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OPTIMIZATION OF PROCESS VARIABLES FOR THE PRODUCTION OF FATTY ACID METHYL ESTERS FROM *BIGPOD SESBANIA* SEED OIL

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ABSTRACT

This study explores the potential of *Bigpod Sesbania* seed oil as a feedstock for the production of biodiesel. The extracted oil was converted into biodiesel through a transesterification reaction using potassium hydroxide as a catalyst. Or: Response Surface Methodology (RSM) based on the Box–Behnken design was employed to optimize the effects of methanol-to-oil molar ratio, catalyst concentration, reaction time, and reaction temperature on biodiesel yield. The maximum biodiesel yield of 96% was obtained at an 8:1 methanol-to-oil ratio, 0.5 wt.% catalyst, 60 min reaction time, and a temperature of 55 °C. The physicochemical properties of the biodiesel conformed to ASTM D6751 and EN 14214 standards: specific gravity 0.8491, viscosity 4.39 cP, acid value 0.32 mg KOH/g, ash content 0.01%, flash point 166°C, and pour point 4.2 °C. These values indicate high purity and stability. FTIR spectra confirmed the successful conversion of triglycerides to fatty acid methyl esters. GC-MS analysis revealed eight major fatty acid methyl esters (FAMEs), with methyl hexadeca-7,10,13-trienoate (29.80%), methyl (9E,12E)-octadeca-9,12-dienoate (15.97%), and carbonic acid octadecyl prop-1-en-2-yl ester (19.30%) as the dominant components. These unsaturated FAMEs accounted for 99.12% of the total composition, indicating a high degree of unsaturation. This characteristic enhances cold-flow properties but may require antioxidant additives for oxidative stability. Overall, *Bigpod Sesbania* seed oil is validated as a promising non-edible feedstock for high-yield, high-quality biodiesel production with favorable physicochemical properties.

Keywords: *Bigpod Sesbania*, biodiesel, transesterification, RSM optimization, fatty acid methyl esters, ASTM standard

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1. INTRODUCTION

1.1 Background to the Study

Volatile petroleum prices and environmental challenges associated with greenhouse gas (GHG) emissions have driven research into renewable energy [1]. However, the use of edible oils for biodiesel has raised food-versus-fuel debates [2,3]. Hence, non-edible feedstocks are being prioritized. The optimization of biodiesel production from renewable feedstocks has, therefore, become a crucial area of research in addressing the dual challenges of energy insecurity and environmental degradation. Biodiesel, a renewable, biodegradable, and non-toxic fuel derived from vegetable oils and animal fats, represents a viable alternative to petroleum diesel due to its similar fuel properties and reduced emission profile [4,5]. Biodiesel consists mainly of fatty acid alkyl esters (FAAE), typically produced through transesterification, where triglycerides react with a short-chain alcohol in the presence of a catalyst [6,7]. The reaction yields fatty acid methyl esters (FAME) as the primary product and glycerol as a valuable by-product. Among various methods for converting triglycerides into biodiesel, such as pyrolysis, microemulsion, and catalytic hydrogenation, transesterification remains the most efficient due to its simplicity and high yield [8,9]. The process efficiency is strongly influenced by parameters such as alcohol-to-oil molar ratio, reaction time, temperature, and catalyst concentration [10]. Alkaline catalysts such as potassium hydroxide (KOH) are widely employed due to their high catalytic activity and economic viability [11]. However, excessive catalyst concentration or high free fatty acid (FFA) content in feedstocks can cause soap formation, reducing biodiesel yield. Therefore, optimizing these reaction parameters is essential to enhance conversion efficiency and product quality.

Given the escalating global energy demand and environmental concerns, there is growing interest in non-edible oil feedstocks for biodiesel production. hence, non-edible sources are being prioritized for sustainable biodiesel production [2,3]. One such promising feedstock is *Bigpod Sesbania* (*Sesbania bispinosa*), a fast-growing, nitrogen-fixing legume native to North America [12]. It is highly adaptable to various soils and climatic conditions [13]. It produces abundant seeds, making it an economically attractive and renewable non-edible oil source for biodiesel [14]. Historically cultivated as a cover crop ([15,16], its seed oil has recently gained attention for fuel applications. Recent interest in this plant has shifted toward its potential as a non-edible oil source for biodiesel production. The oil extracted from its seeds contains a high proportion of unsaturated fatty acids, which can yield methyl esters suitable for use in compression ignition (CI) engines after transesterification. However, limited research exists on optimizing the reaction conditions for biodiesel production from *Bigpod Sesbania* seed oil.

Optimization studies are critical for determining the ideal combination of process parameters to maximize biodiesel yield and quality while minimizing production costs. Statistical modeling approaches such as Response Surface Methodology (RSM) are widely used for such optimization because they efficiently evaluate multiple variables and their interactions [17]. Thus, this research focuses on optimizing the transesterification parameters for biodiesel production from *Bigpod Sesbania* seed oil and evaluating the fuel's physicochemical properties for compliance with ASTM biodiesel standards.

2. MATERIAL AND METHODS

2.1 Sample collection and pre-treatment

Seeds of *Bigpod Sesbania* were collected from farmland in Auno village, Konduga Local Government Area, Borno State, northeastern Nigeria. They were authenticated and identified by a botanist from the Department of Plant Science, Usmanu Danfodiyo University, Sokoto. The seeds were washed thoroughly with clean water to remove surface contaminants. They were then shade-dried at ambient temperature for 7 days to reduce moisture content, thereby facilitating easier cracking and preventing microbial growth during storage (Figure 1). After drying, the seeds were manually cracked using a mortar and pestle to loosen the hulls. The cracked seeds were then gently winnowed to separate the kernels from the hulls. The recovered seed kernels were subjected to a second 7-day shade-drying period to eliminate residual moisture, ensuring efficient milling and oil extraction. The dried kernels were crushed into a fine powder using an electric-powered milling machine. The resulting powder was further dried on a solar dryer for 24 hours to remove any remaining moisture, as excess water can interfere with solvent-based oil extraction.

2.2 Extraction of Oil from *Sesbania bispinosa* Seeds

The powdered seeds of *Sesbania bispinosa* were subjected to the Soxhlet extraction method for oil extraction. Oil was extracted using a conventional Soxhlet apparatus with n-Hexane as the solvent [18].

Figure 1. Seed of Bigpod sesbania

2.3 Characterization of Oil

The physicochemical parameters of the seed oil were determined using the Association of Official Analytical Chemists (AOAC) technique (the acid value by AOAC Ca5a-40, the saponification value by AOAC 920:160, the iodine value by AOAC 920:158, and the peroxide value by AOAC 965.33) [19]. The viscosity and specific gravity were measured using an Ostwald viscometer thermostated at 40°C and a thermal-hydrometer apparatus in accordance with ASTM standards D445 and D1298, respectively, while the density was measured using a density bottle, and the moisture content was determined using the oven method. The ash content was estimated by heating to dryness in a Veisfar muffle furnace.

2.4 Optimization of Transesterification Parameters

The transesterification of *Sesbania bispinosa* seed oil was conducted using methanol and potassium hydroxide (KOH) as a homogeneous catalyst to produce fatty acid methyl esters (biodiesel). The reaction was carried out in a 500 cm³ round-bottom flask fitted with a reflux condenser to prevent methanol vapor loss during heating [20]. The reaction mixture was stirred using a magnetic stirrer to ensure proper mixing and maintained in a water bath under controlled conditions. Upon completion, the mixture was transferred into a separating funnel and allowed to settle under gravity overnight to facilitate phase separation.

The upper methyl ester layer was carefully collected and heated to remove residual methanol. The biodiesel was then neutralized with dilute phosphoric acid (pH 4.0) and subsequently washed with hot distilled water (1:5 v/v) until the wash water attained a neutral pH of 7.0. The purified methyl esters were dried at 100°C over anhydrous sodium sulfate to eliminate residual moisture. The percentage yield of biodiesel was determined using Equation (1):

$$\text{Biodiesel yield (\%)} = \frac{\text{Weight of biodiesel}}{\text{Weight of oil}} \times 100 \qquad (1)$$

[20]

Optimization of the transesterification parameters was performed using the Box–Behnken Design (BBD) under Response Surface Methodology (RSM) to assess the individual and interactive effects of the process variables on biodiesel yield. The Box–Behnken Design, a rotatable second-order statistical model, provides an efficient means of evaluating quadratic effects with a minimal number of experimental runs, while the Response Surface Methodology offers a mathematical and statistical framework for analyzing and optimizing chemical processes [21]. Four independent process variables' reaction time (30–90 min), catalyst concentration (0.1–0.5 wt%), methanol-to-oil molar ratio (4:1–8:1), and reaction temperature

(45–65°C) were selected based on preliminary studies. Each variable was examined at three coded levels (low, medium, and high) to determine its influence on biodiesel production. The operating parameters selected for the Box–Behnken Design, together with their investigated ranges, are presented in Table 1. The experimental design matrix generated by the BBD was employed to conduct the transesterification runs, and the corresponding biodiesel yields were used as response data for RSM-based model development. This approach enabled the identification of optimal process conditions necessary to achieve high conversion efficiency and superior biodiesel quality.

Table 1. Effect of operating parameters on yield

Factors	Low level	High level
Time (mins)	30	90
catalyst (wt%)	0.1	0.5
MeOH:Oil	4:1	8:1
Temp (°C)	45	65

2.5 Determination of Biodiesel Properties

The physicochemical properties of the biodiesel were determined according to ASTM standard methods and AOAC (2000) official procedures. The kinematic viscosity was determined by the ASTM D-445 method, the density was determined by the ASTM D-1298 method, and the pour point determination was made using the ASTM D-97 method. The flash point of the fuel was determined by ASTM D-93, the value of cloud point was estimated according to ASTMD-2500 and the acid value was measured following the ASTM D-664 method. The specific gravity was ascertained using AOAC 920.212 and iodine value using AOAC 920:159. The moisture content was determined using the air-oven method using the Rotary Evaporator Oven (BTOV 1423), while the cetane number (CN) was calculated by the equation developed by [22].

2.6 FT–IR Spectroscopy

Fourier Transform Infrared (FTIR) spectroscopy was used to identify the functional groups present in the produced biodiesel. Characteristic absorption bands corresponding to specific molecular vibrations (e.g., C=O stretching, C–H bending) were analyzed to confirm the conversion of triglycerides to fatty acid methyl esters (FAMEs).

2.6.1 Fourier Transform Infrared (FTIR) Spectroscopic Analysis

FTIR spectra were recorded on an Agilent Cary 630 FTIR spectrometer controlled with Microlab software. Samples were measured using the ATR accessory with a diamond crystal. Spectra were collected over the range 4000–400 cm^{-1} at 4 cm^{-1} resolution with 32 co-added scans; backgrounds were recorded immediately before sample measurements. Each sample was measured in triplicate by removing and reapplying the sample to the ATR crystal to assess reproducibility. Spectra were converted to absorbance, baseline corrected (polynomial order 2), and lightly smoothed (Savitzky–Golay, window 7, 2nd order) in Microlab. Peak positions are reported as the mean of three replicates ± standard deviation and assigned by comparison to literature values.

2.6.2 Gas Chromatography - Mass Spectrometry

GC-MS analysis was conducted on an Agilent 7890A GC-5977 MSD system with an HP-5ms column (30 m × 0.25 mm × 0.25 µm). Helium carrier gas flowed at 1.16 mL/min. The injector (split less mode) and transfer line were set at

280°C. The oven program was: 50 °C (2 min) to 280°C at 10°C/min (5 min hold). The MSD operated at 70 eV (EI) with the ion source at 230°C and quadrupole at 150°C. Mass spectra were acquired over m/z 50–650. Compound identification used the NIST library.

2.7 Gas Chromatography-Mass Spectrometry (GC-MS) Analysis

GC-MS analysis was conducted on an Agilent 7890A GC-5977 MSD system equipped with an HP-5ms column (30 m × 0.25 mm × 0.25 µm). Helium was used as the carrier gas at a flow rate of 1.16 mL/min. The injector operating in splitless mode and the transfer line were both set at 280°C. The oven temperature program was as follows: initial temperature of 50°C held for 2 min, then ramped to 280°C at 10°C/min, with a final hold of 5 min. The mass selective detector (MSD) operated in electron ionization (EI) mode at 70 eV, with the ion source at 230°C and the quadrupole at 150°C. Mass spectra were acquired over a range of m/z 50–650. Compound identification was performed using the NIST mass spectral library.

3. Results and Discussion

3.1 Physical and chemical characteristics of seed oils and biodiesel

The seed oils were evaluated by measuring their physicochemical characteristics and functional groups using FT-IR analysis. The biodiesel derived from seed oils was further studied in terms of physicochemical characteristics, functional groups, and fatty acid compositions. The physicochemical properties of Bigpod sesbania seed oil determined in this study are presented in Table 2.

The oil content is an important consideration when selecting plant seeds as a potential feedstock for biodiesel and other industrial applications. Oil yield of 20% was recovered using Soxhlet extraction of the *Bigpod sesbania seeds oil*, which is much greater than the 12% oil yield reported by [23]. But within the 15-20% range reported by other researchers [24]. The oil content is substantial and similar to that of cottonseed oil, as reported by [25].

Table 2. Physicochemical properties of *Bigpod sesbania* seed oil.

Parameters	Values
Specific Gravity	0.91 ± 0.01
Density (g/cm^3)	0.89 ± 0.01
Viscosity @ 40 C (mPa.s)	30.5 ± 0.40
Acid Value (mg KOH/g)	2.4 ± 0.05
Free fatty acid	1.2 ± 0.03
Ash content	0.17 ± 0.01
Moisture	0.07 ± 0.005
Saponification Value (mg KOH/g)	189 ± 1.50
Flash point (°C)	205 ± 2.0
Pour point (°C)	-6 ± 0.5
Ester value (mg KOH/g)	186.6 ± 1.0
Average Iodine Value (IV)	32.2 g I_2/100g

The density (0.89 g/cm^3) falls within the typical vegetable oil range (0.882–0.925 g/cm^3) and agrees with *Jatropha curcas* (0.87–0.91 g/cm^3) and soybean (0.88–0.90 g/cm^3) oils [26].

The acid value (2.4 mg KOH/g; FFA 1.2%) is below the 2% threshold requiring pretreatment, making the oil suitable for direct base-catalyzed transesterification [27,28]. The saponification value (189 mg KOH/g) aligns with jatropha (193.55 mg

KOH/g) and castor (202 mg KOH/g) oils [29]. The iodine value (32.2 g I_2/100g) indicates moderate unsaturation, balancing oxidative stability and cold-flow performance [30]. The flash point (205°C) confirms thermal stability and safe handling [26]. Moisture content (0.07%) was low compared to other seed oils [31]. promoting high biodiesel yield.

3.2 Optimal biodiesel production parameters of *Bigpod sesbania* seed oil

The experimental design matrix and the corresponding biodiesel yields obtained under the investigated transesterification conditions are presented in Table 3, while the analysis of variance (ANOVA) and response surface regression results for the developed model are presented in Table 4. Transesterification optimization identified the maximum biodiesel yield of 96% at 60 min, 0.5 wt% KOH, 8:1 methanol-to-oil ratio, and 55°C (Table 4, Run 4). Temperature significantly influenced yield, with 55°C promoting faster glycerol separation [32]. Excessive catalyst (0.5 wt% at 30 min) gave a lower yield (71%) due to soap formation, while a higher acid value promoted saponification, drastically reducing ester yield.

Table 3. Process evaluation parameters and experimental results

Run Order	Time (mins)	Catalyst Conc (wt%)	MeOH:Oil	Temp. (^{0}C)	Methyl ester yield (wt %)
					Experimental
1	30	0.1	6:1	55	86
2	90	0.1	6:1	55	87
3	30	0.5	6:1	55	71
4	90	0.5	6:1	55	94
5	60	0.3	4:1	45	71
6	60	0.3	8:1	45	76
7	60	0.3	4:1	65	71.4
8	60	0.3	8:1	65	72
9	30	0.3	6:1	45	76
10	90	0.3	6:1	45	78.5
11	30	0.3	6:1	65	69.5
12	90	0.3	6:1	65	72
13	60	0.1	4:1	55	76.3
14	60	0.5	4:1	55	80
15	60	0.1	8:1	55	78
16	60	0.5	8:1	55	96
17	30	0.3	4:1	55	59
18	90	0.3	4:1	55	83
19	30	0.3	8:1	55	41
20	90	0.3	8:1	55	78
21	60	0.1	6:1	45	94
22	60	0.5	6:1	45	94
23	60	0.1	6:1	65	85
24	60	0.5	6:1	65	76
25	60	0.3	6:1	55	67
26	60	0.3	6:1	55	67
27	60	0.3	6:1	55	66.6

3.4 Response Surface Regression Analysis

The ANOVA results (Table 5) showed the regression model was highly significant ($p < 0.05$) with $R^2 = 98.93\%$, confirming a strong correlation between experimental and predicted values. Reaction time and temperature significantly affected yield ($p < 0.001$), while catalyst and methanol-to-oil ratio were not significant at the linear level. Quadratic effects of catalyst and temperature were highly significant, indicating excessive levels of lower yield due to soap formation [33].

Table 4. Response Surface Regression: yield versus Time, Catalyst, Methanol, Temperature

Source	DF	Adj SS	Adj MS	F-Value	P-Value	SIGNIFICANT
Model	14	2631.58	187.97	79.04	0.000	S
Linear	4	880.38	220.09	92.55	0.000	S
Time	1	665.71	665.71	279.92	0.000	S
Catalyst	1	1.07	1.07	0.45	0.514	Ns
Methanol	1	2.28	2.28	0.96	0.347	Ns
Temp	1	211.32	211.32	88.85	0.000	S
Square	4	1500.57	375.14	157.74	0.000	S
Time*Time	1	7.78	7.78	3.27	0.096	Ns
Catalyst*Catalyst	1	1202.32	1202.32	505.55	0.000	S
Methanol*Methanol	1	6.51	6.51	2.74	0.124	Ns
Temp*Temp	1	162.05	162.05	68.14	0.000	S
2-Way Interaction	6	250.64	41.77	17.56	0.000	S
Time*Catalyst	1	123.63	123.63	51.98	0.000	S
Time*Methanol	1	42.26	42.26	17.77	0.001	S
Time*Temp	1	0.05	0.05	0.02	0.884	Ns
Catalyst*Methanol	1	21.30	21.30	8.96	0.011	S
Catalyst*Temp	1	58.55	58.55	24.62	0.000	S
Methanol*Temp	1	4.85	4.85	2.04	0.179	Ns
Error	12	28.54	2.38			
Lack-of-Fit	10	28.53	2.85	481.39	0.002	S
Pure Error	2	0.01	0.01			
Total	26	2660.12				

Model Summary S= 1.54215 R^2 = 98. 93% R^2(adj) 97.68% R^2(pred) 93.82%

Table 5. Summary of Significant Model Terms for Biodiesel Yield

Source	DF	Adj SS	Aji MS	F-Value	P-Value
Model	9	2606. 77	289.64	92.28	0.000
Linear	2	877. 02	438.51	139.72	0.000
Time	1	665.71	665.71	212.10	0.000
Temp	1	211.32	211.32	67.33	0.000
Square	2	1479.16	739.58	235.64	0.000
Catalyst*Catalyst	1	1437.86	1437.86	458.13	0.000
Temp*Temp	1	192.66	192.66	61.38	0.000
2-Way Interaction	5	250.58	50.12	15.97	0.000
Time*Catalyst	1	123.63	123.63	39.39	0.000
Time*Methanol	1	42.26	42.26	13.46	0.002
Catalyst*Methanol	1	21.30	21.30	6.79	0.018
Catalyst*Temp	1	58.55	58.55	18.66	0.000
Methanol*Temp	1	4.85	4.85	1.55	0.231
Error	17	53.36	3.14		
Lack-of-Fit	15	53.34	3.56	600.12	0.002
Pure Error	2	0.01	0.01		

Total	26	2660.12

Model Summary S= 1.77160 R^2 = 97.99% R^2(adj) 96.93% R^2(pred) 94.39%

The characteristic FTIR absorption bands identified for the produced biodiesel are presented in Table 6.

Table 6. FTIR Analysis of Biodiesel Derived from *Sesbania* Oil

Functional Group	Type of Vibration	Wavenumber (cm^{-1})
C−H	Stretching	3100
C−H	Asymmetric stretching	2926
C-H	Stretching	2851
C=O	Streching	1748
CH_2	Bending	1459

3.5 Regression Equation in Uncoded Units

Yield (%) = 266.80 − 0.537 × Time − 208.7 × Catalyst + 1.58 × Methanol − 5.602 × Temp + 0.0013 × (Time)^2 + 375.4 × (Catalyst)^2 − 0.276 × (Methanol)^2 + 0.05512 × (Temp)^2 + 0.927 × (Time × Catalyst) + 0.0542 × (Time × Methanol) + 0.00038 × (Time × Temp) + 5.77 × (Catalyst × Methanol) − 1.913 × (Catalyst × Temp) − 0.0551 × (Methanol × Temp)

3.6 Analysis of Variance (ANOVA) and Model Summary

The Analysis of Variance (ANOVA) presented in Table 6 evaluates the statistical significance of the regression model and the contribution of each process variable: reaction time, catalyst concentration, methanol-to-oil ratio, and temperature, to the methyl ester yield from *Bigpod Sesbania* seed oil. The model demonstrates a high degree of reliability and precision, as indicated by the strong statistical parameters [34,35].

3.6.1 The regression equation after removing insignificant terms

Yield (%) = 66.979 + 7.448 × Time − 4.196 × Temp + 14.989 × (Catalyst)^2 + 5.487 × (Temp)^2 + 5.559 × (Time × Catalyst) + 3.250 × (Time × Methanol) + 2.308 × (Catalyst × Methanol) − 3.826 × (Catalyst × Temp) − 1.101 × (Methanol × Temp)

3.6.2 Significance of the Model

The model was highly significant ($p < 0.05$) with F-value of 92.28, confirming the quadratic model is superior to the mean response (Meher et al., 2006). R^2 = 97.99% indicates the model explains nearly 98% of yield variation. Close agreement between adjusted R^2 (96.93%) and predicted R^2 (94.39%) confirms predictive accuracy without overfitting [6].

3.6.3 Linear Effects

Both reaction time ($F = 212.10$, $p < 0.001$) and temperature ($F = 67.33$, $p < 0.001$) had a statistically significant influence on biodiesel yield. Among the linear terms, reaction time exhibited the largest F-value, indicating the strongest relative effect on yield within the studied range. This is likely because longer reaction durations improve contact between reactants, enhancing conversion efficiency. Temperature also positively influenced yield by improving reaction kinetics; however, excessive temperatures may lead to methanol vaporization, reducing the effective alcohol-to-oil ratio and thus lowering yield [36].

3.6.4 Quadratic (Square) Effects

The significant quadratic term for catalyst concentration ($F = 458.13$, $p < 0.001$) indicates a curvilinear relationship, with yield increasing up to an optimum (approximately 0.3–0.5 wt%) and declining beyond due to soap formation. Similarly, the quadratic term for temperature ($F = 61.38$, $p < 0.001$) confirms a parabolic response, predicting an optimum around 55°C, where kinetics are favorable, and methanol loss is minimized [37].

3.6.5 Two-Way Interaction Effects

Time×Catalyst (F = 39.39, $p < 0.001$) was the strongest interaction—catalyst effectiveness depends on reaction duration. Time×Methanol (F = 13.46, p = 0.002), Catalyst×Methanol (F = 6.79, p = 0.018), and Catalyst×Temp (F = 18.66, $p < 0.001$) were also significant. Methanol×Temp was insignificant (p = 0.231) [6].

3.6.6 Lack-of-Fit and Model Adequacy

The lack-of-fit test was significant (p = 0.002), suggesting that the model may not fully capture all sources of variation. However, the pure error was very small (0.01), and the low standard deviation (S = 1.7716) indicates acceptable model precision for predictive purposes [37].

3.7 Contour Plots for Significant Interaction Effects

Contour plots (Fig. 2-5) revealed significant interaction effects among variables. Yield increased with time up to 90 min and catalyst up to 0.3 wt%, beyond which soap formation reduced yield [38]. Methanol-to-oil ratio up to 6:1 improved yield, while excess methanol complicated glycerol separation [39]. Temperature up to 55°C enhanced yield, but higher temperatures promoted methanol evaporation [6]. Optimal conditions were: 0.3 wt% catalyst, 6:1 methanol-to-oil ratio, 90 min, and 55°C (R^2 = 97.99%). FTIR confirmed successful transesterification: the carbonyl peak shifted from 1742 cm^{-1} (oil) to 1748 cm^{-1} (biodiesel), indicating conversion to methyl esters, while disappearance of the O–H band confirmed FFA removal [40,41].

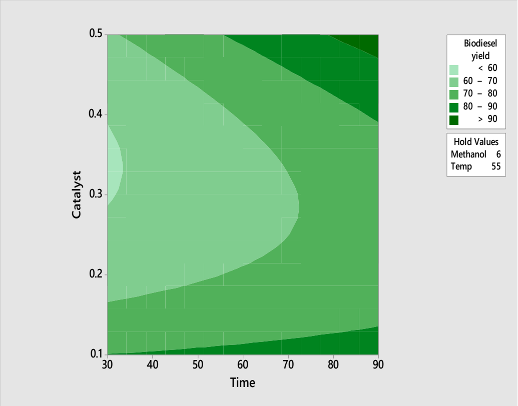

Figure 2. Catalyst and Time

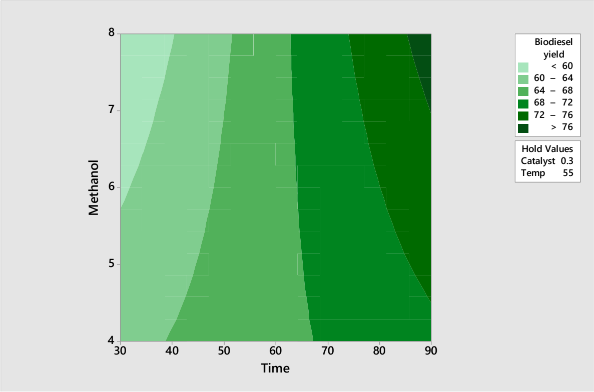

Figure 3. Oil Ratio and Time

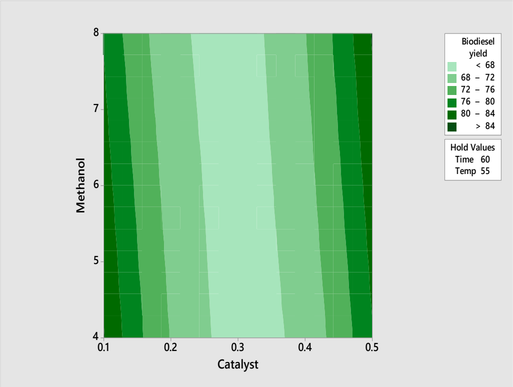

Figure 4. Oil Ratio and Catalyst

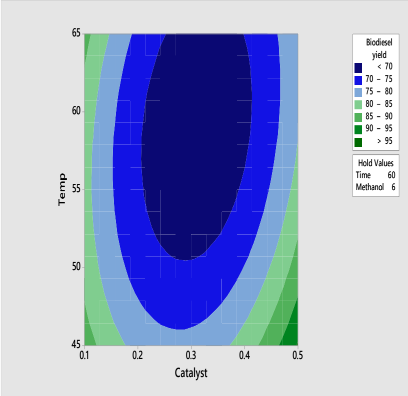

Figure 5. Temp. and Catalyst

FTIR analysis confirmed successful transesterification. The oil spectrum showed a carbonyl (C=O) stretch at 1742 cm^{-1} and C–H stretches at 2922 and 2851 cm^{-1}, characteristic of triglycerides. After transesterification, the biodiesel spectrum exhibited a shift of the carbonyl peak to 1748 cm^{-1}, confirming conversion to methyl esters [40,41]. The =C–H stretch shifted to 3100 cm^{-1}, indicating retained unsaturation. The disappearance of the O–H band (~3400 cm^{-1}) confirmed effective removal of free fatty acids and moisture. These results confirm efficient conversion of *Bigpod Sesbania* seed oil to high-purity biodiesel.

3.8 GC–MS Analysis and Discussion

The GC–MS chromatogram of the trans esterified *Bigpod Sesbania* seed oil revealed a mixture of fatty acid methyl esters (FAMEs) and oxygenated long-chain esters, confirming successful transesterification of the triglycerides into biodiesel. The identified fatty acid methyl esters (FAMEs) and their relative abundances determined by GC–MS analysis are presented in Table 7. Eight major components were identified, with molecular chain lengths ranging from C_{17} to C_{23}. The most abundant compounds were methyl hexadeca-7,10,13-trienoate (29.80%), methyl (9E,12E)-octadeca-9,12-dienoate (15.97%), and carbonic acid, octadecyl prop-1-en-2-yl ester (19.30%).

The dominance of methyl hexadecatrienoate and methyl linoleate indicates that the biodiesel possesses a high level of unsaturation, characterized by multiple double bonds. Such polyunsaturated esters improve cold-flow properties, fuel atomization, and combustion efficiency, but they may reduce oxidative stability due to the susceptibility of double bonds to oxidation during storage [28,41].

The detection of carbonic and oxalic acid esters (approximately 34% of the total composition) suggests the presence of oxygenated derivatives, possibly formed through side reactions involving alcohol intermediates and residual CO_2 during transesterification. These compounds enhance fuel lubricity and combustion performance due to their oxygen-rich structures, though excessive concentrations could slightly increase viscosity.

The absence of triglyceride peaks indicates complete conversion of the oil to methyl esters, demonstrating high reaction efficiency. The overall molecular weight distribution (C_{16}–C_{23}) aligns with values typical of biodiesels derived from long-chain plant oils and agrees with previous studies on *Sesbania* and other non-edible feedstocks [41]. The GC–MS data confirm that *Bigpod Sesbania* seed oil is a promising feedstock for biodiesel production, producing fuel rich in unsaturated methyl esters with favorable ignition quality, low-temperature fluidity, and acceptable viscosity. Nevertheless, the high degree of unsaturation suggests that antioxidant additives or blending with more saturated biodiesel could be recommended to improve oxidative stability during long-term storage. The fuel property profile of *Bigpod Sesbania* biodiesel demonstrates a highly unsaturated composition (99%), dominated by polyunsaturated esters such as methyl hexadeca-7,10,13-trienoate (C16:3) and methyl (9E,12E)-octadeca-9,12-dienoate (C18:2), along with a notable fraction of oxygenated monounsaturated esters. This composition enhances low-temperature operability, promotes complete combustion, and supports efficient atomization, all of which are essential for improved engine performance [28].

Table 7. Fatty Acid methyl esters (FAME) profile of Biodiesel from *Bigpod Sesbania* Seed Oil

Compound Name	Molecular formula	Area %
Methyl hexadeca 7,10,13 trienoate	$C_{17}H_{28}O_2$	29.80

Methyl (9E,12E)-octadeca-9,12 dienoate	$C_{19}H_{34}O_2$	15.97
Carbonic acid, octadecyl prop-1-en -2-yl ester	$C_{22}H_{42}O_3$	19.3
Carbonic acid, eicosyl vinyl ester	$C_{23}H_{44}O_3$	15.36
Carbonic acid, octadecyl vinyl ester	$C_{21}H_{40}O_3$	10.02
Oxalic acid, allyl undecyl ester	$C_{21}H_{38}O_3$	1.60
Oxalic acid, isobutyl tridecyl ester	$C_{19}H_{36}O_4$	2.83
Oxalic acid, dodecyl propyl ester	$C_{17}H_{32}O_4$	4.24

3.9 Conclusions

This study successfully produced high-quality biodiesel from *Bigpod Sesbania* seed oil via KOH-catalyzed transesterification. Optimal conditions (8:1 methanol-to-oil ratio, 0.5 wt% KOH, 60 min, 55°C) yielded 96% biodiesel. The biodiesel met ASTM D6751 and EN 14214 standards (viscosity 4.39 cP, acid value 0.32 mg KOH/g, flash point 166°C, cetane number 56.4). FTIR confirmed conversion to methyl esters, and GC-MS revealed polyunsaturated FAMEs. *Bigpod Sesbania* is a viable non-edible feedstock for sustainable biodiesel production.

Conflict of Interest

The authors declare no conflict of interest.

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