

Biodiesel Production from the Transesterification of Rubber Seed Oil

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Abstract- Over the past decades, researchers have been investigating alternative feedstock to edible oils which contributes up to 80 % production cost limiting the competitiveness of biodiesel with the conventional petroleum diesel. Rubber seeds are wastes processed from rubber production and are usually disposed of indiscriminately in dumpsites and landfills. This study investigated the production of biodiesel from the transesterification of rubber seed oil. The operating parameters were temperature (45 °C), reaction time (75 min), catalyst loading (0.5 g), and methanol-to-oil ratio (4:1, g/g). The yield of the biodiesel obtained was 44.2 wt. %. The physicochemical properties of the biodiesel produced were density (843.2 g/m³), specific gravity (0.8622), viscosity at 40 °C (1.901), kinematic viscosity (1,601 mm²/s), acid value (1.401 mg KOH/g), free fatty acid (0.7007 %), flash point (83.1 °C), pour point (-4.3 °C), cloud point (-5.2 °C), calorific value (38.1042 MJ/kg), and cetane number (48.4). The predominant fatty acid esters in the biodiesel included methyl ester showed that the biodiesel produced contained dodecanoic acid methyl ester, tridecanoic acid methyl ester, Undecanoic acid methyl, and octanoic acid methyl ester. The quality of the biodiesel produced suggests that the fuel has the capacity to provide a high environmental performance, and engine longevity. With this, Nigeria can recover value from the rubber seed, manage waste effectively, and ensure sustainable rubber trees production.

Index Terms- Rubber seeds oil; biodiesel; oil extraction; transesterification; physicochemical properties

I. INTRODUCTION

The increasing global demand for energy, coupled with the depletion of fossil fuel reserves and the environmental consequences of greenhouse gas emissions, has intensified the search for renewable and sustainable alternatives to petroleum-based fuels. Among these alternatives, biodiesel has gained significant attention due to its biodegradability, non-toxic nature, and reduced emissions of carbon monoxide, hydrocarbons, and particulate matter

compared to conventional diesel (Demirbas, 2009; Nguyen, Huynh, & Le, 2020). Biodiesel is produced through transesterification of vegetable oils or animal fats with alcohol, typically methanol, in the presence of a catalyst, yielding fatty acid methyl esters (FAMES) and glycerol as a by-product (Knothe, 2010).

Despite the advantages of biodiesel, the commercial viability of biodiesel is constrained by the high cost of feedstock, which accounts for up to 70 - 80 % of the total production cost (Berhanu et al, 2021). Most biodiesel production depends on edible oils such as soybean, sunflower, rapeseed, and palm oil, raising concerns about food security and competition between food and fuel uses (Atabani et al., 2013). This has resulted to the research into non-edible oils as alternative feedstocks, which are often cheaper, underutilized, and do not compete directly with food supply chains. Nigeria is naturally endowed with abundant non-edible biodiesel production feedstock such as *Jatropha* (*Jatropha curcas*), rubber seed (*Heavea brasiliensis*), neem (*Azadirachta Indica*), and Castor (*Ricinus communis L.*) (Edeh, 2020). Unfortunately, the utilization of these foodstock in production of biodiesel in Nigeria has not been harnessed. In general, biodiesel production in Nigeria is still at its nascent stage with insignificant capacity in comparison to the abundant resources with Fortafric Biodiesel contributing 400 liters per day (Edeh and Israel, 2026). The reasons for the under development of biodiesel diesel in Nigeria may be due to inconsistent policy implementation, lack of investment and infrastructure, and high production cost in relation to fossil fuels (Edeh and Israel, 2026; Edeh and Okpo, 2023).

As mentioned earlier. rubber seed oil (RSO), is one of the potential non-edible feedstocks in Nigeria. The trees are cultivated primarily for latex production,

leaving their seeds largely unused despite their high oil content (Maliki & Ifijen, 2020). The annual production of the Nigeria's rubber sector is approximately 149,396 tons from estimated 361,396 hectares. The rubber trees are predominantly grown in Edo, Delta, Ondo, Abia, Anambra, Akwa-Ibom, Cross River, Rivers and Bayelsa States, respectively. Although, the rubber trees in Nigeria are aging, the country is currently revitalizing them through the adoption of high-yield, disease-resistant clones such as NIG800/900 (Adeleye, 2024).

Studies have revealed that oil yields from rubber seeds can be comparable to those of rapeseed and palm oil, and significantly higher than soybean and sunflower oils, making RSO a potential feedstock for biodiesel production (Baidoo et al., 2022; Sugebo, 2021). Furthermore, biodiesel derived from RSO has shown to possess favorable physicochemical properties such as viscosity, calorific value, and cetane number, which align with international biodiesel standards (Reshad et al., 2015a; Karmakar et al., 2022a). However, there are challenges, particularly the high free fatty acid (FFA) content of RSO, which can lead to soap formation when base catalysts are used, thereby reducing biodiesel yield. Acid catalysts are therefore preferred for transesterification of RSO to achieve higher conversion efficiency (Lakshmi et al., 2020). This study investigates biodiesel production from rubber seed oil under controlled operating conditions, analyzing yield, physicochemical properties, and fatty acid composition. The study is focused on evaluating the potential of RSO as a sustainable feedstock for biodiesel production in Nigeria, contributing to energy diversification and reducing dependence on fossil fuels.

II. MATERIALS AND METHODS

2.1 Material

Freshly harvested rubber seeds (*Hevea brasiliensis*) were collected from the Rubber Research Institute of Nigeria (RRIN), situated at Iyanomo, Edo State, Nigeria. The seeds were dried at 60 °C for 6 h before deshelling and milling using a hydraulic press machine.

The following chemicals were used H₂SO₄ (acid catalyst) with a purity of 98 %, KOH and NaOH (base catalyst) with 96.5 % purity, and methanol with a purity of 99 %, were purchased from Wintech Chemical Industry, Benin City, Nigeria. The n-hexane with 98 % purity was also used, and was purchased from Rovet Scientific (NIG) Limited, Benin City, Nigeria.

2.2 Methods

2.2.1 Extraction of rubber seed oil

This was carried out using Soxhlet apparatuses (Figure 1). A 100 g of the ground rubber seed was charged into the thimble of the apparatus. A distillation setup was then assembled, with a 3L round bottom flask containing 300 mL of the organic solvent (n-hexane) heated using a heating mantle until it reached its boiling point. The vapor produced was directed to the condenser, where it condensed and dripped down into the thimble containing the sample. This facilitated the dissolution of the soluble components of the sample by the condensed liquid. The process continued until the liquid level reached a predetermined mark. The soluble portion of the sample was then aspirated into the distillation flask, and the extraction process continued for 8 h. The yield of the rubber seed oil was obtained using Equation 1.

$$\text{Rubber see oil (RSO) yield (wt. \%)} = \frac{\text{Weight of the RSO extracted}}{\text{Weight of the ground rubber seed}} \times 100 \quad (1)$$



Figure 1: Extraction of rubber seed oil using Soxhlet apparatuses

2.2.2 Analysis of the rubber seed oil

(1). Acid value (AV) and free fatty acid (FFA)

A 2g of the RSO was transferred into a 250 mL conical flask before adding 50 mL of a mixture containing an equal volume of ethanol and toluene, and vortexed until the RSO was completely dissolved (Lestari *et al.*, 2023). 3 drops of phenolphthalein indicator were added to the resulting solution, causing it to change color from colorless to pinkish at the endpoint. Standardized sodium hydroxide solution was titrated with the solution of the equal volume mixture of ethanol and toluene. The volume of sodium hydroxide required to reach the endpoint was noted, and the acid value and the Free Fatty Acid (FFA) value were determined using Equations 1 and 2, respectively.

$$\text{Acid value (mg KOH/g)} = \frac{\text{volume of NaOH used} \times \text{normality of NaOH} \times 56.1}{\text{Weight of oil}} \quad (2)$$

$$\text{FFA value (\%)} = \frac{\text{Acid value} \times 28.2}{\text{Weight of oil}} \quad (3)$$

2.2.3 Production of biodiesel from rubber seed oil

The biodiesel was produced as prescribed by Bokhari *et al.* (2014). The rubber seed oil was charged into a three-necked round-bottom flask using a methanol-to-oil ratio of 4:1 (g/g). The flask was put in a water bath with the central neck connected to a condenser and the other two neck fixed with thermometer and mechanical stirrer, respectively. The oil was heated to 45 °C by adjusting the water bath temperature accordingly. A predetermined amount of methanol and 0.5 g of the catalyst (sulfuric acid) were added through one neck of the flask. The mixture was stirred using a magnetic stirrer at a constant speed of 200 rpm. The temperature was maintained at 45 °C for 75 min. Upon completion of the reaction, the mixture was transferred to a separating funnel where it was left to settle for 24 hr under gravity after vortexing. The process separated the mixture into two distinct layers: the upper layer containing fatty acid methyl ester and the lower layer containing glycerol as a by-product. The upper layer was then separated and washed with warm deionized water to eliminate impurities.

2.2.4 Analysis of the biodiesel obtained from the transesterification of RSO

(A). Physicochemical properties of the biodiesel

These included cetane number, pour point, flash point and acid value. The properties were analysed as shown below.

1. Cetane number: this was analyzed according to ASTM D975-08a ASTM D6751-12, EN 590:2004 and EN 14214:2012 Standards.
2. Pour point: This was analyzed according to ASTM D975-08a ASTM D6751-12, EN 590:2004 and EN 14214:2012 Standards.
3. Flash point: this was analyzed according to ASTM D975-08a ASTM D6751-12, EN 590:2004 and EN 14214:2012 Standards.
4. Acid value: this was analyzed according to ASTM D975-08a ASTM D6751-12, EN 590:2004 and EN 14214:2012 Standards.

(B). Determining the fatty acid composition of the biodiesel produced from the rubber seed oil

This was conducted using Agilent-Technologies 6890N Network GC system equipped with an Agilent-Technologies 5975 inert XL Mass selective detector and an Agilent-Technologies 7683B series auto injector. The capillary column was HP-5MS (30 m × 0.25 mm; film thickness 0.25 µm), and 1.0 µL of the sample was injected into the column with a split ratio of 100:1. The carrier gas was helium gas with a flow rate of 1.2 mL min⁻¹. The column temperature was programmed from 150 - 250 °C at a linear ramp rate of 4 °C min⁻¹. The initial and final hold-up times were 1 and 5 min, respectively. GC/MS detection was conducted in electron ionization mode with an ionization energy of 70 eV. Injector and MS transfer line temperatures were 250 °C and 260 °C, respectively. The scanning mass range was selected from 30-550 m/z (mass-to-charge ratio). The identification of unknown samples was predicated on matching of their relative retention times with those of standards. Furthermore, samples were authenticated through comparison of their mass spectra with those from the NIST mass spectral library of the GC/MS system.

III. RESULTS AND DISCUSSION

3.1 Extraction of oil from rubber seed and analysis

The oil was extracted to be used as a cheap feedstock for the production of biodiesel via transesterification. The extraction was carried out using Soxhlet apparatuses as have been described earlier. The yield obtained was 41.05 %. This is greater than 40.3 % recorded by Baidoo et al. (2022). The yield obtained is within the range of those of rapeseed (38 - 46 %) and palm (30 - 60 %), and greater than those of soybean (15 - 20 %), and sunflower (25 -35 %) which are currently been used for the commercial production of biodiesel (Edeh, 2020). This shows that rubber seed oil may be a potential commercial feedstock for biodiesel production.

Upon analysis, the physicochemical properties of the rubber seed oil included viscosity 0.422 ± 0.16 poise, specific gravity at 29 °C (0.922 ± 0.10), and melting point (28.657 ± 0.35 °C). The free fatty acid and acid values were 17.05 %, and 34.10 mgKOH/g oil. The high free fatty acid value obtained suggested that the rubber seed oil is acidic and using a base catalyst may result in low yield of biodiesel due to the formation of soap, hence, the use of an acidic catalyst is preferred. Although, the acid value is high, it is less than 34 mg KOH/g oil, and 82 mg KOH/g oil recorded by Lakshmi et al. (2020), and Jilse et al. (2016), respectively.

3.2 Production of biodiesel from rubber seed oil

The biodiesel was produced at temperature (45 °C), reaction time (75 min), catalyst loading (0.5 g), and methanol-to-oil ratio (4:1, g/g), and the yield obtained was 44.2 wt. %. This yield is lower than 86 % reported by Mahbud et al. (2011). The disparity may be due to the difference in the production methods, and operating conditions.

3.3 Analysis of the biodiesel produced from the transesterification of rubber seed oil

(A) The physicochemical properties of biodiesel

This was carried out to assess the potential of using the biodiesel from rubber seed oil as fuel. The results obtained are shown in Table 1. The result shows that the density of the diesel produced was 843.2 g/m³. Density provides information about the energy content and the compatibility with the existing fuel

infrastructure. The density of the biodiesel is within the prescribed range of 860 - 900 g/m³ specified in the ASTM standard. This shows the fuel may be easily transported and have a good performance in the engines (Hidayat et al., 2023; Wahyudi *et al.*, 2023). The biodiesel may also be compatible with the existing infrastructure within the biodiesel industry. As shown in Table 1, the kinematic viscosity of the biodiesel was 1.601 mm²/s and this was within the range of 1.3 - 2.4 mm²/s specified in the ASTM D875-08a. The kinematic viscosity affects fuel flow, atomization, and combustion. Based on this result, it is expected that the biodiesel will flow smoothly into the combustion chamber of the engine, thereby increasing the combustion efficiency and engine lubrication (Suardi *et al.*, 2023).

Table 1. The physicochemical properties of biodiesel

S/ N	Property	ASTM D975-08a	ASTM	EN 590:2004	EN 14214 :2012	Biodiesel from the current work	Biodiesel property from literature	Ref.
1.	Refractive Index					1.441		
2.	Density (g/m ³)		860-900			843.2	860 ±0.015	Karmakar et al. (2022b)
3.	Specific gravity		0.86 - 0.9 (at 40 °C)			0.8622	0.8705	Reshad et al. (2015b)
4.	Viscosity at 40 °C (mm ² /s)		1.9 - 6.0			1.901	3.82	Adam et al. (2017)
5.	Kinematic viscosity (mm ² /s)	1.3 - 2.4	1.9 - 6.0	2.0 - 4.5	3.5 - 5.0	1.601	3.89	Ahmed et al. (2014)
6.	Acid value (mg KOH/g)		0.5	0.5	0.5	1.401	0.58 ±0.01	Karmakar et al. (2022)
7.	Free fatty acid (%)					0.7007		
8.	Flash point, min (°C)	38		55	101	83.1	136 ±1.25	Karmakar et al. (2022)
9.	Pour Point (°C)			-15 to -5		-4.3	-3	Reshad et al. (2015b)
							-1.3	Adam et al. (2017)
10.	Cloud Point (°C)			-35 to -15		-5.2	3.2	Ahmed et al. (2014)
11.	Calorific value (MJ/kg)		35 min			38.1042	39.53	Reshad et al. (2015b)
12.	Cetane number		47 min			48.4	49.8 ±0.3	Karmakar et al. (2022)

The acid value of the biodiesel was 1.401 mgKOH/g and this was greater than the 0.5 ± 0.01 mg KOH/g presented by Karmakar et al. (2022b). The difference in the result may be attributed to the method used in producing the biodiesel. The acid value shows the presence of free fatty acids in the biodiesel and the current value is 0.7007 % which is greater than the prescribed 0.5 %. The high acid number suggests high acidity, and the implication of this could lead to corrosion and engine damage. From Table 1, the flash point of the biodiesel was 83.1 °C, and this is greater than 38 °C specified in the ASTM standard.

The flash point is the lowest temperature at which fuel vapour can ignite upon exposure to an open flame or spark (Kaisan *et al.*, 2020). The result shows that the biodiesel has a tendency to undergo spontaneous ignition in the presence of any spark or open flame.

The pour and cloud points were -4.3 °C and -5.2 °C, respectively (Table 1) and these values are within the pour point (-15 to -5 °C), and cloud point (-35 to -15°C), respectively, prescribed in EN 590:2004. The pour point is the lowest temperature at which fuel remains fluid and can flow freely without forming wax crystals (Yang *et al.*, 2020). The result of the pour point shows that the biodiesel will remain fluid at -4.3 °C. The cloud point shows the temperature at which dissolved particles in the biodiesel begin to crystallize, resulting to cloudy formation (Qubeissi *et al.*, 2023). This means that the biodiesel produced with a cloud point of -5.2 °C would not support crystal formation at that temperature. Since, both the pour and cloud points of the biodiesel produced are within the specification, the fuel would maintain their fluidity and clarity. This will help to ensure smooth operation and performance in extremely cold weather.

The calorific or heating value of the biodiesel was 38.10 MJ/kg. This value is higher than the minimum of 35 prescribed in the ASTM. The calorific or heating value indicates the quantity of heat released, when a unit mass of fuel is burned. The result shows that the biodiesel has the capacity to produce more heat per unit mass of the fuel.

From Table 1, the cetane number of the biodiesel was 48.4, and this value is slightly lower 49.8 ± 0.3 obtained by Karmakar *et al.* (2022b). The cetane number shows the ability of the fuel to ignite. The higher the value, the lower the time required for ignition to occur (Ahmad *et al.*, 2021). The result shows that the biodiesel has the capacity to provide smoother, quieter idling and improved starting.

(B). Determining the fatty ester composition of the biodiesel obtained from the transesterification of rubber oil

The biodiesel was produced from the transesterification of rubber seed oil at temperature

(45 °C), time (75 min), catalyst loading (0.5 g) and methanol-to-ratio (4:1, g/g), and analysed using Agilent-Technologies 6890N Network GC system equipped with an Agilent-Technologies 5975 inert XL Mass selective detector and an Agilent-Technologies 7683B series auto injector. The method had been described in section 2. The results obtained are shown in Table 2, and it shows that dodecanoic acid methyl ester, tridecanoic acid methyl ester, Undecanoic acid methyl, and octanoic acid methyl ester, are the predominant methyl ester in the biodiesel produced. The presence of these esters in the biodiesel show that the fuel contains the following fatty acids lauric acids, tridecanoic acid, hendecanoic acid and caprylic acid. This means that the biodiesel was produced from predominantly saturated fatty acids

Table 2. Fatty acid composition of the biodiesel produced from the transesterification of rubber seed oil.

S/N	Systemic Name	Trivial Name	Composition % Area
1.	Dodecanoic acid, methyl ester	Dodecanoic acid, methyl ester	98
2.	Tridecanoic acid, methyl ester	Tridecanoic acid, methyl ester	98
3..		Decanoic acid, methyl ester	97
4.	Undecanoic acid, methyl ester	Undecanoic acid, methyl ester	96
5.		Decanoic acid, methyl ester	95
6.		Undecanoic acid, methyl ester	95
7.	Mesitylene	Benzene, 1,2,3-trimethyl-	95
8..	Octanoic acid, methyl ester	Octanoic acid, methyl ester	94
9.	Benzene, propyl-	Nonane, 2-methyl-	83

IV. CONCLUSION

The study demonstrated that biodiesel can be produced via transesterification of rubber seed oil at temperature (45 oC), reaction time (75 min), catalyst loading (0.5 g), and methanol-to-oil ratio (4:1, g/g). The yield of the biodiesel obtained was 44.2 wt. %, and can be improved through optimization. The physicochemical properties such as density, kinematic viscosity, acid value, flash point, pour point, cloud point, calorific value, cetane number and fatty acid composition of the biodiesel produced, suggest that the fuel possesses a desired quality for enhanced engine performance.

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