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Optimization of Biodiesel Synthesis from Kapok Seed Oil (*Ceiba pentandra*) through Transesterification Reaction with a TiO_2 Catalyst

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Abstract: Biodiesel represents a renewable, environmentally friendly, and locally producible alternative to conventional diesel fuel. This research synthesized biodiesel from kapok seed oil (*Ceiba pentandra*) via transesterification using a TiO_2 catalyst, with process parameters optimized to maximize yield. Kapok seed oil was extracted using Soxhlet extraction with n-hexane and purified by vacuum column chromatography. The transesterification process involved systematic variation of the oil-to-methanol molar ratio, catalyst mass, reaction temperature, and reaction time. Optimal conditions were established at a 1:10 molar ratio, 0.20 g of catalyst mass, a reaction temperature of 60 °C, and a reaction time of 90 minutes, resulting in a biodiesel yield of 71.58%. Characterization of the biodiesel revealed a density of 0.88 g/mL, viscosity of 3.10 cSt, acid value of 1.89 mg NaOH/g, and saponification value of 231.879 mg KOH/g. GC-MS analysis identified methyl palmitate, methyl linoleate, and methyl oleate as the principal methyl ester components, with methyl oleate as the predominant species. These findings demonstrate that biodiesel derived from kapok seed oil possesses properties suitable for use as an alternative fuel that meets established quality standards.

Keywords: Biodiesel; *Ceiba pentandra*; Process Optimization; TiO_2 Catalyst; Transesterification.

Introduction

Fossil fuels constitute the primary source of energy in Indonesia. In 2023, the National Energy Council reported that coal (40.46%), petroleum (30.18%), natural gas (16.28%), and renewable energy (13.09%) comprised the main components of the national energy mix [1]. Dependence on fossil fuels presents significant challenges, including limited availability due to their non-renewable nature and adverse environmental impacts from exhaust emissions. These emissions contain hazardous substances such as carbon monoxide, carbon dioxide, hydrocarbons, sulfur oxides, and lead [2]. Consequently, there is a critical need to develop alternative energy sources that are both environmentally sustainable and renewable.

Biodiesel represents a promising alternative energy source. It is synthesized from vegetable oil and alcohol through a transesterification reaction in the presence of a catalyst [3][4]. Compared to fossil fuels, biodiesel offers several advantages: it is environmentally friendly, biodegradable, non-toxic, possesses a high cetane number, and can be produced locally. These attributes contribute to enhanced energy independence [5]. In addition, biodiesel also provides environmental and economic benefits, including reduced exhaust emissions and particulate matter compared to fossil diesel [6], increased energy security through reduced dependence on fuel imports [7][8], and economic opportunities from utilizing local plant resources.

The use of edible oils such as palm, coconut, and soybean for biodiesel production is limited by issues of food competition and elevated costs [9]. Non-edible vegetable oils present a more viable alternative. Kapok seed oil (*Ceiba pentandra*) is particularly promising due to the abundance of kapok plants in Indonesia and the underutilization of their seeds. Kapok seeds contain 30% oil and are mainly composed of linoleic acid (33.6%), oleic acid(23.4%) and palmitic acid (22.4%), as well as malvalic acid (9.1%) and sterculic acid (2.8%) [10]. These characteristics position kapok seed oil as a strong candidate for use as a biodiesel feedstock.

Catalysts play a crucial role in the synthesis of biodiesel. Homogeneous catalysts frequently lead to issues such as soap formation and equipment corrosion, while enzyme catalysts are costly and exhibit slow reaction rates [11]. Heterogeneous catalysts are therefore preferred, as they are environmentally benign, reusable, easily separable, and contribute to cost reduction [12]. Titanium dioxide (TiO_2) is a promising heterogeneous catalyst; studies indicate that TiO_2 enhances product separation [13]. However, biodiesel yields from kapok seed oil using TiO_2 remain relatively low, approximately 51.99% [14].

These challenges underscore the necessity for further research aimed at improving biodiesel yield from kapok seed oil through optimization of the transesterification process using a TiO_2 catalyst. Optimization focuses on adjusting the oil-to-methanol molar ratio, catalyst mass, reaction temperature, and reaction time. This strategy seeks to produce biodiesel with both high yield and quality.

Research Methods

Kapok Seed Oil Extraction and Purification

Kapok fruit was obtained from North Lombok Regency, West Nusa Tenggara, using a random sampling technique in which fruits were randomly selected from trees distributed across the area. The sampled trees were identified

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using random numbers generated from a table. Seeds from selected fruit samples were then removed, sorted to remove damaged seeds, and air-dried at room temperature to achieve a low moisture content. The dried seeds were then milled and sieved to obtain a uniform particle size.

A total of 35 g of kapok seed powder was wrapped in filter paper and placed in a Soxhlet apparatus. Extraction was performed using 250 mL of n-hexane at 40°C for 6 hours. The resulting extract was separated from the solvent using a rotary evaporator at 40°C and 120 rpm to obtain solvent-free oil. The oil was weighed, and the oil content was calculated using the following formula:

$$\text{Oil yield (\%)} = \frac{m_1}{m_2} \times 100\%$$

Information:
m_1 = oil weight (g)
m_2 = sample weight (g)

The oil extract was purified using column chromatography, which separates mixture components based on their interactions with the stationary and mobile phases. Silica gel served as the polar stationary phase, while n-hexane functioned as the non-polar mobile phase. The purified fraction was concentrated using a rotary evaporator at 40°C and 120 rpm to yield pure kapok seed oil. Oil purity was assessed by thin-layer chromatography (TLC). The oil sample was applied to a silica gel plate and developed with a mixture of n-hexane and ether (8.5:1.5 v/v). After development, the plate was dried and exposed to iodine vapor to visualize the spots. The retention factor (Rf) was calculated as follows:

$$R_f = \frac{a}{b} \times 100\%$$

Information:
R_f = retention factor
a = sample spot distance (cm)
b = eluent path length (cm)

Biodiesel Synthesis Using a Transesterification Reaction

The procedure of Juniar and Rahayu (2019) was followed with minor modifications [14]. A mixture of 0.25 g TiO_2 catalyst and 6 mL methanol was stirred for 15 minutes. Pure kapok seed oil was then added, and the mixture was stirred at 65°C and 180 rpm for 120 minutes. After standing for 24 hours, two layers formed: biodiesel (upper) and glycerol (lower). The biodiesel layer was separated, washed with warm water until it was clear, and then heated at 110°C to a constant weight to obtain pure biodiesel.

Optimization of Biodiesel Synthesis from Kapok Seed Oil

Biodiesel synthesis was optimized using the method of Juniar and Rahayu (2019), with several modifications to accommodate the specific raw material [14]. Parameters optimized included the oil-to-methanol molar ratio, catalyst mass, reaction temperature, and reaction time.

Molar Ratio

The oil-to-methanol molar ratios tested were 1:8, 1:9, 1:10, and 1:11. This variation was designed to identify the most effective methanol ratio for maximizing biodiesel production through the transesterification reaction.

Catalyst Mass

The TiO_2 catalyst mass was varied at 0.15 g, 0.20 g, 0.25 g, and 0.30 g to determine the optimal catalyst amount for enhancing the reaction rate.

Reaction Temperature

Reaction temperatures of 50°C, 55°C, 60°C, and 65°C were selected based on the boiling point range of methanol and the stability of the oil. The objective was to identify the temperature that yields the highest biodiesel conversion.

Reaction Time

Reaction times of 60, 90, 120, and 150 minutes were evaluated to determine the optimal duration that achieves high conversion without causing product degradation.

Biodiesel Synthesis under Optimum Conditions

Biodiesel synthesis under optimal conditions was conducted using the parameter values identified during the optimization process. The procedure incorporated the optimal oil-to-methanol ratio, catalyst mass, reaction temperature, and reaction time as previously determined. The transesterification process followed the established methodology described earlier.

Characterization of Kapok Seed Oil and Biodiesel

Thin Layer Chromatography (TLC)

TLC analysis was conducted to qualitatively identify the presence of methyl esters in biodiesel samples. The identification was confirmed by comparing the Rf values of the sample with those of the standard reference compound.

Determination of Percent Biodiesel Yield

The percentage conversion of kapok seed oil to biodiesel was calculated using the following equation:

$$\text{Biodiesel yield (\%)} = \frac{W_b}{W_m} \times 100\%$$

Information:
W_b = weight of biodiesel (g)
W_m = weight of oil (g)

Density

An empty pycnometer is weighed (W_1). The pycnometer was then filled with kapok seed oil until full, tightly closed, and reweighed to obtain W_2. The density of the oil was calculated according to AOAC (2005) Method 920.212 using the following equation:

$$\rho = \frac{W_2 - W_1}{v\ (mL)}$$

Information:
ρ = density (g/mL)
W_1 = empty pycnometer weight (g)
W_2 = weight of pycnometer + sample (g)

Viscosity

Biodiesel viscosity testing was conducted using an Ostwald viscometer [15]. A sample of biodiesel was placed into the viscometer and sucked until it passed the upper limit line. The sample was allowed to flow freely, and the time required to pass from the initial to the final line was recorded. The same procedure was carried out for distilled water and the reference solution. The viscosity of biodiesel is calculated using the following equation:

$$\eta = \frac{\rho_s \times t_s}{\rho_0 \times t_0} \times \eta_0$$

Information:
η = sample viscosity (mm^2 /s)
η_0 = reference viscosity (mm^2 /s)
ρ = sample density (g/cm^3)
ρ_0 = reference density (g/cm3)
t = sample averaging time (s)
t_0 = reference averaging time (s)

Acid Number

The acid number was determined based on the SNI 7431:2015 method. 5 grams of kapok seed oil were placed in a 250 mL Erlenmeyer flask, followed by 50 mL of 96% ethanol. The mixture was refluxed for 60 minutes, cooled, and then 1 mL of phenolphthalein indicator was added. The mixture was then titrated with 0.1 N NaOH solution while shaking until a pink color formed, stable for 30 seconds. The acid number is calculated using the following equation:

$$AN = \frac{V_{NaOH} \times N_{NaOH} \times 40}{m}$$

Information:
AN = acid number (mgNaOH/g)
V = volume of NaOH (mL)
N = concentration of NaOH (N)
40 = Mr NaOH
m = sample weight (g)

Saponification

The saponification number was determined using the SNI 7431:2015 method. Two grams of oil were refluxed for 60 minutes with 25 mL of alcoholic KOH solution. After the mixture had cooled, 1 mL of phenolphthalein indicator was added. The mixture was titrated using a 0.5 N HCl solution until the pink color disappeared (stable for at least 15 seconds). The same procedure was performed for the blank without the oil sample. The saponification number is calculated using the following equation:

$$SN = \frac{(B - S) \times N \times 56.1}{m}$$

Information:
SN = saponification number (mgKOH/g)
B = volume of HCl required for blank titration (mL)
S = volume of HCl required for sample titration (mL)
N = normality of HCl solution (N)
56.1 = Mr KOH
m = sample weight (g)

GC-MS Analysis of Biodiesel

Identification of fatty acid methyl esters was performed using a Shimadzu GC-MS (Gas Chromatography-Mass Spectrometry) instrument, following the method described by Gunawan et al. (2014). Analysis used a Rastek RXi-5MS column (30 m × 0.25 mm), with an injector temperature of 290°C and a column temperature program of 40-280°C. The detector used is a Flame Ionization Detector (FID) with helium as the carrier gas.

Results and Discussion

The kapok seeds used in this study were obtained from mature, dried kapok pods, with an average seed weight percentage of 63.03%. Mature kapok seeds were selected based on their higher oil content and lower water content [16]. Analysis showed that the moisture content of kapok seeds before drying was 12.58%, decreasing to 8.85% after the drying process. High water content can reduce yield and affect oil quality because water can trigger the hydrolysis of triglycerides into free fatty acids, which causes rancidity and changes in oil color [17]. Therefore, kapok seeds were dried for 2-3 days to reduce the water content before the oil extraction process.

Kapok Seed Oil Extraction and Purification

Kapok seed oil extraction was carried out using the Soxhlet method using n-hexane as a solvent for 6 hours. This method was chosen because it is efficient, produces high yields, and allows the solvent to be reused. N-hexane is nonpolar, making it suitable for dissolving triglycerides in vegetable oils. The extracted oil is yellow, with a distinctive aroma reminiscent of kapok seeds, and an average yield of 20.03%. This value is lower than the 33.475% found in [14] study, likely due to differences in geographic conditions, sampling locations, and raw material moisture content.

Figure 1. TLC results: (a) VCO standard with n-hexane:diethyl ether (8.5:1.5 v/v) eluent; (b) kapok seed oil

Oil purification was performed using vacuum column chromatography (VCC) with silica gel as the stationary phase and n-hexane as the mobile phase. This process separates free fatty acids from triglycerides, producing a pure, clear, and yellow oil. Purity testing using thin-layer chromatography (TLC) showed an Rf value of 0.60, close to the VCO standard (0.63) (Figure 1) and within the acceptable Rf range (0.2–0.8). These results indicate that the kapok seed

oil obtained has a good level of purity and is suitable for use as a raw material for biodiesel production [18].

Biodiesel Synthesis Using Transesterification

Transesterification is the process of converting triglycerides into methyl esters with glycerol as a by product using methanol and a catalyst. The molar ratio of oil to methanol used was 1:10, with a catalyst mass of 0.25 g, a reaction temperature of 65°C, and a reaction time of 120 minutes. The yield obtained was 61.71%. This result is higher than the research by [14], who synthesized biodiesel from kapok seed oil with a yield of 51.9938%. This indicates that the results obtained were not optimal, so optimization of the molar ratio, catalyst mass, reaction temperature, and reaction time was carried out.

Optimization of Biodiesel Synthesis from Kapok Seed Oil

Molar Ratio

The molar ratio, or the ratio of oil to alcohol, is an important factor influencing the transesterification reaction [19].

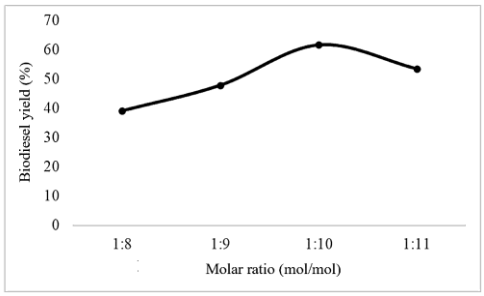

Figure 2. Effect of molar ratio on biodiesel yield, reaction conditions: 0.25 g catalyst; temperature 65˚C; time 120 minutes

The graph in Figure 2 shows that the optimum molar ratio was achieved at a ratio of oil to methanol of 1:10, resulting in a biodiesel yield of 61.71%. This result aligns with research by [20], who also obtained a molar ratio of 1:10 methanol to water. Using excess methanol can shift the equilibrium toward the product, but too much can trigger a reverse reaction, reducing the yield [21].

Catalyst Mass

The use of a catalyst can lower the activation energy in the transesterification process, allowing the reaction to proceed quickly [22].

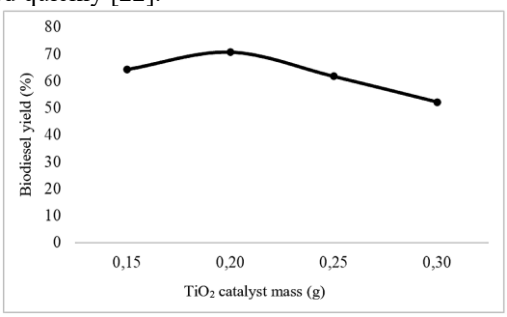

Figure 3. Effect of catalyst mass on biodiesel yield, reaction conditions: molar ratio 1:10 mol/mol; temperature 65˚C; 120 minutes.

The highest biodiesel yield of 70.70% was obtained with the addition of 0.20 g of catalyst (Figure 3). This result differs slightly from the study by [20], which found an optimal catalyst concentration of 0.25 g. Excessive catalyst use can trigger a saponification reaction between free fatty acids and base, forming soap, which can increase biodiesel viscosity and disrupt the glycerol separation process [23]. Conversely, too little catalyst results in an incomplete reaction, resulting in decreased yield [4]. Therefore, the optimum catalyst mass for achieving the highest biodiesel yield is 0.20 g.

Reaction Temperature

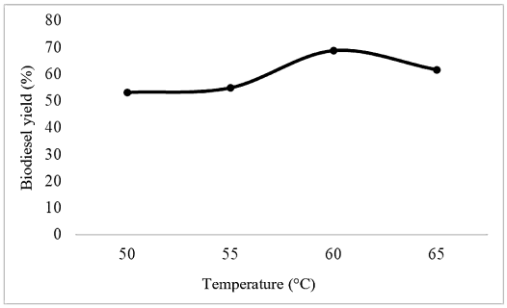

Figure 4. Effect of reaction temperature on biodiesel yield (molar ratio 1:10; catalyst 0.25 g; reaction time 120 minutes).

High reaction temperatures can accelerate the transesterification reaction, thereby increasing biodiesel yield. Based on Figure 4, the yield at 50-55°C is still low because the reaction is not yet optimal [24]. The highest yield was obtained at 60°C, at 68.82%, because the reaction has reached equilibrium. At temperatures above 65°C, the yield decreases due to a shift in equilibrium toward the reactants and an increase in free fatty acid levels due to oxidation and hydrolysis [5][25]. These results align with research by [20], which found an optimum temperature of 60°C. The reaction temperature is generally set slightly below the boiling point of methanol to prevent excessive evaporation, which can reduce solvent volume and biodiesel yield.

Reaction Time

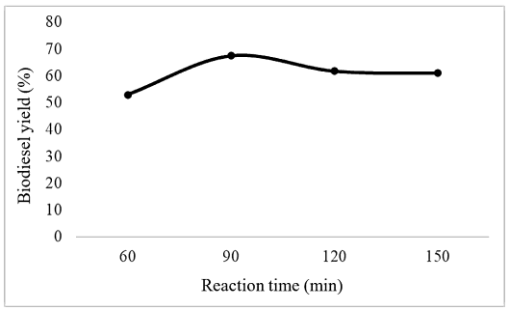

Figure 5. Effect of reaction time on biodiesel yield (molar ratio 1:10 mol/mol; catalyst 0.25 g; temperature 65°C)

Reaction time affects the rate and equilibrium of the transesterification reaction [26]. Based on Figure 5, the highest yield of 67.50% was obtained after 90 minutes, indicating that the reaction had reached equilibrium. Excessive reaction time can decrease yield due to increased formation of free fatty acids and glycerol [25]. This is in line

with research by [27][5], which states that after the optimum time, yield decreases because the reaction is reversible. Reaction time exceeding the equilibrium point does not increase conversion and can even decrease yield due to reverse reactions and component degradation.

Biodiesel Synthesis under Optimum Conditions

Biodiesel synthesis was conducted under optimal conditions to achieve the maximum yield. Optimum conditions were achieved at a molar ratio of oil to methanol of 1:10, a catalyst mass of 0.20 g, a reaction temperature of 60°C, and a reaction time of 90 minutes. This process was repeated three times, yielding an average of 71.58%. These results indicate that this combination of parameters provides the most effective conditions for the transesterification reaction. A high molar ratio increases the contact between triglycerides and methanol. The catalyst accelerates the reaction, the optimum temperature promotes methyl ester formation without excessive evaporation, and a sufficient reaction time ensures maximum conversion. This yield is higher than the pre-optimization conditions, demonstrating the effectiveness of the optimization process in increasing biodiesel production efficiency.

Characterization of Kapok Seed Oil and Biodiesel

Thin Layer Chromatography (TLC) was performed to qualitatively identify the presence of methyl esters in biodiesel samples. Methyl oleate was used as a standard because it is one of the dominant fatty acids in kapok seed oil. Separation was carried out using a mobile phase of n-hexane:diethyl ether (8.5:1.5 v/v). The analysis results showed an Rf value of 0.62 for the biodiesel and 0.63 for the standard. The closeness of these values indicates that the main compound in biodiesel is methyl esters, specifically methyl oleate.

Figure 6. TLC Results (a) biodiesel (b) methyl oleate eluent n-hexane:diethyl ether (8.5:1.5 v/v)

The formation of two spots on the TLC plate (Figure 6) indicates the presence of components other than methyl esters. The main spot aligns with the standard, while the second spot at the bottom likely represents incompletely esterified free fatty acids. This indicates that the transesterification reaction proceeded successfully, although small amounts of free fatty acids remained.

The characterization of kapok seed oil and biodiesel aimed to determine the physical and chemical properties of the materials before and after the transesterification process. According to Table 1, the density of kapok seed oil was 0.90 g/mL and decreased to 0.88 g/mL after the biodiesel conversion process. This value aligns with the SNI 7182:2015 range (0.85–0.89 g/mL), indicating that the conversion process produced biodiesel with density characteristics that meet the standards.

Table 1. Characterization Results of Kapok Seed Oil and Biodiesel

Parameter	Unit	Value		Biodiesel Standard (SNI 7182:2015)
		Oil	Biodiesel	
Density	g/mL	0.90	0.88	0.85-0.89
Viscosity	cSt	3.60	3.10	2.3-6.0
Acid Number	mg NaOH/g	3.20	1.89	0.5
Saponification Number	mg KOH/g	225.796	231.879	-

The initial oil viscosity of 3.60 cSt decreased to 3.10 cSt in the biodiesel. This decrease indicates the success of the transesterification reaction in converting triglycerides into methyl esters, which have a lower viscosity, thus improving combustion performance. This value is also within the SNI range (2.3–6.0 cSt).

The acid value decreased from 3.20 mg NaOH/g to 1.89 mg NaOH/g after the process. This decrease indicates a reduction in free fatty acid content due to the esterification and transesterification reactions. Although this value is higher than the Indonesian National Standard (SNI) standard (0.5 mg NaOH/g), this significant decrease indicates that the purification process has been quite successful.

The saponification value increased from 225.796 mg KOH/g to 231.879 mg KOH/g, indicating a change in the triglyceride structure to a lower molecular weight methyl ester.

Overall, the characteristics of the resulting biodiesel have approached or met most SNI parameters, particularly density and viscosity, making it potentially suitable for use as an alternative fuel.

GC-MS Analysis of Biodiesel

The results of GC-MS analysis (Figure 7) of biodiesel from kapok seed oil showed the presence of three main methyl ester components: methyl palmitate (1.21%), methyl linoleate (0.74%), and methyl oleate (4.30%), with retention times of 11.899, 12.397, and 12.488 minutes, respectively (Table 1). The characteristic fragment ions that appeared, such as m/z 74 (ester group) and M–32 (methanol loss), confirmed that the compounds formed were methyl esters resulting from the transesterification reaction.

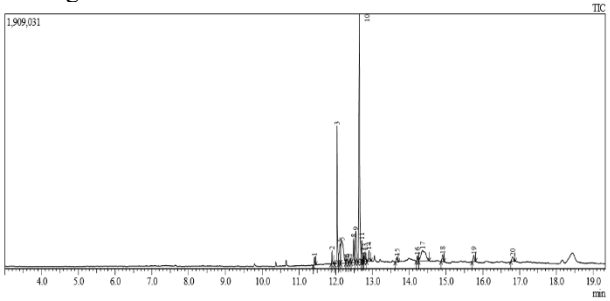

Figure 7. GC-MS chromatography results of biodiesel

Table 2. Methyl ester components in biodiesel

Methyl Ester	t_R (min) Sample	Amount (%)
Methyl Palmitate	11.899	1.21
Methyl Linoleate	12.397	0.74
Methyl Oleate	12.488	4.30

The predominance of methyl oleate indicates that biodiesel from kapok seed oil is rich in monounsaturated fatty acids, which support oxidative stability and combustion efficiency. The methyl palmitate content helps improve shelf stability and cetane value, while the low methyl linoleate content reduces the risk of oxidation. This methyl ester composition reflects the good quality of biodiesel and its potential as an alternative biofuel [28][29][30].

In general, the methyl ester composition profile of biodiesel from kapok seed oil is quite good, as it is dominated by monounsaturated fatty acids, which support cold flow properties and efficient combustion, while still containing small amounts of saturated fatty acids, which contribute to oxidative stability [31][30]. This composition is similar to biodiesel from soybean oil or palm oil, so biodiesel from kapok seed oil has the potential to be used as an environmentally friendly alternative biofuel.

Conclusion

The results showed that the optimized transesterification process of kapok seed oil using TiO_2 catalyst increased biodiesel yield from 61.71% to 71.58%. The resulting biodiesel met the density and viscosity parameters according to SNI 7182:2015; however, the acid content still exceeded the standard, necessitating further purification. GC-MS analysis identified methyl palmitate, methyl linoleate, and methyl oleate as the dominant components, with methyl oleate as the primary component. This composition supports the good oxidative properties and combustion performance of biodiesel, making kapok seed oil a potential non-edible feedstock for renewable energy.

Author's Contribution

Mona Rozitawati: Conducted the extraction and purification of kapok seed oil, performed biodiesel synthesis and optimization, and carried out the characterization of kapok seed oil and biodiesel. Erin Ryantin Gunawan: Conceptualized the research, developed the methodology, and supervised the entire experimental process. Dedy Suhendra: Analyzed and interpreted the experimental data. Farida Ariani: Wrote and edited the results and discussion sections, and compiled tables, graphs, and conclusions..

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