

Optimisation of Biodiesel Production from *Cola rostrata* Seed Oil Using an Activated Plantain Peel Ash Biocatalyst for Sustainable Fuel

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Abstract: Biodiesel produced from non-edible oils with waste-derived heterogeneous catalysts can reduce competition with food crops and avoid the drawbacks of homogeneous catalysts. This study investigated biodiesel production from *Cola rostrata* seed oil (CRSO) using a KOH-activated plantain peel ash (AAPPA) catalyst. CRSO was extracted with n-hexane (50.7 wt% oil yield) and characterised by ASTM methods, GC–MS and FTIR. It had an acid value of 2.29 mg KOH/g (free fatty acid 1.145%) and a fatty acid profile dominated by unsaturated acids, mainly linoleic (42.72%) and oleic (28.18%) acids. KOH activation increased the total porosity of the ash from 18.77 to 47.74% and its BET surface area from 113.6 to 387.2 m²/g. One-factor-at-a-time experiments showed that the activated catalyst outperformed the raw ash and that yield peaked at 3 wt% catalyst, a 10:1 methanol/oil molar ratio, 60 °C, 3 hrs and 300 rpm. A 32-run central composite design (Design-Expert 13) was then used to model the five factors. The quadratic model was highly significant ($F = 100.04$, $p < 0.0001$; $R^2 = 0.995$) with a non-significant lack of fit; all linear and quadratic terms and the AB, AC, AE and CD interactions were significant, and stirring speed and methanol/oil ratio had the largest linear effects. The highest yields, 78.3–82.0 wt% (mean 80.6 wt%), were obtained at the centre-point conditions, in agreement with the model prediction of 80.5 wt%. CRSO and plantain peel ash are therefore a promising non-edible, waste-derived feedstock–catalyst pair for biodiesel production.

Keywords: *Cola rostrata* Seed Oil; Plantain Peel Ash; Heterogeneous Catalyst; Biodiesel; Response Surface Methodology; Central Composite Design.

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I. INTRODUCTION

The escalating global demand for energy, coupled with increase in greenhouse gas emissions and air pollution has necessitated a shift towards green and renewable energy sources. Biodiesel, defined as mono-alkyl esters of long-chain fatty acids derived from vegetable oils or animal fats, has gained prominence as a viable alternative to petro-diesel due to its biodegradability, renewability, and cleaner combustion profile (Aderinlewo & Layode, 2018; Agatemor, 2008). Biodiesel is obtained from oils or animal fats by micro-emulsification with alcohols, catalytic cracking,

pyrolysis, and transesterification (Onukwuli et al., 2017; Umeagukwu, Onukwuli, Ude, Esonye, Ekwueme, Asadu, Okey-Onyesolu, Mbabuike, Ekuma, Trobjon, et al., 2024). Transesterification involves the conversion of triglyceride from oil or animal sources to methyl esters using alcohol in the presence of a catalyst (Asadu et al., 2024). Catalysts play a pivotal role in the transesterification reaction. Most biodiesel production has been carried out using conventional homogeneous catalysts. These catalysts have some drawbacks: they are difficult to separate from the product, wastewater is generated during biodiesel purification, and they are limited to oils with FFA values below 2%. Pre-

treatment of high-FFA feedstocks can therefore increase the cost of biodiesel production (Umeagukwu et al., 2023). Furthermore, the shift from homogeneous to heterogeneous catalysts offers significant advantages in terms of catalyst reusability, easier product separation, and reduced environmental pollution (Jamil, 2024; Kannan et al., 2024; Kesharvani et al., 2024).

The use of agricultural waste as feedstock has both environmental benefits, in terms of pollution control, and economic benefits (Asadu et al., 2021). This has informed the use of plantain peel as a catalyst source for biodiesel production, since plantain peels are rich in potassium and other alkali metals that remain in the ash after combustion. To avoid competition with food, non-edible oils are used as alternative feedstocks: jatropha (Adebayo et al., 2011), African pear (Ogunsuyi, 2015; Umeagukwu, Onukwuli, et al., 2022a) and avocado (Umeagukwu, Onukwuli, Ude, Esonye, Ekwueme, Asadu, Okey-Onyesolu, Mbabuike, Ekuma, Trobjon, et al., 2024) seed oils, as well as rubber seed oil, have been studied as feedstocks for biodiesel production. *Cola rostrata* K. Schum. is a West African tree that has been studied mainly for its proximate and phytochemical composition and the bioactivity of its extracts (Ajayi et al., 2023; Asogwa et al., 2022; Ogbu et al., 2023). Its seed oil (CRSO) was only recently evaluated as a biodiesel feedstock (Iyang et al., 2026).

Response Surface Methodology (RSM) models the effects of transesterification variables such as time, alcohol/oil ratio, agitation speed, catalyst loading and temperature, and their interactions, on biodiesel yield and identifies the optimum conditions. RSM has been widely adopted as a reliable optimisation tool. Onukwuli et al. (2017) employed response surface methodology in optimization of biodiesel production from crude cottonseed oil using sodium hydroxide. Umeagukwu, Onukwuli, et al. (2022a) and Ofoefule et al. (2013), on the other hand, employed the RSM and ANN models to optimize and predict the yield of African pear seed oil methyl ester via esterification and transesterification processes.

Despite the wide use of RSM, to the best of our knowledge no study has optimised biodiesel production from CRSO using an activated plantain peel ash (PPA) catalyst. Although the physicochemical properties of CRSO have recently been reported (Iyang et al., 2026), to the best of our knowledge its conversion to biodiesel with a waste-derived heterogeneous catalyst, and the optimisation of that process, have not been studied. This study therefore extracts and characterises CRSO, prepares and characterises KOH-activated plantain peel ash (AAPPA), investigates the effects of catalyst concentration, methanol/oil molar ratio, temperature, reaction time and stirring speed on biodiesel yield, and models and optimises the process using a central composite design of RSM.

II. MATERIALS AND METHODS

➤ Materials

The materials for the production of biodiesel include plantain peel and *Cola rostrata* seed which were both obtained from Ikot Abasi in Akwa Ibom State Nigeria. Methanol and other chemicals were bought from main market in Uyo, Akwa Ibom State, Nigeria.

➤ Seed and Catalyst Preparation

The seeds were dried in sunlight for fourteen days, crushed using a grinder into small pieces and air dried for 30 days. The plantain peels were prepared in the same way, and the ground, peel and seed were sieved to 800 µm and 900 µm, respectively. The plantain peel particles were submerged in distilled water to remove impurities and later sun-dried for five days. The plantain peel was converted to ash by burning at 600 °C in a furnace prior to alkaline activation.

• Alkaline Activation of the Plantain Peel Ash

The dried PPA was mixed with KOH in the ratio of 1:2 (mol/L) in a three-neck round bottom flask (500ml) equipped with mechanical stirrer. The mixture was placed in a water bath and heated to a temperature of 60 °C for 3 hrs with a constant speed of 300 rpm. After the impregnation process, the catalyst was then heated in a muffle furnace at a temperature of 200 °C for 2 hrs.

• Oil Extraction

The extraction of oil from the *Cola rostrata* seed was performed using solid-solvent extraction method applied by Umeagukwu, Ude, et al. (2022). 300 cm³ of n-hexane was poured into 2-L round-bottom flask. The whole set-up was then mounted on a heating mantle and allowed to reflux for some hours. The condensed extract (oil and n-hexane) was heated at a temperature of 70 °C using a rotary evaporator to isolate the free flow lipid from the solvent. The percentage of oil extracted was determined using Equation 1.

$$N = \frac{C_o}{C} \times 100 \quad (1)$$

Where N is the oil yield (%),

C_o is the mass of pure oil extracted (g)

C is the mass of the sample of seed used in the experiment (g)

➤ Characterisation of the Oil and Catalysts

The extracted CRSO was characterised by ASTM methods, FTIR spectroscopy and GC–MS. The raw and activated PPA were characterised by ASTM methods and Brunauer–Emmett–Teller (BET) analysis for their physicochemical properties and surface texture, by FTIR spectroscopy to identify functional groups, and by scanning electron microscopy (SEM) to examine surface morphology. The FTIR spectrum of the oil was recorded in the mid-infrared region (500–4000 cm⁻¹) in an evacuated chamber of a Shimadzu FTIR-8400S spectrophotometer using potassium bromide (KBr) discs as matrices.

- **Gas Chromatography–Mass Spectrometry (GC–MS)**

The Fatty acid analysis of the oil was performed with a Thermo Finnigan Trace GC/Trace DSQ/A1300, (E.I Quadropole) equipped with an SGE-BPX5 MS fused silica capillary column (film thickness 0.25µm).

- **FTIR Spectroscopy of the Catalysts**

The method employed by Ude and Onukwuli (2019) was adopted to carry out FTIR analysis of raw and activated PPA using BUCK model spectrophotometer. The sample was prepared using KBr and the analysis was done by scanning the sample through a wave number range of 600 to 4000 cm⁻¹.

- **BET Analysis of the Catalysts**

BET analysis ascertained the surface area, pore volume and pore size with nitrogen as the adsorbate and the catalyst as the adsorbent.

➤ **Transesterification Reaction**

The extracted oil from the seed was measured and transferred into a flat bottom flask placed on a hot magnetic stirrer. Then specific amount of catalyst (by weight of oil sample) mixed with the required amount of methanol (according to the specified methanol/oil molar ratio) was added. The reaction flask was kept on a hot magnetic stirrer under constant temperature with defined agitation speed throughout the reaction. At the defined time, the flask was taken out, cooled, and the biodiesel (fatty acid methyl esters, FAME) is separated from glycerol by difference in density.

The percentage of the biodiesel yield was determined by comparing the weight of biodiesel with the weight of oil used.

$$\text{Biodiesel (\% yield)} = \frac{\text{weight of biodiesel}}{\text{weight of oil used}} \times 100 \quad (2)$$

➤ **Effects of Process Parameters on Biodiesel Yield**

Catalyst concentration, methanol/oil molar ratio, temperature, time and speed are process variables that were studied at different conditions. Catalyst concentration 1–5 wt%, Methanol/oil molar ratio of 6–14 molar ratio, Temperature at 40–80 °C, reaction time of 1–5 h and stirring speed of 100–500 rpm. By keeping the other factors constant, the influence of one component at five different magnitudes on biodiesel yield was experimentally achieved.

➤ **Optimisation and Modelling of Biodiesel Production**

Design-Expert software version 13 was used to design and analyse the experiments. A central composite design (CCD) with 32 runs (a 2⁵⁻¹ half-fraction factorial of 16 runs, 10 axial runs at $\alpha = \pm 2$ and 6 centre-point runs) was generated from the factor ranges in Table 1, and the experimental matrix is presented in Table 2. Catalyst concentration, methanol/oil molar ratio, reaction temperature, reaction time and stirring speed were the five independent variables, and the biodiesel yield from transesterification of *Cola rostrata* seed oil was the response. The second-order polynomial relating the response to the independent variables is given in Equation 3.

$$Y = \beta_0 + \sum_{i=1}^5 \beta_i X_i + \sum_{i=1}^5 \beta_{ii} X_i^2 + \sum_{i=1}^5 \sum_{j=1}^5 \beta_{ij} X_i X_j \quad (3)$$

Table 1 Factor Levels Used in the Central Composite Design (Actual and Coded Values).

Factor	Symbol	-2 (-α)	-1	0	+1	+2 (+α)
Catalyst concentration (wt%)	A	1	2	3	4	5
Methanol/oil molar ratio	B	6	8	10	12	14
Temperature (°C)	C	40	50	60	70	80
Reaction time (h)	D	1	2	3	4	5
Stirring speed (rpm)	E	100	200	300	400	500

Source: Authors' Compilation

Table 2 Central Composite Design Matrix (Coded and Actual Values) for the Transesterification of CRSO.

Run	A: Catalyst (wt%)		B: Methanol/oil (mol/mol)		C: Temperature (°C)		D: Time (h)		E: Speed (rpm)	
	Coded	Actual	Coded	Actual	Coded	Actual	Coded	Actual	Coded	Actual
1	-1	2	-1	8	-1	50	-1	2	+1	400
2	+1	4	-1	8	-1	50	-1	2	-1	200
3	-1	2	+1	12	-1	50	-1	2	-1	200
4	+1	4	+1	12	-1	50	-1	2	+1	400
5	-1	2	-1	8	+1	70	-1	2	-1	200
6	+1	4	-1	8	+1	70	-1	2	+1	400
7	-1	2	+1	12	+1	70	-1	2	+1	400
8	+1	4	+1	12	+1	70	-1	2	-1	200
9	-1	2	-1	8	-1	50	+1	4	-1	200
10	+1	4	-1	8	-1	50	+1	4	+1	400
11	-1	2	+1	12	-1	50	+1	4	+1	400
12	+1	4	+1	12	-1	50	+1	4	-1	200
13	-1	2	-1	8	+1	70	+1	4	+1	400
14	+1	4	-1	8	+1	70	+1	4	-1	200
15	-1	2	+1	12	+1	70	+1	4	-1	200
16	+1	4	+1	12	+1	70	+1	4	+1	400
17	-2	1	0	10	0	60	0	3	0	300

18	+2	5	0	10	0	60	0	3	0	300
19	0	3	-2	6	0	60	0	3	0	300
20	0	3	+2	14	0	60	0	3	0	300
21	0	3	0	10	-2	40	0	3	0	300
22	0	3	0	10	+2	80	0	3	0	300
23	0	3	0	10	0	60	-2	1	0	300
24	0	3	0	10	0	60	+2	5	0	300
25	0	3	0	10	0	60	0	3	-2	100
26	0	3	0	10	0	60	0	3	+2	500
27	0	3	0	10	0	60	0	3	0	300
28	0	3	0	10	0	60	0	3	0	300
29	0	3	0	10	0	60	0	3	0	300
30	0	3	0	10	0	60	0	3	0	300
31	0	3	0	10	0	60	0	3	0	300
32	0	3	0	10	0	60	0	3	0	300

Source: Authors' Compilation

III. RESULTS AND DISCUSSION

➤ Oil Extraction

The oil yield of CRSO was 50.7 wt%, which is comparable to the value reported by Iyang et al. (2026). This high oil content makes the seed attractive as a feedstock for biodiesel synthesis.

➤ Characterisation of the Oil

The seed oil was characterized for physicochemical properties, functional groups and fatty acid composition using ASTM, FTIR and GC-MS, respectively.

• Physicochemical Properties of *Cola rostrata* Seed Oil

The free fatty acid (FFA) content of the oil was 1.145% (acid value 2.29 mg KOH/g), as shown in Table 3. This is below the 2% limit for single-step base-catalysed

transesterification, so no acid pre-treatment is required. The FFA content is lower than the value (12.33%) obtained by Ogunsuyi (2015) for African pear seed oil (APSO) and the 4.49% obtained by Ofoefule et al. (2013) for tiger nut oil. The density and viscosity of the oil are high enough to make atomisation in an internal combustion engine difficult, hence the oil cannot be used directly as fuel. The low acid value indicates that little soap will form, which eases separation of the biodiesel from glycerol and favours a higher yield. The physicochemical properties of the raw oil compare favourably with those of other non-edible oils such as *Jatropha curcas* (Adebayo et al., 2011) and *Madhuca indica* (Azam et al., 2005). The saponification value (202.26 mg KOH/g) is in close agreement with that of *jatropha* seed oil (193.55 mg KOH/g), while the peroxide value (1.95 meq/kg) satisfies the Codex Alimentarius recommendation and suggests that the oil is fairly stable and may not readily become rancid during storage.

Table 3 Physicochemical Properties of *Cola rostrata* Seed Oil (CRSO).

Property	<i>Cola rostrata</i> seed oil
Relative density	0.9138
Kinematic viscosity at 90 °C (mm ² /s)	6.21
Energy value (J/g)	32345
Flash point (°C)	194
Fire point (°C)	224
Acid value (mg KOH/g)	2.29
Saponification value (mg KOH/g)	202.26
Peroxide value (meq/kg)	1.95
Iodine value (g I ₂ /100 g oil)	105.02
Molecular weight (g/mol)	842.2
Ester value (mg KOH/g)	199.97
Free fatty acid (%)	1.145
Moisture content (%)	0.01
Refractive index	1.47

Source: Authors' Compilation

• GC-MS Analysis of *Cola rostrata* Seed Oil

The fatty acid composition of *Cola rostrata* seed oil was determined by gas chromatography–mass spectrometry (GC-MS). Table 4 shows that the oil is dominated by unsaturated fatty acids, mainly linoleic acid (42.72%) and oleic acid

(28.18%). The saturated acids (capric, myristic, palmitic, margaric, stearic, arachidic, lignoceric and behenic) account for about 26.3% of the listed components and the unsaturated acids for about 73.7%; hence CRSO is a linoleic–oleic type oil. Its linoleic and oleic contents are comparable with those of common vegetable oils such as cottonseed oil (Rashid et

al., 2009). The high degree of unsaturation is favourable for the cold-flow properties of the resulting biodiesel but may reduce its oxidative stability. Malvalic and sterculic acids are cyclopropenoid fatty acids, which are sensitive to heat and

acidic conditions, so their reported amounts should be regarded as approximate. Gadoleic, erucic and eicosenic acids were not detected(nd).

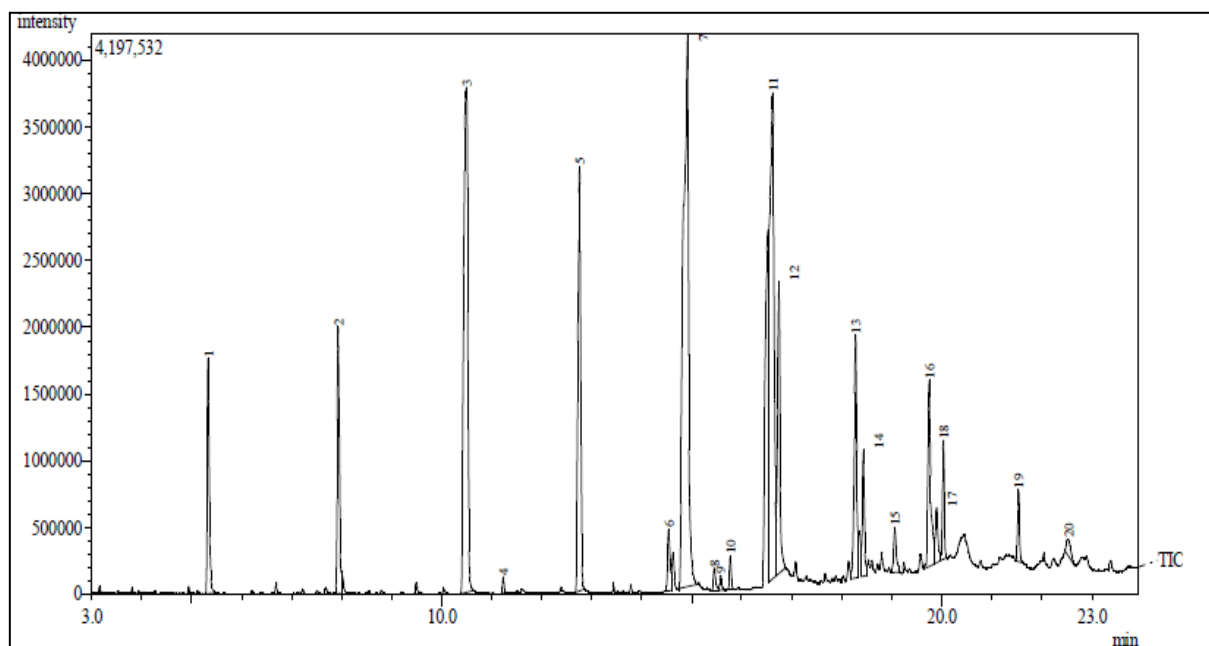


Fig 1 GC–MS Chromatogram of *Cola rostrata* Seed Oil.

Table 4 Fatty Acid Composition of *Cola rostrata* Seed Oil Determined by GC–MS.

S/N	Fatty acid	Carbon number (C:D)	Composition (%)
1	Capric acid	C10:0	0.72
2	Myristic acid	C14:0	7.32
3	Palmitic acid	C16:0	12.35
4	Palmitoleic acid	C16:1	0.42
5	Margaric acid	C17:0	2.54
6	Stearic acid	C18:0	2.15
7	Oleic acid	C18:1	28.18
8	Vaccenic acid	C18:1	0.32
9	Malvalic acid ^a	C18:1	0.56
10	Linoleic acid	C18:2	42.72
11	Linolenic acid	C18:3	0.72
12	Sterculic acid ^a	C19:1	0.78
13	Arachidic acid	C20:0	0.64
14	Gadoleic acid	C20:1	nd
15	Eicosenic acid	C20:1	nd
16	Behenic acid	C22:0	0.31
17	Erucic acid	C22:1	nd
18	Lignoceric acid	C24:0	0.27
	Total saturated		26.30
	Total unsaturated		73.70

nd = Not Detected. ^aCyclopropenoid Fatty Acids; their Amounts are Approximate because these Acids are Sensitive to Heat and Acid Derivatisation.

Source: Authors' Compilation

• FTIR Spectrum of *Cola rostrata* Seed Oil

The FTIR spectrum of CRSO (Figure 2) was recorded to identify the functional groups present in the oil, and the labelled bands are assigned in Table 5 following the assignments typically reported for vegetable oils (Shuit et al., 2010). The strong band at 1724.42 cm⁻¹ is attributed to the

C=O stretching vibration of the ester carbonyl of the triglycerides, and the band at 1197.83 cm⁻¹ to the C–O stretching of the ester group. The intense band at 2908.82 cm⁻¹ arises from aliphatic C–H stretching of the CH₂ and CH₃ groups, while the bands at 1425.44 and 1340.57 cm⁻¹ correspond to C–H bending. The band at

721.40 cm^{-1} is characteristic of the rocking of long $(\text{CH}_2)_n$ chains and of the out-of-plane bending of $\text{cis}=\text{C}-\text{H}$ groups, consistent with the unsaturated fatty acid profile in Table 4. The broad band at 3475.84 cm^{-1} is assigned to $\text{O}-\text{H}$

stretching of traces of moisture or hydroxyl groups, and the weak band at 2351.30 cm^{-1} is due to atmospheric CO_2 . The weak features at 2081.97 cm^{-1} and above 3700 cm^{-1} are not diagnostic.

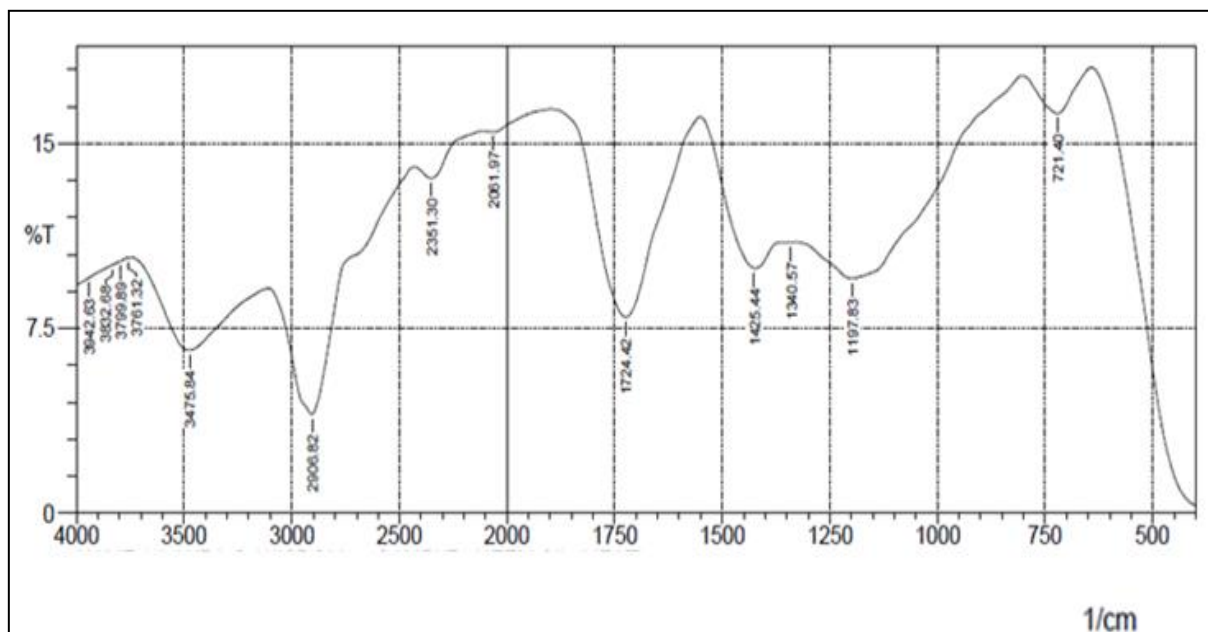


Fig 2 FTIR Spectrum of *Cola rostrata* Seed Oil.

Table 5 FTIR Band Assignments for *Cola rostrata* Seed Oil.

S/N	Wavenumber (cm^{-1})	Assignment	Vibration mode
1	721.40	$-(\text{CH}_2)_n$ rocking; $\text{cis}=\text{C}-\text{H}$ out-of-plane bending	Bending
2	1197.83	$\text{C}-\text{O}$ of the ester group ($\text{C}-\text{O}-\text{C}$)	Stretching
3	1340.57	$\text{C}-\text{H}$ of CH_3/CH_2 groups	Bending
4	1425.44	$\text{C}-\text{H}$ of CH_2/CH_3 groups	Bending
5	1724.42	$\text{C}=\text{O}$ of the ester carbonyl (triglyceride)	Stretching
6	2081.97	Weak band (not diagnostic)	—
7	2351.30	Atmospheric CO_2	Asymmetric stretching
8	2908.82	$\text{C}-\text{H}$ of CH_2/CH_3 groups (aliphatic)	Stretching
9	3475.84	$\text{O}-\text{H}$ (moisture/hydroxyl)	Stretching

Source: Authors' Compilation

➤ Characterisation of the Catalysts

The raw and activated catalysts were characterized with BET, SEM, FTIR and ASTM standard.

the total porosity from 18.77% to 47.74% and reduced the bulk density from 0.8147 to 0.58 g cm^{-3} , which may be attributed to the opening of pores during activation.

• Physicochemical Properties of the Catalysts

Table 6 summarises the physicochemical properties of the catalysts. Activation with the alkaline solution increased

Table 6 Physicochemical Properties of Raw and Activated Plantain Peel Ash (PPA).

Property	Raw PPA	Activated PPA
Particle density (g cm^{-3})	1.0080	1.01
Total porosity (%)	18.77	47.74
Moisture (%)	0.39	0.84
Ash (%)	6.08	2.85
Bulk density (g cm^{-3})	0.8147	0.58
Organic carbon (%)	0.26	0.29
Organic matter (%)	0.80	0.86

Source: Authors' Compilation.

• FTIR Analysis of the Catalysts

Fourier transform infrared spectrometer (FT-IR) was used to determine the functional group. The presence of OH and SiO₂ functional groups was confirmed by the FT-IR

spectra. Comparing the raw and modified catalysts, there was no significant decrease in the intensity of the bands, suggesting that activation changed the functional groups only slightly and acted mainly on the textural properties (porosity and surface area (Tables 6 and 7).

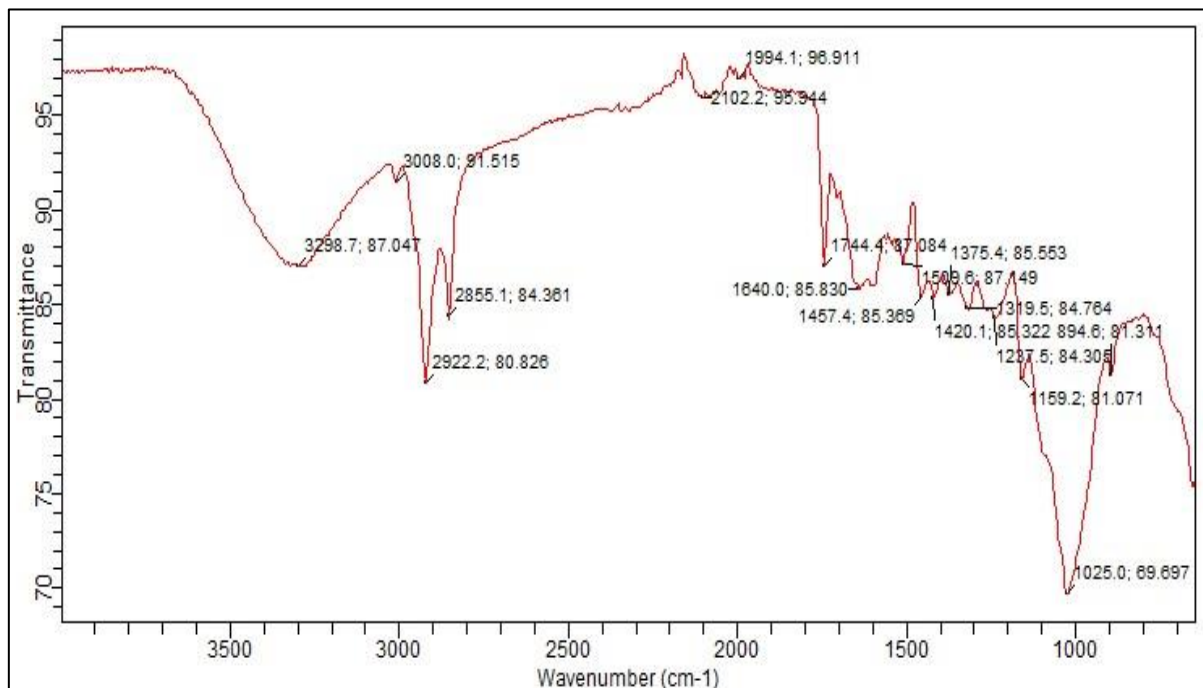


Fig 3 FTIR Spectrum of Raw Plantain Peel Ash (PPA).

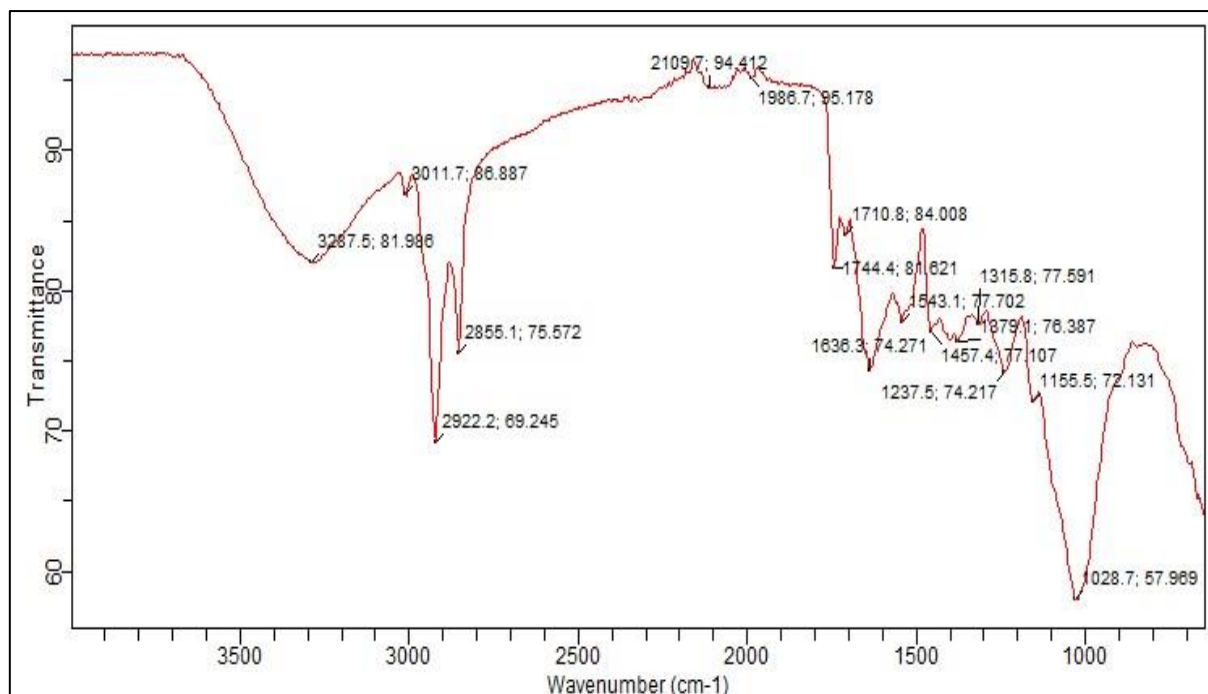


Fig 4 FTIR Spectrum of KOH-Activated Plantain Peel Ash (AAPP).

• Scanning Electron Microscopy (SEM) of the Catalysts

The morphology of the raw and activated PPA catalyst were investigated by SEM as shown in Plates 1 and 2 (300×

magnification). Micrographs of the PPA catalyst samples synthesized by alkaline activation showed an increase in the number of pores and surface area and a decrease in pore size.

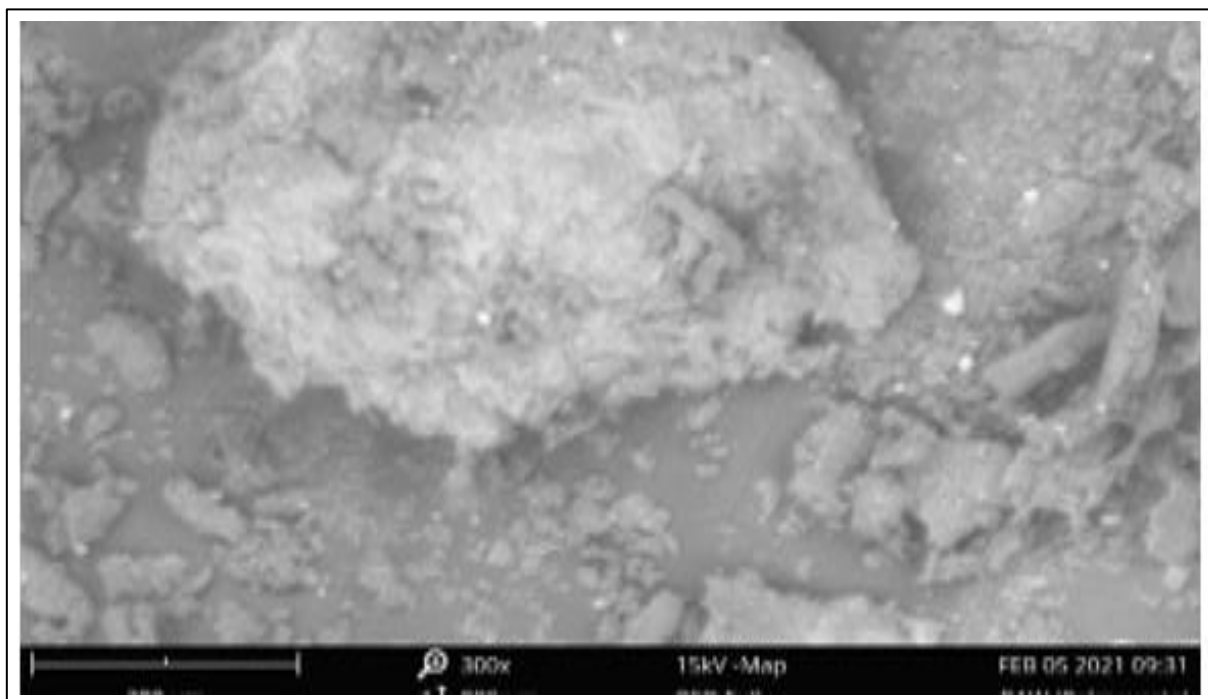


Plate 1 SEM Micrograph of Raw PPA (300× Magnification).

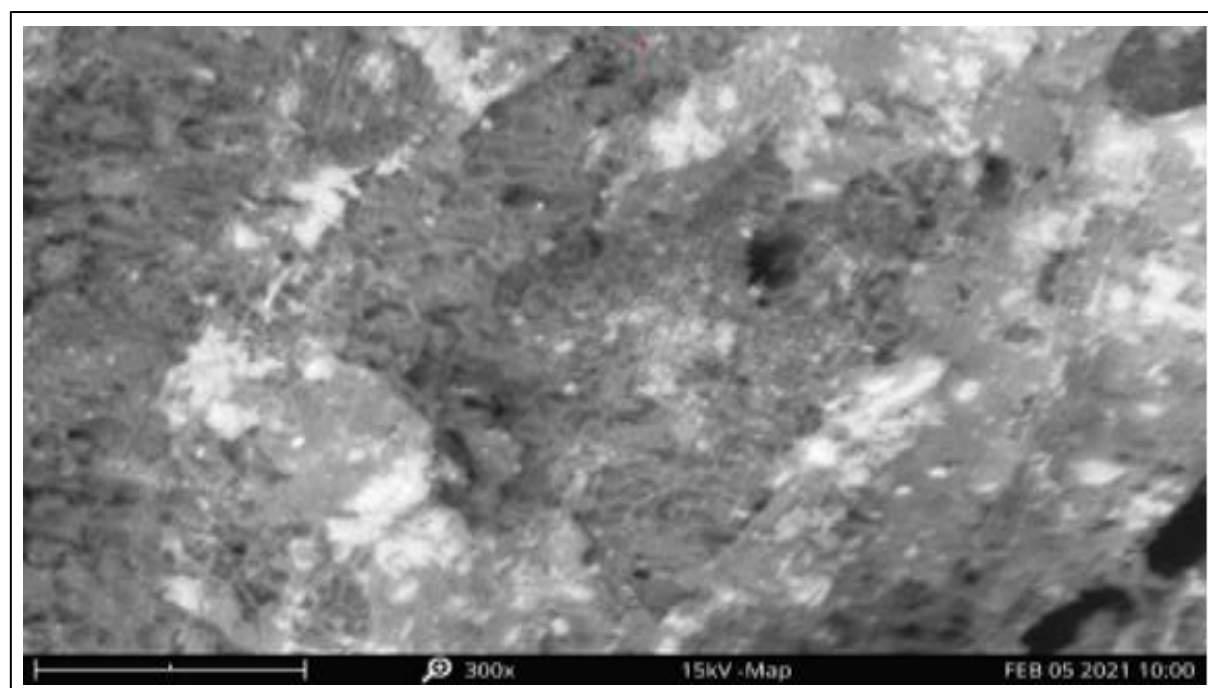


Plate 2 SEM Micrograph of Activated PPA (300× Magnification).

- BET Analysis of the Catalysts*

Table 7 shows the change in the structure of raw PPA after activation with KOH. The BET surface area increased

from 113.6 to 387.2 m²/g and the pore volume from 0.228 to 0.575 cm³/g, while the pore diameter decreased slightly from 1.855 to 1.771 nm. This confirms the formation of more pores in the activated PPA particles.

Table 7 BET Surface Area, Pore Volume and Pore Diameter of Raw and Activated PPA.

Catalyst	BET surface area (m ² /g)	Pore volume (cm ³ /g)	Pore diameter (nm)
Raw PPA	113.6	0.228	1.855
Activated PPA	387.2	0.575	1.771

Source: Authors' Compilation

➤ Effects of Process Parameters on Biodiesel Yield

The effects of the process conditions on biodiesel yield from CRSO are presented in Figures 5 to 9, which plot the biodiesel yield (wt%) as a function of each operating variable.

• Effect of Reaction Time

The effect of reaction time from 1 hr. to 5 hrs on the yield of biodiesel using the heterogeneous catalysts (raw PPA and activated PPA) is shown in Figure 5. The yield decreased

after the optimum time of 3 hrs because transesterification is a reversible reaction. The activated catalyst produced a higher biodiesel yield than the raw catalyst. Ude and Onukwuli (2019) also obtained a high yield of biodiesel using alkaline-activated clay.

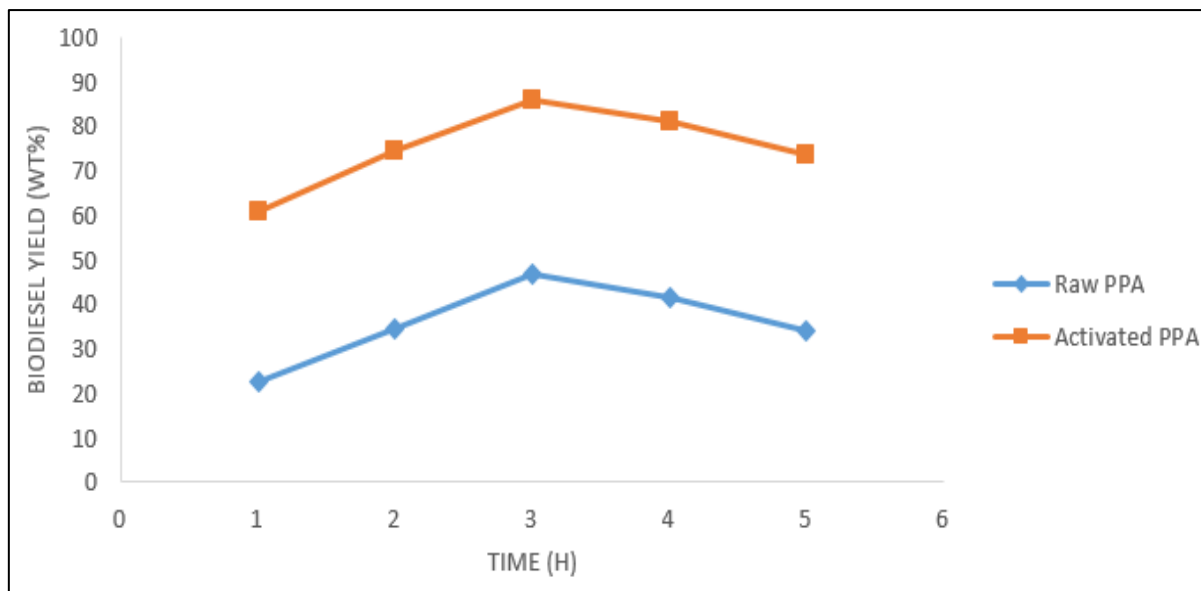


Fig 5 Effect of Reaction Time on FAME Yield.

✓ Conditions: catalyst 3.0 wt%, stirring speed 300 rpm, 60 °C, methanol/oil molar ratio 10:1.

• Effect of Temperature

The effect of temperature on the yield of the transesterification reaction is shown in Figure 6. The reaction

temperature was varied as 40, 50, 60, 70 and 80 °C while the other parameters were held constant. The biodiesel yield first increased and then decreased as the temperature rose beyond 60 °C. Generally, higher temperatures give a faster reaction rate, but methanol (boiling point about 65 °C) is lost by vaporisation at higher temperatures. A similar result was reported by Ude and Onukwuli (2019).

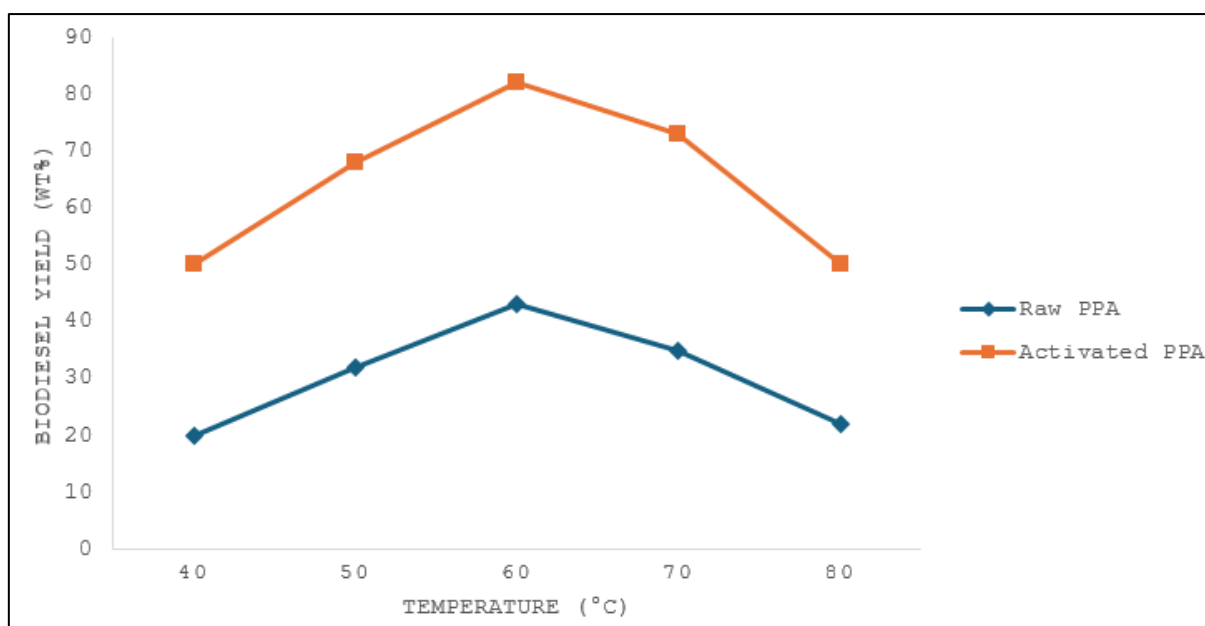


Fig 6 Effect of Reaction Temperature on FAME Yield.

- ✓ Conditions: catalyst 3.0 wt%, stirring speed 300 rpm, methanol/oil molar ratio 10:1, 3 hrs.

and the yield decreased at higher ratios. This could be due to dilution of the catalyst by the excess alcohol (Liu et al., 2007).

- *Effect of Methanol/Oil Molar Ratio*

In Figure 7, the maximum FAME yield for both catalysts was obtained at a methanol/oil molar ratio of 10:1,

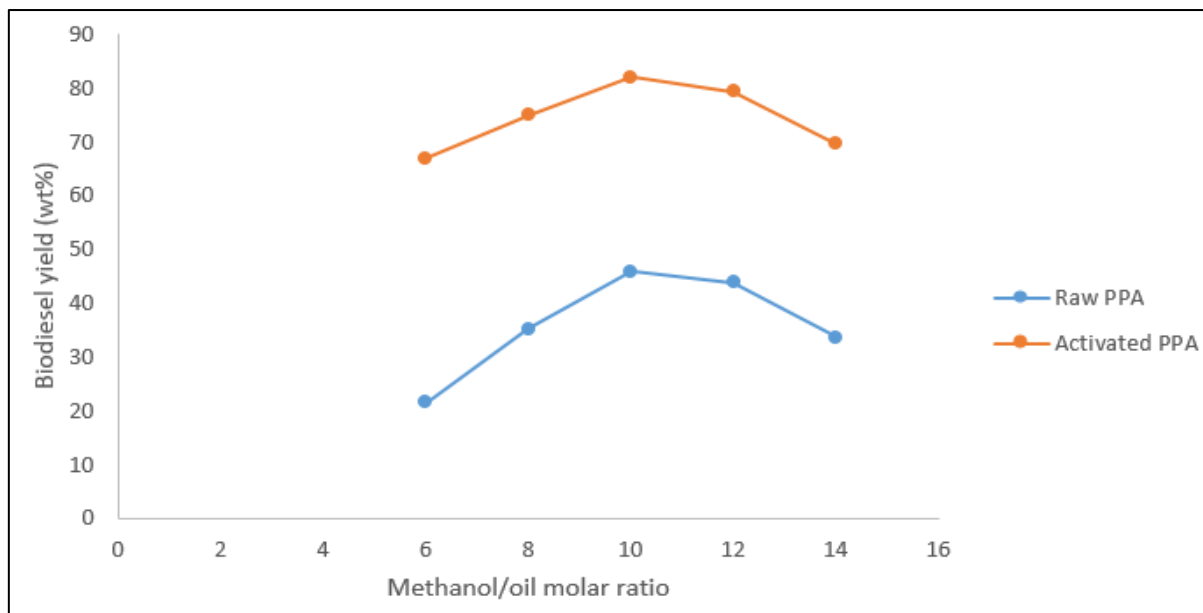


Fig 7 Effect of Methanol/Oil Molar Ratio on FAME Yield.

- ✓ Conditions: catalyst 3.0 wt%, stirring speed 300 rpm, 3 hrs, 60 °C.

energy barrier. The lower yield of methyl ester at catalyst loadings above 3 wt% may be due to dispersion and mixing problems caused by excess catalyst, which inhibit the formation of the product (Zhang et al., 2003).

- *Effect of Catalyst Concentration*

Figure 8 shows that the biodiesel yield initially increased with the amount of catalyst, which accelerates the reaction by increasing the reaction rate and lowering the

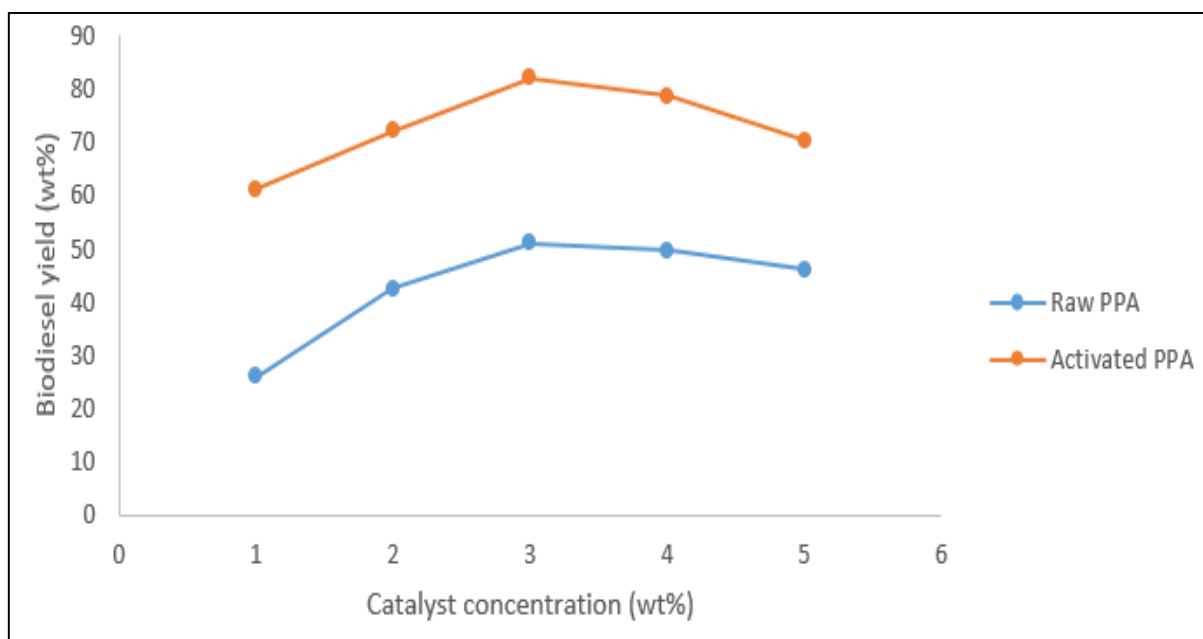


Fig 8 Effect of Catalyst Concentration on FAME Yield.

- ✓ Conditions: methanol/oil molar ratio 10:1, stirring speed 300 rpm, 3 hrs, 60 °C.

• Effect of Stirring Speed

Mixing ensures homogeneity of the reaction mixture, initiates the reaction and increases the contact area between the reactants. The yield of methyl esters at different stirring speeds (100, 200, 300, 400 and 500 rpm) is shown in Figure 9. This agrees with Umeagukwu, Onukwuli, et al. (2022b),

who reported that higher agitation promoted homogenisation of the reactants in the transesterification of non-edible oil and led to higher yields. The optimum yield was obtained at 300 rpm, and the yield decreased at higher stirring speeds for both raw and modified PPA. This could be due to the strong collision effect caused by the increase in the kinetic energy of the molecules.

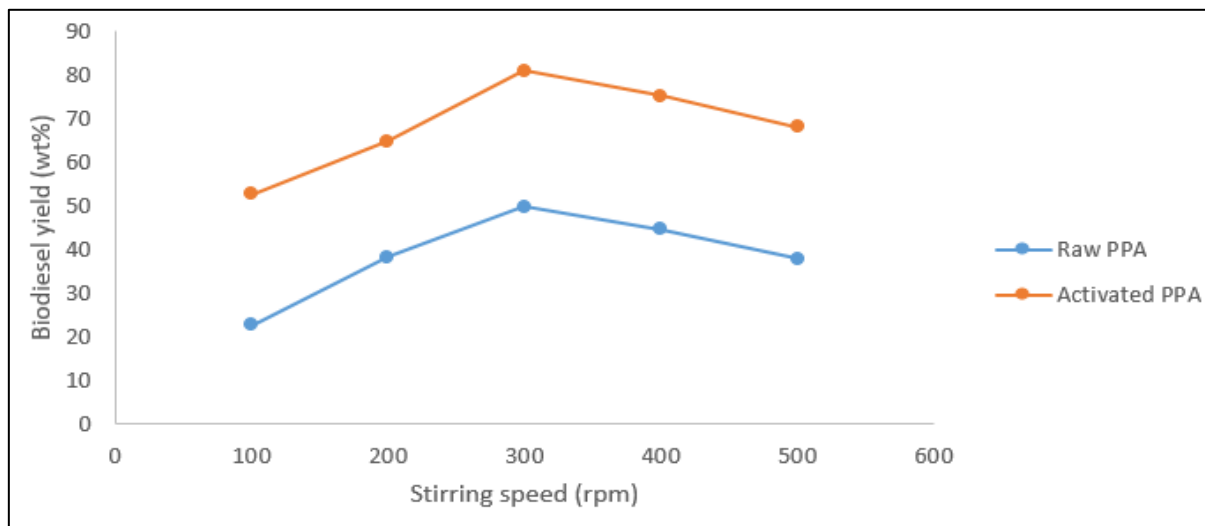


Fig 9 Effect of Stirring Speed on FAME Yield.

✓ Conditions: catalyst 3.0 wt%, 3 hrs, 60 °C, methanol/oil molar ratio 10:1.

➤ Response Surface Modelling of CRSOME Production

Design-Expert software version 13 was used to develop the model and to analyse it statistically for the production of CRSO methyl ester (CRSOME) using the alkaline-activated plantain peel ash (AAPPA) catalyst.

The central composite design (CCD) comprised 32 experiments (16 factorial runs from a two-level, five-factor half-fraction design, 10 axial runs and 6 centre-point runs), and the biodiesel yield from transesterification was the

response. Analysis of the experimental results (Table 8) gave the second-order model in Equation 4, where A–E are the coded independent variables defined below.

$$\text{FAME yield} = 80.53 + 0.67A + 2.63B + 1.98C + 1.63D + 3.04E - 4.64A^2 - 4.31B^2 - 3.99C^2 - 4.10D^2 - 3.71E^2 - 1.13AB - 0.68AC + 0.30AD - 1.15AE - 0.47BC + 0.14BD + 0.006BE + 0.72CD - 0.35CE + 0.14DE \quad (4)$$

Where A = catalyst concentration; B = methanol/oil molar ratio; C = temperature; D = reaction time; E = stirring speed (coded variables).

Table 8 Observed and Predicted Yields of CRSO Methyl Ester (CRSOME) from the Central Composite Design.

Run	A: Catalyst (wt%)	B: Methanol/oil (mol/mol)	C: Temp. (°C)	D: Time (h)	E: Speed (rpm)	Observed yield (wt%)	Predicted yield (wt%)
1	2	8	50	2	400	56.32	56.16
2	4	8	50	2	200	54.21	54.03
3	2	12	50	2	200	55.72	55.53
4	4	12	50	2	400	62.01	61.87
5	2	8	70	2	200	53.11	52.88
6	4	8	70	2	400	59.79	59.61
7	2	12	70	2	400	66.78	66.58
8	4	12	70	2	200	58.79	58.58
9	2	8	50	4	200	48.18	48.00
10	4	8	50	4	400	60.76	60.64
11	2	12	50	4	400	66.27	66.13
12	4	12	50	4	200	60.25	60.10
13	2	8	70	4	400	64.56	64.38
14	4	8	70	4	200	61.75	61.56
15	2	12	70	4	200	63.46	63.25
16	4	12	70	4	400	67.43	67.28

17	1	10	60	3	300	60.25	60.64
18	5	10	60	3	300	63.02	63.33
19	3	6	60	3	300	57.67	58.03
20	3	14	60	3	300	68.2	68.54
21	3	10	40	3	300	60.35	60.63
22	3	10	80	3	300	68.12	68.54
23	3	10	60	1	300	60.48	60.87
24	3	10	60	5	300	67.09	67.40
25	3	10	60	3	100	59.17	59.59
26	3	10	60	3	500	71.48	71.77
27	3	10	60	3	300	81.19	80.53
28	3	10	60	3	300	82.01	80.53
29	3	10	60	3	300	78.25	80.53
30	3	10	60	3	300	82.04	80.53
31	3	10	60	3	300	79.24	80.53
32	3	10	60	3	300	81.14	80.53

Predicted Yields are from the Full Quadratic Model (Equation 4)

Source: Authors' Compilation

• Model Adequacy

The ANOVA result for the model given in Table 9 at the 5% significance level shows that variables with a p-value less than or equal to 0.05 are significant. From the p-values presented in Table 9, it can be stated that all the linear terms A, B, C, D and E, interaction terms AB, AC, AE and CD, and quadratic terms A^2 , B^2 , C^2 , D^2 , and E^2 are significant model

terms. Based on this, the insignificant terms of the model were removed and Equation 4 was reduced to Equation 5, while Equation 6 gives the model in terms of the actual (uncoded) variables. The ANOVA shows that the quadratic terms have the largest F-values, followed by the linear terms (in the order $E > B > C > D > A$), while the interaction terms contribute less.

$$\text{FAME yield} = 80.53 + 0.67A + 2.63B + 1.98C + 1.63D + 3.04E - 4.64A^2 - 4.31B^2 - 3.99C^2 - 4.10D^2 - 3.71E^2 - 1.13AB - 0.68AC - 1.15AE + 0.72CD \quad (5)$$

The model in terms of the actual (uncoded) variables is:

$$\text{FAME} = -350.41 + 41.684 * \text{Catalyst concentration} + 24.562 * \text{Methanol/oil molar ratio} + 4.9683 * \text{Temperature} + 21.894 * \text{Reaction time} + 0.28785 * \text{Agitation speed} - 0.56531 * \text{Catalyst concentration} * \text{Methanol/oil molar ratio} - 0.068063 * \text{Catalyst concentration} * \text{Temperature} - 0.011544 * \text{Catalyst concentration} * \text{Agitation speed} + 0.072063 * \text{Temperature} * \text{Reaction time} - 4.6353 * \text{Catalyst concentration}^2 - 1.0776 * \text{Methanol/oil molar ratio}^2 - 0.039853 * \text{Temperature}^2 - 4.0978 * \text{Reaction time}^2 - 0.00037128 * \text{Agitation speed}^2 \quad (6)$$

Table 9 Analysis of Variance (ANOVA) of the Full Quadratic Model for CRSOME Yield.

Source	Sum of squares	df	Mean square	F value	Prob > F	Remark
Model	2530.48	20	126.52	100.04	< 0.0001	Significant
A	10.84	1	10.84	8.57	0.0137	
B	165.85	1	165.85	131.13	< 0.0001	
C	93.97	1	93.97	74.30	< 0.0001	
D	63.86	1	63.86	50.49	< 0.0001	
E	222.47	1	222.47	175.89	< 0.0001	
AB	20.45	1	20.45	16.17	0.0020	
AC	7.41	1	7.41	5.86	0.0340	
AD	1.47	1	1.47	1.16	0.3040	Not significant
AE	21.32	1	21.32	16.86	0.0017	
BC	3.54	1	3.54	2.80	0.1223	Not significant
BD	0.33	1	0.33	0.26	0.6208	Not significant
BE	0.0005	1	0.0005	0.0004	0.9844	Not significant
CD	8.31	1	8.31	6.57	0.0264	
CE	1.93	1	1.93	1.52	0.2430	Not significant
DE	0.33	1	0.33	0.26	0.6178	Not significant
A^2	630.27	1	630.27	498.32	< 0.0001	
B^2	544.99	1	544.99	430.89	< 0.0001	
C^2	465.90	1	465.90	368.36	< 0.0001	

D ²	492.57	1	492.57	389.45	< 0.0001	
E ²	404.37	1	404.37	319.71	< 0.0001	
Residual	13.91	11	1.26			
Lack of fit	1.85	6	0.31	0.13	0.9868	Not significant
Pure error	12.06	5	2.41			
Cor total	2544.39	31				

A–E are the Coded Factors Defined Under Equation 4; df = Degrees of Freedom.

Source: Authors' Compilation

Figure 10 shows a positive correlation between the actual and predicted biodiesel yields obtained with the activated catalyst, and the normal plot of residuals in Figure 11 shows the relationship between normal probability and externally studentised residuals, which supports the adequacy of the model equation. From the ANOVA in Table 9 (full quadratic model), $R^2 = 0.995$, adjusted $R^2 = 0.985$ and predicted $R^2 = 0.977$, with adequate precision of 35.7 and a coefficient of variation of 1.7%. After removing the six non-significant interaction terms, the reduced model (Equation 5) retained $R^2 = 0.992$, adjusted $R^2 = 0.985$ and predicted $R^2 = 0.977$, i.e. no meaningful loss of fit, i.e. the model explains more than 98% of the variation in yield.

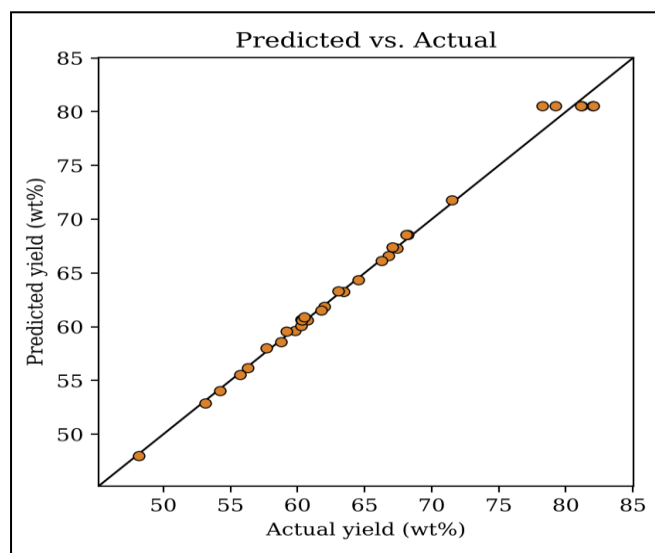


Fig 10 Predicted Versus Observed CRSOME Yields.

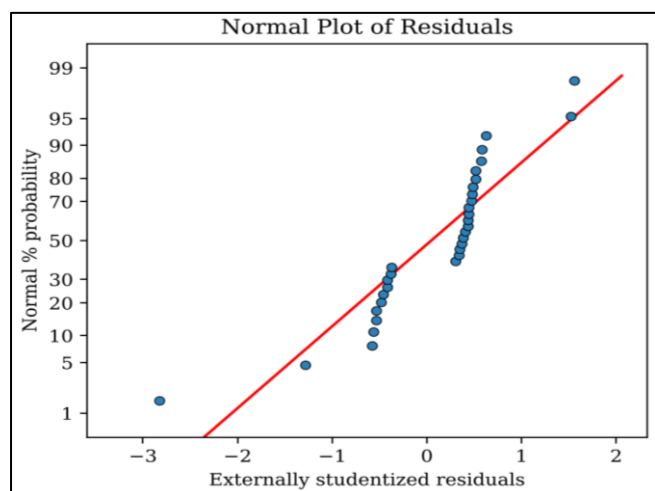


Fig 11 Normal Probability Plot of the Externally Studentised Residuals for CRSOME Yield.

• Three-Dimensional Response Surface Plots

The 3D surface plots in Figures 12–14 show the effect of the interactions AB, AE and CD on biodiesel yield, together with the corresponding quadratic curvature; the AC interaction is also significant ($p = 0.034$) but weaker, and the remaining interactions (AD, BC, BD, BE, CE and DE) were not significant. In each plot the other three factors are held at their centre values. Figure 12 shows the interaction between catalyst concentration and methanol/oil molar ratio. The methyl ester yield increases with both variables up to an intermediate level, but at higher catalyst concentration and methanol/oil ratio the yield decreases. This may be because excess catalyst and methanol dilute the reaction mixture and hinder mass transfer.

The interaction between catalyst concentration and stirring speed is also significant (Figure 13). As in Figure 12, the yield increases with catalyst concentration and stirring speed, which may be due to better mixing, but at higher catalyst concentration and stirring speed the yield decreases, possibly because excess catalyst and vigorous agitation hinder phase separation and favour the backward reaction.

The interaction between reaction temperature and time is shown in Figure 14. The positive CD coefficient means that the gain in yield from a longer reaction time is larger at higher temperature; the yield is highest near 60 °C and 3 h and falls on either side of these values because of the quadratic terms. The fall at higher temperature may be due to vaporisation of methanol near and above its boiling point (about 65 °C).

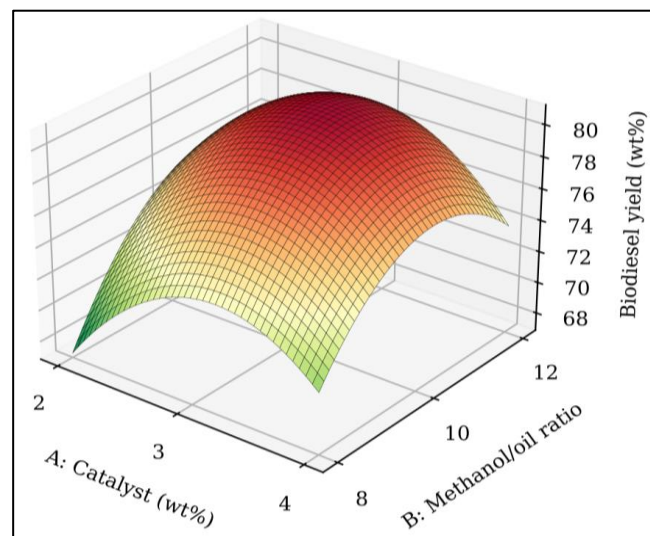


Fig 12 Response Surface of CRSOME Yield as a Function of Catalyst Concentration (A) and Methanol/Oil Molar Ratio (B); the Other Factors are at their Centre Values.

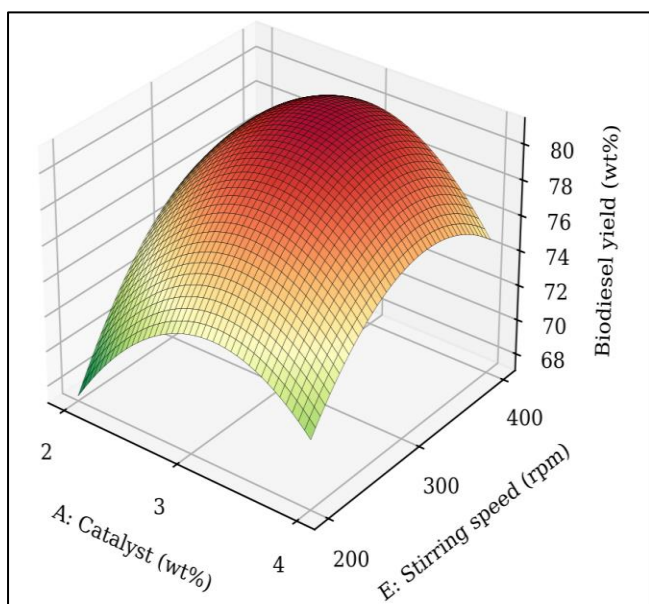


Fig 13 Response Surface of CRSOME Yield as a Function of Catalyst Concentration (A) And Stirring Speed (E); The Other Factors are at their Centre Values.

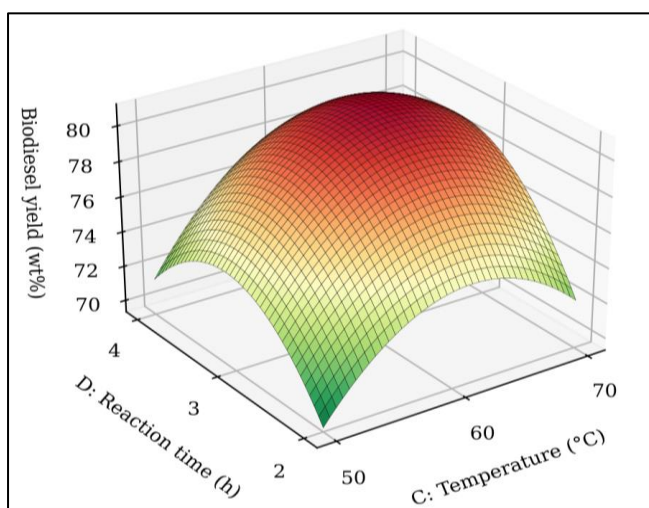


Fig 14 Response Surface of CRSOME Yield as a Function of Reaction Temperature (C) and Time (D); the Other Factors are at their Centre Values.

• Optimum Conditions

The highest biodiesel yields were obtained at the centre point of the design (3 wt% catalyst, 10:1 methanol/oil molar ratio, 60 °C, 3 h and 300 rpm). The six replicate runs at these conditions gave experimental yields of 78.25–82.04 wt% (mean 80.65 wt%), compared with the model prediction of 80.53 wt% (Table 8), and this agrees with the optimum found in the one-factor-at-a-time study (Figures 5–9). All other design points gave lower yields, between 48.18 and 71.48 wt%. Numerical optimisation of the reduced model (Equation 5) within the design region gave a stationary point that is a true maximum (all eigenvalues of the quadratic form are negative) at 2.96 wt% catalyst, a 10.6:1 methanol/oil molar ratio, 62.7 °C, 3.2 h and 342 rpm, with a predicted yield of 82.0 wt% (95% confidence interval 81.1–82.9 wt%; 95% prediction interval 79.5–84.6 wt%). This is only about 1.5 wt% above the model prediction at the centre point and is comparable to the scatter of the centre-point replicates (standard deviation 1.55 wt%), so the centre-point conditions can be taken as a practical optimum.

➤ Comparison with Other Biowaste-Derived Catalysts

To place the yield in context, Table 10 compares the optimum conditions and yields of this study with recent studies that used plantain- or banana-derived catalysts for transesterification. The values are those reported by the original authors. They are not strictly comparable, because the feedstocks, alcohols, catalyst preparation and, in particular, the way yield or conversion was determined differ between studies.

Table 10 Comparison of this Study with Other Plantain- and Banana-Derived Catalysts for Biodiesel Production.

Reference	Feedstock	Catalyst (calcination)	Cat. (wt%)	Alcohol: oil molar ratio	Temp. (°C)	Time	Yield / conversion
Etim et al. (2018)	Neem (<i>Azadirachta indica</i>) oil, after FFA pre-treatment	Ripe plantain peel ash (700 °C)	0.65	0.73 v/v MeOH	65	57 min	99.2 wt% yield
Falowo et al. (2022)	Waste cooking oil	Ripe and unripe plantain peel mixture (500 °C, 4 h)	0.5	6:1	45	45 min	97.96 wt% yield
Jisieike et al. (2025)	Rubber seed oil (ethanolysis)	Blend of plantain peel, fluted pumpkin, cocoa and kola nut husks (300 °C, 4 h)	1.5	9:1 (ethanol)	60	40 min	93.05 ± 0.11 wt% yield; above 85 wt% beyond the 5th cycle

Efeovbokhan et al. (2026)	Waste cooking oil (acid value 8.8 mg KOH/g)	KOH/CaO composite from snail shell and plantain peel	7	9:1	60.5	1.93 h	92.10 wt% (gravimetric)
Tarigan et al. (2023)	Palm oil	Waste banana peel (600 °C, 2 h)	7	15:1	Room temp.	30 min	97.7 ± 0.6% conversion (GC); yield 90.0 ± 2.3%
Rosli et al. (2023)	Used frying oil	Modified banana peel waste	1	12:1	65	2 h	88.7% yield
Laskar et al. (2022)	Waste cooking oil	Banana flower petal ash (open-air burning)	5.63	6.24:1	28	3.68 h	98.55 ± 0.4% conversion (¹ H NMR); 82% at the 4th cycle
This study	<i>Cola rostrata</i> seed oil (FFA 1.1%)	KOH-activated plantain peel ash (600 °C)	3	10:1	60	3 h	80.6 wt% yield (gravimetric; mean of 6 centre-point runs)

The yield obtained here (80.6 wt%) is lower than those reported in most of these studies (88.7 to 99.2%). Part of the difference may come from how yield is measured. Laskar et al. (2022) and Tarigan et al. (2023) report conversion determined by ¹H NMR or GC, which is not directly comparable with the gravimetric yield used here (Equation 2), and Efeovbokhan et al. (2026), who also used a gravimetric yield, obtained 92.10% from a different catalyst system. The two highest reported yields (Etim et al., 2018; Falowo et al., 2022) were reached in less than one hour with 0.5–0.65 wt% of a plantain peel catalyst, whereas six of the seven reference studies used shorter reaction times than the 3 h used here. This suggests that the reaction time and catalyst loading used here are not the main limit on yield, but the reason for the lower yield cannot be established from the present data. Measuring the basicity of the catalyst and quantifying conversion by GC or ¹H NMR would help to resolve it. Reusability was reported for three of the studies (Laskar et al., 2022; Jisieike et al., 2025; Tarigan et al., 2023), the last of which found poor recyclability because K₂O was lost from the catalyst, and it has not yet been tested for the AAPPA catalyst.

IV. CONCLUSIONS

This study showed that *Cola rostrata* seed oil (CRSO), which has a high oil content (50.7 wt%) and a low free fatty acid content (1.145%), can be converted into biodiesel in a single transesterification step using a catalyst prepared from plantain peel ash, an agricultural waste. KOH activation raised the total porosity of the ash from 18.77 to 47.74% and its BET surface area from 113.6 to 387.2 m²/g, and the activated catalyst gave higher biodiesel yields than the raw ash.

The one-factor-at-a-time study and the central composite design agreed on the optimum conditions of 3 wt% catalyst, a 10:1 methanol/oil molar ratio, 60 °C, 3 h and 300 rpm, which gave biodiesel yields of 78.3–82.0 wt% (mean 80.6 wt%). The quadratic RSM model was highly significant ($p < 0.0001$, $R^2 = 0.995$) with a non-significant lack of fit. Stirring speed and methanol/oil molar ratio had the largest linear effects, and the AB, AC, AE and CD interactions were significant. Numerical optimisation of the

fitted model predicts a maximum of about 82.0 wt% close to these conditions (3.0 wt% catalyst, 10.6:1, 62.7 °C, 3.2 h and 342 rpm), which is within the replicate scatter.

Overall, CRSO and plantain peel ash form a promising non-edible, waste-derived feedstock catalyst pair for biodiesel production. Further work on the fuel properties of the biodiesel, catalyst reusability and process economics is needed before scale-up can be recommended.

V. LIMITATIONS OF THE STUDY

➤ This Study has the Following Limitations

- The work was done at laboratory batch scale with seeds and plantain peels from a single location (Ikot Abasi), so variation in feedstock composition between sites and seasons was not assessed.
- The optimisation covered five factors within fixed ranges, and the highest yields occurred at the centre of the design, so the response surface was not explored beyond the studied ranges.
- Catalyst reusability, leaching and stability were not tested, and no kinetic or thermodynamic analysis of the reaction was made.
- The fuel properties of the CRSO biodiesel (compared with ASTM D6751 and EN 14214) and the FTIR and GC–MS characterisation of the methyl esters are not reported in this paper.
- No economic or life-cycle assessment was carried out, so the cost and environmental benefit of the process remain unquantified.

RECOMMENDATIONS FOR FUTURE STUDIES

Future studies should measure and report the fuel properties of the CRSO biodiesel (density, viscosity, acid value, cetane number, iodine value, oxidation stability, flash point and cold-flow properties) against ASTM D6751 and EN 14214, and evaluate its blends with petro-diesel, test catalyst reusability and leaching over repeated cycles and characterise the catalyst further (XRD, EDX/XRF and basicity measurements), confirm the optimum with independent

validation runs and compare RSM with artificial neural network or other machine-learning models for yield prediction, study the kinetics and thermodynamics of the reaction, assess seed and peel from several locations and scale up to pilot level, carry out a techno-economic and life-cycle assessment, as done for other seed oils by Umeagukwu, Onukwuli, Ude, Esonye, Ekwueme, Asadu, Okey-Onyesolu, Mbabuike, Ekuma, and Ugwele (2024); and evaluate engine performance and emissions of the biodiesel.

➤ CRediT Authorship Contribution Statement

Ephraim R. Afia: Conceptualization, Supervision, Formal analysis, Writing original draft; Emmanuel U. Okwudili: Methodology, Investigation; Sifon J. Umoh: Methodology, Investigation; Inuaeyen N. Inuaeyen: Methodology, Writing, review & editing; Abasiemek A. Archibong: Writing, review & editing; Owoidoho I. Brown: Formal analysis, Writing.

➤ Declaration of Competing Interest

The authors declare that they have no known financial competing interest or personal relationship that could have appeared to influence the output of this study.

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