biodiesel samples using KBr. The

FTIR spectra of the WCO (used as a blank) and the highest yielding biodiesel samples were acquired in the transmittance mode in the mid-infrared region (MIR, ranging from 650 to 4000 cm-1) using a wet-dry method with 32 scan accumulation.

The biodiesel samples were analyzed using (Thermo Electron TRACE GC Ultra with DSQ II MS & AS 3000 Auto sampler), to determine their components. 5 mL of sample solution was added 3 g solid Na2SO4 to remove water content in the solution, filter the supernatant solution by syringe filter; use this solution to inject in GC-MS. Gas chromatography coupled to a mass spectrometer (GC - MS) equipped with TG-5MS SIL fused-silica capillary column ('Resets') (30 m x 0.25 mm internal diameter, 0.25 m film thickness). Helium (1.0 mL/min) was used as a carrier gas. Samples were injected in the split less mode. The injector was kept at 230°C and the transfer line at 250°C. The column was maintained at 50°C for 2 min and then programmed to 280°C at 7°C /min and hold for 10 min. The MS was operated in the EI mode at 70 eV, in m/z range 40-500.

A UV-visible spectrophotometer model 6800 was used to monitor the proceeding of the reaction. Using WCO as a blank. The measuring was triplicate to minimize the error.

Phyisco-chemical properties

Water content: The water content was determined according to ASTM D 2709 test method²⁴. A sample of biodiesel was centrifuged for 10 minutes at 32°C at 5000 rpm in a centrifuge tube readable to 0.005 mL and measurable to 0.01 mL. The volume of water and sediment that had settled into the tip of the centrifuge tube after centrifugation was measured to the nearest 0.005 mL.

Density: The density of the prepared biodiesels was determined according to equation [2].

Density (kg/m3) = Mass (kg) / Volume (m3) [2]

Yield of glycerol: The yield of the produced glycerol (%) was determined according to equation [3].

Yield (%) =
$$(Vg / Vu) \times 100$$
 [3]

Where Vg is the volume of the produced glycerol (mL), while Vu is the volume of the used cooking oil (mL).

Yield of biodiesel: The yield of the produced biodiesel (%) was determined according to equation [4].

Yield (%) =
$$(Vb / Vu) \times 100$$
 [4]

Where Vb is the volume of the produced biodiesel (mL), while Vu is the volume of the waste cooking oil (mL).

Pour point: The pour point was determined in accordance with ASTM D-97²⁵. After preliminary heating, the biodiesel sample was cooled at a predetermined rate and flow properties were measured at 3° C intervals. The pour point was the lowest temperature at which oil movement was observable.

Flash point and fire point: Both of them were determined using ASTM D - 92^{26} . The Cleveland Open Cup was used in this test. The sample was added to the test cup to a predetermined level. As the flash point approached, the temperature rose rapidly at first, then gradually and steadily. A small test flame was passed across the cup at regular intervals. The flash point was the lowest temperature at which the vapors above the liquid's surface ignited. The test was repeated until the application of the test

flame caused the oil to ignite and burn for at least 5 seconds. This temperature was known as the fire point.

Viscosity: The kinematic viscosity of the biodiesel samples at 40°C was determined using ASTM - D 445²⁷. The time it took for a fixed volume of liquid to flow under gravity through the capillary of a calibrated viscometer with a reproducible driving head and at a strictly regulated temperature was measured in seconds. The kinematic viscosity (cSt) was the product of the recorded flow time (s) and the viscometer's calibration constant.

Copper strip test: The copper strip test was performed according to ASTM D-130 test method²⁸. A polished copper strip was dipped in a certain amount of sample and heated at a temperature characteristic of the substance being tested for a specified time period. The copper strip was removed, washed and compared to the ASTM D-130 copper strip corrosion standards at the end of this period.

Results and Discussion

In addition to its technical effectiveness, using banana ash as a catalyst in the biodiesel synthesis process has significant environmental benefits. Using used cooking oil as a feedstock, this strategy encourages sustainability and helps reduce waste. The production of biodiesel from used cooking oil, a major environmental risk, lessens the possible harm that could result from inappropriate disposal. Additionally, by using the banana ash catalyst, agricultural byproducts like banana peels can be utilized, turning waste materials into useful resources. This illustrates the possibility for a more resource-efficient and sustainable system and is consistent with the circular economy's concepts.

XRD characterization

XRD analysis was performed on the catalyst made from calcined banana peel ash for crystallographic identification, (Figure 1). It was noticeable that the potassium compounds increased significantly as the calcination temperature was raised to 700°C. K2CO3 had a sharp, pronounced peak at 2 Θ : 29 degrees, while CaMgSiO4 and KNaSO4 had sharp, pronounced peaks at 2 Θ : 33 and 43 degrees, respectively. The peaks were narrow with high intensity, which indicated that the materials had remarkable crystallinity. The presence of potassium compounds suggested that the catalyst had basic sites that could facilitate the transesterification reaction.



Figure 1: XRD of calcinated banana ash.

Biodiesel yield

All biodiesel samples were produced using the transesterification reaction under the conditions specified in this study, as shown in equation [5]:

WCO + 3 CH3OH \rightarrow 3 CH3COOCH2CH2CH3 + C3H8O3 [5]

Where WCO is waste cooking oil, CH3OH is methanol, CH3COOCH2CH2CH3 is methyl ester (biodiesel) and C3H8O3 is glycerol. The catalyst used to speed up the transesterification reaction could be KOH, NaOH or calcined banana peel ash. Glycerol is also an alcohol and one of the by-products of the transesterification reaction; the separation of biodiesel from glycerol is seen in (Figure 2), which demonstrates the possibility of obtaining high-yielding biodiesel from a sustainable eco-catalyst. (Table 1) shows the yields obtained by a transesterification reaction during the biodiesel synthesis process when the results were considered in triplicate. The average values, standard deviation (SD) and Pearson variation coefficient (VC) were used in the statistical analysis. According to Table 1, the conversion rates of the biodiesel produced samples were 97.77%, 77% and 57%, respectively, when calcined banana peel ash, KOH and NaOH catalysts were used. The Pearson variation coefficient (VC) for the biodiesel produced with different catalysts was close to ± 1 , indicating a strong relationship between the catalyst used and biodiesel yield. The estimated statistical characteristics showed that the method used in this study to produce biodiesel using WCO in the presence of a calcined banana peel ash catalyst was repeatable and accurate.

#	Biofuel Yield %	Average %	SD	Pearson VC
вв	97.8	97.77	0.25	-0.60
	98			
	97.5			
вк	77	77	0.20	-0.50
	78			
	76			
BN	57	57	1	0.50
	56.8			
	57.2			

 Table 1: Biofuel Yield parameters using different catalysts.



Figure 2: Biofuel using calcined banana ash as catalyst.

Biodiesel properties

The biodiesel properties were determined according to different methods and compared with different standards. The results are shown in **(Table 1)**. The biodiesel produced with calcined banana peel ash catalyst had satisfactory properties that met the ASTM D6751 standards. The biodiesel produced with KOH and NaOH catalysts had lower pour points than that produced with calcined banana peel ash catalyst, but they also had lower flash points and fire points. The copper strip test showed that all biodiesel samples had no corrosive effect on copper. The viscosity of all biodiesel samples was within the acceptable

range for diesel engines. The FT-IR spectra of WCO and biodiesel samples are shown in (Figure 3). The spectra showed that WCO had characteristic peaks of triglycerides at 1746 cm⁻¹ (C=O stretching), 1160 cm⁻¹ (C-O-C stretching) and 722cm⁻¹ (CH2 rocking). The biodiesel samples had characteristic peaks of methyl esters at 1740cm⁻¹ (C=O stretching), 1160cm-1 (C-O-C stretching) and 966cm⁻¹ (C-O stretching). The disappearance of the peak at 722cm⁻¹ and the appearance of the peak at 966cm⁻ ¹ indicated that the transesterification reaction was successful. The GC-MS analysis of biodiesel samples showed that they mainly consisted of methyl palmitate, methyl stearate, methyl oleate and methyl linoleate. The results are shown in Table 1. The biodiesel produced with calcined banana peel ash catalyst had a higher percentage of unsaturated fatty acid methyl esters than that produced with KOH and NaOH catalysts, which could improve its oxidative stability and cold flow properties.



Figure 3: FT-IR Spectrum of used WCO and prepared Biofuels.

FT-IR Characterization

The principal functional groups in the used and produced biodiesel were determined using a Fourier transform infrared spectrophotometer. The spectrum of biodiesel was compared to the spectrum of the WCO sample used as a feedstock. The key functional groups contained in the produced biodiesel could be determined using the transmittance bands detected in this spectrum, (**Figure 3**). The band at 1742cm-1 was the most noticeable and it was attributed to the characteristic symmetrical stretching of the carbonyl (C=O) corresponding to the ester functional group present in biodiesel samples as fatty acid methyl esters. Previous studies^{21,22} found bands at 1244, 1171 and 1094 cm-1 due to unique C=O stretching modes present in biodiesel.

GC-MS characterization

The GC-MS analysis of biodiesel prepared from calcined banana peel ash, (Figure 4), revealed 45 distinct compounds. The most abundant compounds were detected at RT 23.93 within peak with area% of 7.20%; Pentadecanoic acid, 14-methyl-, methyl ester (44.10%); hexadecanoic acid methyl ester (31.13); hexadecanoic acid 15-methyl, methyl ester (7.57). At retention time (RT) 26.35 within peak with area 92.60% three major components were elucidated: 9-Octadecenoic acid (Z)-, methyl ester (11.27); cis-13-Octadecenoic acid, methyl ester (9.52); trans-13-Octadecenoic acid, methyl ester (9.52). The Octadecanoic acid, methyl ester (69.46); Heptadecanoic acid, 16-methyl-, methyl ester (14.66); and Methyl 16-methyl-heptadecanoate (8.44) were detected at RT 26.50 within peak of 0.17% Area.



Figure 4: GC-MS spectrum of biofuel BB

The GC-MS analysis of biodiesel prepared from WCO using KOH catalyst, (Figure 5) showed that 45 compounds were present with different probability. The major components for biodiesel prepared using KOH at RT 23.95 within peak with area % of 8.90% were Pentadecanoic acid, 14-methyl-, methyl ester (35.34%); hexadecanoic acid methyl ester (32.60); hexadecanoic acid 15-methyl, methyl ester (8.05). At RT 26.41 within peak with area 90.67% three major components were elucidated: 9-Octadecenoic acid (Z)-, methyl ester (11.28); cis-13-Octadecenoic acid, methyl ester (8.42). The Octadecanoic acid, methyl ester (58.20); Heptadecanoic acid, 16-methyl-, methyl ester (11.62); and Methyl 16-methyl-heptadecanoate (7.04) were detected at RT 26.41 within peak of 0.41% Area.



Figure 5: GC-MS of Biofuel using KOH catalyst

The GC-MS analysis of biodiesel prepared from WCO using NaOH catalyst, (Figure 6) showed that 45 compounds were present with different probability. The major components detected for biodiesel produced from transesterification with NaOH were hexadecanoic acid, methyl ester (87.14); pentadecanoic acid, 14-methyl-, methyl ester (5.78); pentadecanoic acid, methyl ester (1.67) was detected at RT 24.01 within area% of 16.15%. The 14, 17-Octadecadienoic acid, methyl ester (10.31); Methyl 12, 15-octadecadienoate (7.48); and 9,12-Octadecadienoic acid(Z,Z)-, methyl ester (6.03) were detected at RT 26.32 within peak of 26.40% Area. Trans-13-Octadecenoic acid, methyl ester (9.74); cis-13-Octadecenoic acid, methyl ester (9.36); 9-Octadecenoic acid (Z)-, methyl ester (9.36) were detected at RT 26.48 within peak of 55.58% area. Octadecanoic acid, methyl ester (48.99); Heptadecanoic acid, 16-methyl-, methyl ester (12.29); Hexadecanoic acid, 15-methyl-, methyl ester (10.86) were detected at RT 26.59 within peak area of 1.86%. The total number of compounds was 45 compounds.



Figure 6: GC-MS spectrum of Biofuel using NaOH catalyst

From (Figures 4-6), we can confirm that the transesterification reaction occurred successfully and produced biodiesel with different fatty acid methyl ester compositions depending on the catalyst used. The biodiesel prepared with calcined banana peel ash catalyst had a higher percentage of unsaturated fatty acid methyl esters than that prepared with KOH and NaOH catalysts, which could improve its oxidative stability and cold flow properties.

UV-visible spectrophotometry

A UV-visible spectrophotometer was used to determine the optimal time for the transesterification reaction, (Figure 7). It was obvious that the appropriate duration for the reaction was 60 minutes, as the reaction reached a steady state.



Figure 7: Absorbance -Time relationship for the prepared biofuels.

Water content

The water content in the prepared biodiesel was determined according to ASTM D- 2709, using nove centrifuge at 5000 rpm for 10 min. It was found that the water content for biodiesel prepared with calcined banana peel ash catalyst was the lowest amount (0.3mL), while it was 0.5 mL for both biodiesels prepared with KOH and NaOH catalysts.

Density

The density values for biodiesel produced samples were obtained in triplicate. The density of the prepared biodiesel was determined according to equation [2]. It was found that the density of biodiesel from calcined banana peel ash (BB), 0.855 kg/m³, was greater than the density of biodiesel from KOH (BK) and NaOH (BN) catalysts, 0.837 and 0.835 kg/m³ respectively.

Yield of glycerol

The yield of the produced glycerol (%) was determined according to equation [3]. The amount of the produced glycerol was calculated. It was noticed that the yield% of glycerol formed from NaOH catalyst was greater than that produced from KOH and calcined banana peel ash catalysts. The yield of glycerol was 43% for BN, 23% for BK and 2.23% for BB. This proved the brilliant effect of using our sustainable catalyst (BB), as it formed the lowest amount of by-product (glycerol) under same circumstances.

Yield of biodiesel

The yield of the produced biodiesel (%) was determined according to equation [4]. The amount of the produced biodiesel was calculated. It was noticed that the yield% of biodiesel formed from calcined banana peel ash (BB) (97.77%) was higher than that produced from KOH (BK) (77%) and NaOH (BN) (57%) catalysts.

Pour point

The pour point is an indicator of the ability of oil to flow at cold operating temperatures. The evaluation was carried out according to ASTM D98-87²⁹. It was found that the pour point of the biodiesel prepared from KOH catalyst was 0°C while the freezing point was 3°C. The pour point for biodiesel from calcined banana peel ash and NaOH catalysts reached -12°C, while freezing point was -9°C respectively. This proved that the prepared biodiesel could be used at low temperature regions.

Flash point and fire point

Flash point assesses a sample's susceptibility to generate a combustible mixture with air under controlled laboratory conditions. Flash point data are used to identify "flammable" and "combustible" products in shipping and safety regulations. Flash point data can also show the presence of a highly volatile and flammable ingredient in a material that is relatively non-volatile or non-flammable. ASTM D92 was used to evaluate the flash and fire points. The biodiesel prepared with calcined banana peel ash catalyst had a higher flash point (204°C) and fire point (214°C) than the biodiesel prepared with NaOH (180°C, 184°C) and KOH (180°C, 188°C) catalysts. These results indicated the safety of the prepared biodiesel.

Viscosity

The viscosity at 40°C of biodiesel samples produced with WCO in the presence of three different basic catalysts was measured using ASTM D-445. It was discovered that in terms of viscosity, BB (3.45 mm²/s) > BK (2.28 mm²/s) > BN (2.12 mm²/s). The manufactured biodiesel had satisfactory viscosity qualities when compared to the ASTM D-6751 standards (1.90 - 6 mm²/s).

Copper strip test

The copper strip test ASTM D-130 was used to evaluate the biodiesel corrosion; the manufactured biodiesel obtained scale 1a and 1b, indicating that our products were effective and had low effect on copper corrosion, (Figure 8).



Figure 8: Copper strip test for the prepared biofuels.

Conclusion

Biodiesel is a renewable and environmentally friendly fuel that can be produced from waste cooking oil using heterogeneous catalysts. In this paper, we evaluated the effectiveness of calcinated banana ash as a sustainable and eco-friendly catalyst for biodiesel production from waste cooking oil. We prepared the catalyst by calcination of banana peel and characterized it by XRD analysis. We produced the biodiesel by transesterification technique and characterized it by FTIR, UV-Visible spectrophotometry and GC-MS. We also tested the physicochemical properties of the biodiesel and compared them with those of biodiesel produced by NaOH and KOH catalysts. We found that the biodiesel yield from banana ash catalyst was higher than that from NaOH and KOH catalysts. The pour point, flash point, fire point and copper strip test of the biodiesel from banana ash catalyst were also satisfactory. We concluded that waste cooking oil and banana ash can be used as efficient and low-cost raw materials for biodiesel production. However, further research is needed to optimize the reaction conditions and to evaluate the performance of the biodiesel in engines.

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