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Biodiesel Production from Brown Grease Using Potassium Hydroxide Catalyst: Optimization Via Response Surface Methodology

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ABSTRACT

This study investigated the production of biodiesel from brown grease using potassium hydroxide (KOH) as a homogeneous catalyst through an optimized transesterification process. Brown grease, a waste product from commercial kitchens with high free fatty acid (FFA) content (15.428%), was pretreated via acid esterification using sulphuric acid to reduce the FFA content to 0.75 mg KOH/g. Response Surface Methodology (RSM) with a Central Composite Design was employed to optimize three critical process parameters: reaction temperature (50-60 °C), catalyst loading (3-6 wt.%), and methanol-to-oil ratio (3:1-20:1). The optimization yielded a maximum biodiesel conversion of 64.91% at optimal conditions of 59.76 °C, 3.04 wt.% catalyst load, and 18.93:1 methanol-to-oil ratio. The produced biodiesel exhibited favorable properties: flash point of 66 °C, kinematic viscosity of 7.90 cSt at 40 °C, cloud point of -3.0 °C, specific gravity of 0.84, and water content below 0.05%, all meeting ASTM D6751 specifications. ANOVA analysis confirmed the statistical significance of the quadratic model ($p < 0.05$, $R^2 = 0.9744$), with temperature and catalyst load showing the most significant effects on biodiesel yield. This research demonstrates that brown grease is a viable, cost-effective feedstock for sustainable biodiesel production, contributing to waste management and renewable energy development in Nigeria.

Keywords: Biodiesel, brown grease, transesterification, potassium hydroxide, response surface methodology.

Introduction

The global energy landscape is experiencing a paradigm shift driven by increasing environmental concerns, fossil fuel depletion, and the urgent need for sustainable energy alternatives [1]. Conventional petroleum-based fuels contribute significantly to greenhouse gas emissions, air

pollution, and climate change, necessitating the development of renewable energy sources [2] Among various renewable energy options, biodiesel has emerged as a promising alternative to conventional diesel fuel due to its biodegradability, non-toxicity, renewable nature, and compatibility with existing diesel engines [3]

Biodiesel, chemically defined as fatty acid alkyl esters, is typically produced through the transesterification of triglycerides from vegetable oils or animal fats with short-chain alcohols in the presence of catalysts [2]. While first-generation biodiesel feedstocks such as palm oil, soybean oil, and rapeseed oil have been extensively utilized, their use raises significant concerns regarding food security and competition with agricultural resources [4]. Consequently, research efforts have shifted toward non-edible and waste-derived feedstocks for biodiesel production.

Brown grease, recovered from grease traps in commercial kitchens, restaurants, and food processing facilities, represents an attractive alternative feedstock for biodiesel production [4]. This waste material, primarily consisting of fats, oils, and greases (50-60% w/w), water (25-30% w/w), and biosolids (15-20% w/w), is characterized by high free fatty acid content, typically ranging from 15% to 50% [5]. The utilization of brown grease for biodiesel production addresses two critical challenges simultaneously: waste management and renewable energy generation.

In Nigeria, despite abundant biomass resources and significant waste generation from the food service industry, biodiesel production remains underdeveloped compared to global standards. While countries like Ghana, Niger, and Togo have initiated biodiesel production in Africa, Nigeria has yet to establish a robust biodiesel industry [6]. The development of efficient processes for converting waste materials like brown grease into biodiesel could contribute significantly to Nigeria's energy security, waste management, and environmental sustainability goals.

The transesterification process, the primary method for biodiesel production, can be catalyzed by acids, bases, or enzymes in either homogeneous or heterogeneous forms [6] Homogeneous base catalysts, particularly potassium hydroxide and sodium hydroxide (NaOH), are preferred due to their high reaction rates, lower costs, and ability to operate under mild conditions (60-90 °C, 1.5-4 atm) [4]. However, the presence of high FFA content in brown grease necessitates pretreatment through acid esterification to prevent soap formation and maximize biodiesel yield.

Process optimization is crucial for achieving economically viable biodiesel production. Response Surface Methodology (RSM), a statistical and mathematical technique, has proven effective in optimizing complex processes by systematically investigating the effects of multiple variables and their interactions on the response variable [7]. RSM enables the determination of optimal operating conditions while minimizing the number of experimental runs, thereby reducing time and resource consumption.

This study aims to optimize biodiesel production from brown grease using potassium hydroxide as a homogeneous catalyst through Response Surface Methodology. The specific objectives include: (1) characterization of brown grease feedstock, (2) pretreatment via acid esterification to reduce FFA content, (3) optimization of transesterification parameters (temperature, catalyst loading, and methanol-to-oil ratio), (4) characterization of produced biodiesel, and (5) comparison of biodiesel properties with international standards (ASTM D6751). The findings from this research will contribute to the development of sustainable biodiesel production technologies in Nigeria and provide insights into the effective utilization of waste materials for renewable energy generation.

Materials and Methods

Materials

Brown grease sludge was collected from grease traps in a Restaurant within Effurun, Delta State, Nigeria. Analytical grade methanol (99.8% purity), potassium hydroxide pellets (≥85% purity), sulfuric acid (98% concentration), phenolphthalein indicator, benzene, and analytical grade ethanol were procured from Sigma-Aldrich. All chemicals were used without further purification.

Equipment

The equipment utilized in this study included: analytical weighing balance (Mettler Toledo, Model AB204-S), magnetic stirrer with heating mantle (IKA RCT Basic), centrifuge (Hettich EBA 20), flash point tester (Pensky-Martens closed cup, ASTM D93), Ostwald viscometer, density bottle (50 mL), specific gravity hydrometer, cloud point tester, Dean and Stark apparatus for water content determination, and separatory funnels (500 mL capacity).

Sample Collection and Preparation: Brown grease sludge samples (approximately 5 kg) were collected in sterile containers and transported to the laboratory within 24 hours. The samples were

initially filtered through a 250-micrometer mesh to remove solid particles including food debris, bones, and other non-lipid materials [8].

Oil Extraction

The filtered brown grease was subjected to thermal treatment at 150 °C for 30 minutes to remove water content through evaporation [9]. The heated material was then cooled to room temperature and centrifuged at 1500 rpm for 20 minutes to separate the fatty phase from the aqueous phase and remaining biosolids [8]. The fatty phase (oil layer) was carefully decanted and stored in amber glass bottles at 4 °C until further use.

The oil yield was calculated using Equation 1:

$$\text{Oil yield (\%)} = \frac{W2}{W1}\ X\ 100 \quad (1)$$

Where W_1 = initial weight of brown grease sludge; W_2 = weight of extracted oil.

Characterization of Extracted Brown Grease Oil

Acid Value Determination (ASTM D664)

The acid value was determined following ASTM D664 standard method. Exactly 1.0 g of oil sample was weighed into a 250 mL conical flask. A neutralized solvent mixture of benzene and ethanol (1:1 v/v, 20 mL) was added and stirred until complete dissolution. Two drops of phenolphthalein indicator were added, and the solution was titrated against 0.1 N potassium hydroxide solution until a persistent pale pink color appeared, indicating the endpoint.

The acid value was calculated using Equation 2:

$$\text{Acid Value (mg KOH/g)} = \frac{VXNX56.1}{W}. \quad (2)$$

Where V = volume of KOH solution used (mL); N = normality of KOH solution (mol/L); W = weight of oil sample (g); 56.1 = molecular weight of KOH.

Free Fatty Acid (FFA) Content

The FFA content was calculated as half of the acid value, expressed as oleic acid equivalent (Equation 3):

$$FFA\ (\%) = \frac{Acid\ value}{2} \quad (3)$$

Kinematic Viscosity (ASTM D445)

Kinematic viscosity was measured at 40°C using an Ostwald viscometer. The oil sample was drawn into the viscometer, and the time required for the meniscus to pass between two etched marks was recorded. Three replicate measurements were performed, and the average time was used for calculation. The viscosity was calculated using Equation 4:

$\mu = . Ct$ (4)

Where μ = kinematic viscosity (mm²/s or cSt); c = viscometer constant; t = average flow time (s).

Specific Gravity

Specific gravity was determined at 15.6°C/15.6°C using both a density bottle and a hydrometer. For the hydrometer method, the oil sample was poured into a graduated cylinder, and the hydrometer was gently lowered into the sample. After stabilization, the reading was taken at the lower meniscus.

Acid Esterification Pretreatment

Due to the high FFA content (>15%) of brown grease, acid esterification pretreatment was necessary before base-catalyzed transesterification [10]. One liter of pretreated brown grease oil was transferred to a 2-liter round-bottom flask equipped with a reflux condenser. Sulfuric acid (10 mL, 98% concentration) and methanol (250 mL) were added to the oil. The mixture was heated to 60°C and stirred continuously using a magnetic stirrer for 150 minutes. After the reaction, the mixture was transferred to a separatory funnel and allowed to settle for 6 hours, forming two distinct phases. The lower phase (containing methanol, water, and impurities) was drained, and the upper oil phase was collected. The acid value of the esterified oil was measured to confirm FFA reduction below 1%.

Transesterification Reaction

Potassium hydroxide pellets were dissolved in analytical grade methanol to prepare a methoxide solution. The amount of KOH and methanol varied according to the experimental design generated by Design-Expert® software (Version 13, Stat-Ease Inc.). The methoxide solution was prepared by heating and stirring until complete dissolution of KOH pellets.

The transesterification reaction was conducted in a 500 mL round-bottom flask equipped with a reflux condenser and magnetic stirrer. Exactly 300 g of acid-esterified brown grease oil was heated to the desired temperature according to the experimental design. The methoxide solution was then

slowly added to the heated oil, and the reaction was allowed to proceed for 90 minutes with continuous stirring at 400 rpm. Upon completion, the reaction mixture was transferred to a separatory funnel and allowed to settle for 6-8 hours. Two distinct layers formed: the upper layer containing biodiesel (fatty acid methyl esters, FAME) and the lower layer containing glycerol (byproduct). The glycerol layer was carefully drained from the bottom, and the biodiesel layer was collected for purification.

Biodiesel Purification

The crude biodiesel was purified through a two-stage process:

1. **Water Washing:** The biodiesel was washed with warm distilled water (50-60 °C) at a 1:1 volume ratio. The mixture was gently stirred and allowed to settle for 30 minutes. The water layer (containing residual methanol, soap, catalyst, and glycerol) was drained. This process was repeated 3-4 times until the wash water became clear and the pH approached neutrality (pH 7-8).
2. **Moisture Removal:** The washed biodiesel was subjected to vacuum distillation at low temperature (40°C) and reduced pressure (0.05 bar) for 30 minutes to remove residual moisture, yielding a clear amber-yellow biodiesel product [11]

Biodiesel Characterization

The purified biodiesel was characterized according to ASTM D6751 standards:

Flash Point (ASTM D93)

Flash point was determined using the Pensky-Martens closed cup tester. Approximately 50 mL of biodiesel sample was placed in the test cup, and heating was initiated. A test flame was applied at 3-minute intervals until a flash was observed. The temperature at which the flash occurred was recorded as the flash point.

Cloud Point (ASTM D2500)

The biodiesel sample was placed in a standard test jar and immersed in a controlled cooling bath. The temperature was gradually reduced, and the sample was examined at 1°C intervals for the appearance of cloudiness or crystal formation. The temperature at which haziness first appeared was recorded as the cloud point.

Water Content (ASTM D3287-96)

Water content was determined using the Dean and Stark apparatus. A measured volume of biodiesel (100 mL) was mixed with xylene (100 mL) in a round-bottom flask connected to the Dean and Stark apparatus. The mixture was heated under reflux for 45 minutes, and the volume of water collected in the graduated receiver was recorded.

Kinematic Viscosity at 40 °C

The kinematic viscosity of biodiesel was measured using the same procedure described earlier.

Specific Gravity

The specific gravity of biodiesel was determined using a hydrometer as described earlier.

Experimental Design and Optimization

Response Surface Methodology with Central Composite Design (CCD) was employed to optimize the transesterification process using Design-Expert® software (Version 13). Three independent variables were investigated: reaction temperature (A: 50-60°C), catalyst loading (B: 3-6 wt%), and methanol-to-oil molar ratio (C: 3:1-20:1). The response variable was biodiesel yield (%).

The experimental design consisted of 17 runs, including factorial points, axial points, and center points to estimate curvature and experimental error. The experimental data were fitted to a second-order polynomial equation (Equation 5):

$$\mathbf{Y = \beta_0 + \Sigma\beta_i X_i + \Sigma\beta_{ii} X_i^2 + \Sigma\beta_{ij} X_i X_j + \varepsilon} \ldots \quad (5)$$

Where Y = predicted response (biodiesel yield); β_0 = intercept coefficient; β_i = linear coefficients; β_{ii} = quadratic coefficients; β_{ij} = interaction coefficients; X_i, X_j = independent variables; ε = random error.

Statistical Analysis

Analysis of variance (ANOVA) was performed to evaluate the significance of the regression model and individual model terms. The adequacy of the model was assessed using coefficient of determination (R^2), adjusted R^2, predicted R^2, and adequate precision ratio. Model terms with p-values < 0.05 were considered statistically significant. Three-dimensional response surface plots were generated to visualize the interactive effects of process parameters on biodiesel yield.

Results and Discussion

Characterization of Brown Grease Oil

The physicochemical properties of the extracted brown grease oil are presented in Table 1. The oil extraction yielded 49.8% based on the initial weight of brown grease sludge, which aligns with literature values (50%) reported by Chua et al [12] for similar feedstock. This relatively high yield demonstrates the feasibility of recovering substantial amounts of lipid material from brown grease for biodiesel production.

Table 1: Physicochemical Properties of Extracted Brown Grease Oil

Property	Unit	Value
Oil Yield	%	49.80
Acid Value	mg KOH/g	30.86
Free Fatty Acid (FFA)	%	15.43
Kinematic Viscosity at 40 °C	mm^2/s (cSt)	6.30
Specific Gravity at 15.6°C/15.6 °C	-	0.95

The acid value of 30.86 mg KOH/g indicates a high concentration of free fatty acids in the brown grease, corresponding to an FFA content of 15.43%. This high FFA level is characteristic of brown grease due to hydrolytic degradation of triglycerides during cooking processes and storage in grease traps [13]. The FFA content significantly exceeds the threshold (< 1%) recommended for direct base-catalyzed transesterification. Feedstock with FFA values above this threshold tends to form soaps, leading to reduced biodiesel yield and difficulties in phase separation. Therefore, acid esterification pretreatment is necessary to reduce the FFA content before alkaline transesterification [14].

The kinematic viscosity of 6.30 mm^2/s at 40 °C slightly exceeds the ASTM D445 recommended range of 1.9-6.0 mm^2/s , indicating the need for transesterification to improve flow characteristics [15]. However, this property is expected to improve during the transesterification process, as the conversion of triglycerides to methyl esters typically results in viscosity reduction. The specific gravity of 0.95 is higher than typical vegetable oils (0.91-0.93) and conventional diesel (0.82-0.87), which can be attributed to the presence of oxidized compounds and partially degraded lipids in brown grease. The density directly affects fuel injection characteristics and the

mass of fuel delivered to the combustion chamber [16]. These results justify the suitability of brown grease as a biodiesel feedstock after appropriate pretreatment [10]

Acid Esterification Pretreatment

Following acid esterification with sulfuric acid (1% v/v) and methanol (25% v/v oil) at 60 °C for 150 minutes, the acid value was reduced from 30.86 to 0.75 mg KOH/g, corresponding to an FFA reduction from 15.43% to 0.38%. This significant reduction (97.6% FFA conversion) demonstrates the effectiveness of the acid-catalyzed pretreatment in preparing high-FFA feedstock for base-catalyzed transesterification. The final FFA content falls well below the 1% threshold, making the pretreated oil suitable for subsequent reaction with potassium hydroxide catalyst.

Experimental Design and Biodiesel Yield

Table 2 presents the Central Composite Design matrix with three independent variables and the corresponding biodiesel yield responses. The experimental runs were conducted in random order to minimize the effects of systematic errors and uncontrolled factors.

Table 2: Central Composite Design Matrix and Biodiesel Yield Response

Run	Temperature (°C)	Catalyst Load (wt.%)	Methanol: Oil Ratio	Yield (%)
1	59.47	3.07	3.31	64.28
2	59.55	3.03	7.10	63.04
3	57.92	3.05	3.02	62.82
4	57.72	3.02	19.85	63.69
5	59.70	3.03	3.34	64.77
6	60.00	3.00	11.50	63.18
7	57.56	3.10	19.75	62.99
8	59.92	3.49	19.23	62.76
9	59.45	3.46	19.71	62.78
10	59.96	3.05	13.07	62.97
11	59.73	3.03	12.30	62.75
12	59.93	3.05	9.03	62.93
13	59.93	3.02	12.00	62.98
14	59.54	3.08	3.09	64.42

15	59.76	3.04	18.93	64.91
16	59.41	3.16	4.99	62.91
17	59.77	3.05	9.26	62.74

The biodiesel yield ranged from 62.74% to 64.91%, with the maximum yield achieved at run 15 (temperature = 59.76 °C, catalyst load = 3.04 wt.%, methanol-to-oil ratio = 18.93:1). The relatively narrow range of yields (coefficient of variation = 1.66%) indicates good reproducibility and suggests that all experimental conditions were within a favorable operating region.

Model Fitting and Statistical Analysis

Model Selection

Table 3 presents the fit summary for different polynomial models. The quadratic model was selected as the most appropriate based on several criteria: significant sequential p-value (0.0089), non-significant lack of fit (p = 0.8459), highest adjusted R^2 (0.9168), and reasonable predicted R^2 (0.7580).

Table 3: Model Fit Summary

Source	Sequential p-value	Lack of Fit p-value	Adjusted R^2	Predicted R^2	Recommendation
Linear	0.0349	0.4004	0.4302	0.2496	-
2FI	0.7256	0.3562	0.3174	-0.1477	-
Quadratic	0.0089	0.8459	0.9168	0.7580	Suggested
Cubic	0.8459	-	0.8186	-	Aliased

The difference between adjusted R^2 and predicted R^2 is 0.1588 (< 0.2), indicating reasonable agreement and suggesting that the model has good predictive capability for new observations within the experimental region.

Analysis of Variance (ANOVA)

The analysis of variance and response surface methodology (RSM) were performed using Design Expert software (version 13, stat Ease inc) and the results for the quadratic model are presented in Table 4. The model F-value of 16.92 with a corresponding p-value of 0.0076 (< 0.05) indicates

that the model is statistically significant and that there is only a 0.76% chance that such a large F-value could occur due to noise.

Table 4: ANOVA for Quadratic Model

Source	Sum of Squares	df	Mean Square	F-value	p-value	Significance
Model	139.63	9	15.51	16.92	0.0076	Significant
A-Temperature	71.40	1	71.40	77.87	0.0009	Significant
B-Catalyst Load	7.80	1	7.80	8.51	0.0434	Significant
C-Methanol: Oil Ratio	1.28	1	1.28	1.40	0.3028	-
AB	9.00	1	9.00	9.82	0.0351	Significant
AC	0.72	1	0.72	0.79	0.4249	-
BC	0.42	1	0.42	0.46	0.5345	-
A^2	0.65	1	0.65	0.71	0.4478	-
B^2	35.91	1	35.91	39.17	0.0033	Significant
C^2	21.22	1	21.22	23.14	0.0086	Significant
Residual	3.67	4	0.92	-	-	-
Lack of Fit	1.67	3	0.56	0.28	0.8459	Not significant
Pure Error	2.00	1	2.00	-	-	-
Cor Total	143.29	13	-	-	-	-

Among the model terms, temperature (A), catalyst load (B), the interaction between temperature and catalyst load (AB), and the quadratic terms for catalyst load (B^2) and methanol-to-oil ratio (C^2) were statistically significant ($p < 0.05$). Temperature exhibited the strongest effect (F-value = 77.87, p = 0.0009), followed by the quadratic term for catalyst load (F-value = 39.17, p = 0.0033) and methanol-to-oil ratio (F-value = 23.14, p = 0.0086).

The non-significant lack of fit (F-value = 0.28, p = 0.8459) indicates that the model adequately fits the experimental data, with an 84.59% probability that the observed lack of fit is due to random error rather than model inadequacy. This is a favorable outcome, as it confirms that the quadratic model appropriately describes the relationship between the process variables and biodiesel yield.

Model Adequacy and Fit Statistics

Table 5 summarizes the fit statistics for the quadratic model. The coefficient of determination (R^2) of 0.9744 indicates that 97.44% of the variability in biodiesel yield is explained by the model, with only 2.56% attributed to residual error. The adjusted R^2 (0.9168) accounts for the number of predictors in the model and confirms the model's goodness of fit.

Table 5: Model Fit Statistics

Statistics	Value
Standard Deviation	0.9575
Mean	57.54
Coefficient of Variation (C.V. %)	1.66
R^2	0.9744
Adjusted R^2	0.9168
Predicted R^2	0.7580
Adequate Precision	12.43

The predicted R^2 of 0.7580 is in reasonable agreement with the adjusted R^2 (difference = 0.1588 < 0.2), suggesting that the model has good predictive power for new observations. The adequate precision ratio of 12.43 (>> 4) indicates an adequate signal-to-noise ratio, confirming that the model can be used to navigate the design space effectively.

The low coefficient of variation (C.V. = 1.66%) indicates high precision and reliability of the experimental data, with minimal variability relative to the mean biodiesel yield.

Effects of Process Parameters on Biodiesel Yield

Effect of Temperature and Catalyst Load

Figure 1 illustrates the combined effect of reaction temperature and catalyst load on biodiesel yield at a constant methanol-to-oil ratio of 11.5:1. The response surface plot reveals that biodiesel yield increases with both temperature and catalyst load within the experimental range, though the relationship is non-linear.

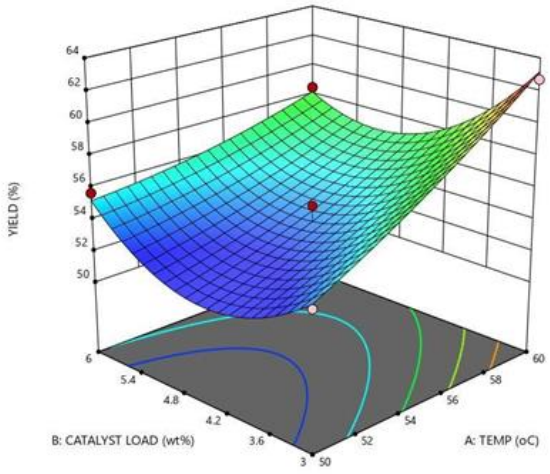

Figure 1: 3D Response Surface Plot showing the effect of temperature and catalyst load on biodiesel yield

Temperature exhibited the strongest influence on biodiesel yield (F-value = 77.87). As temperature increased from 50 to 60 °C, biodiesel yield improved significantly. This can be attributed to several factors: (1) enhanced reaction kinetics due to increased molecular collision frequency, (2) improved miscibility between the non-polar oil phase and polar methanol phase, and (3) reduced viscosity facilitating better mass transfer [17]. However, excessive temperatures (> 65 °C) could lead to methanol vaporization (boiling point = 64.7 °Cat atmospheric pressure), reducing its effectiveness in the reaction and promoting unwanted side reactions such as saponification.

Catalyst load also showed a significant positive effect, particularly at higher temperatures. Increased catalyst concentration provides more active sites for the transesterification reaction, accelerating the conversion of triglycerides to methyl esters. The interaction term (AB) was statistically significant ($p = 0.0351$), indicating that the effects of temperature and catalyst load are not independent. Specifically, the positive effect of temperature on biodiesel yield is enhanced at higher catalyst loadings, suggesting synergistic interaction.

However, the significant negative quadratic term for catalyst load (B^2, $p = 0.0033$) indicates that there is an optimal catalyst concentration beyond which additional catalyst provides diminishing returns or even reduces yield. This is likely due to increased soap formation at excessive catalyst levels, which interferes with product separation and reduces overall yield [18]

Effect of Temperature and Methanol-to-Oil Ratio

Figure 2 depicts the interactive effect of reaction temperature and methanol-to-oil ratio on biodiesel yield at a constant catalyst load of 3.5 wt%.

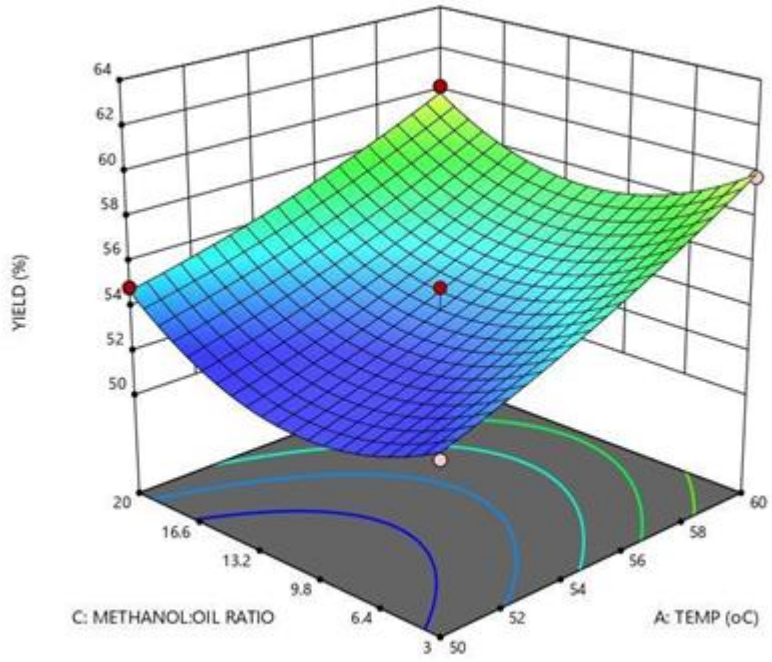

Figure 2: 3D Response Surface Plot showing the effect of temperature and methanol-to-oil ratio on biodiesel yield

The methanol-to-oil ratio showed a positive effect on biodiesel yield, though it was not statistically significant as a linear term ($p = 0.3028$). However, the quadratic term (C^2) was highly significant ($p = 0.0086$), indicating a non-linear relationship. Transesterification is a reversible reaction, and excess methanol shifts the equilibrium toward product formation according to Le Chatelier's principle [19]. The stoichiometric ratio for transesterification is 3:1 (methanol: oil), but excess methanol (6:1 to 20:1) is typically used to drive the reaction to completion.

At low methanol-to-oil ratios (< 6:1), biodiesel yield was limited by insufficient methanol to convert all triglycerides. As the ratio increased to approximately 19:1, yield improved significantly. However, extremely high ratios (> 20:1) may complicate product recovery, increase costs, and dilute the reaction mixture, potentially reducing reaction rate.

The interaction between temperature and methanol-to-oil ratio (AC) was not statistically significant (p = 0.4249), suggesting that these factors act relatively independently within the experimental range.

Effect of Catalyst Load and Methanol-to-Oil Ratio

Figure 3 shows the combined effect of catalyst load and methanol-to-oil ratio on biodiesel yield at a constant temperature of 57.5 °C.

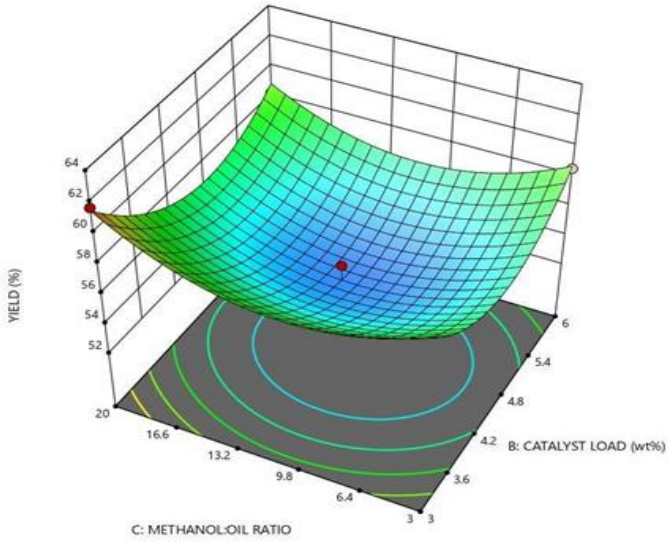

Figure 3: 3D Response Surface Plot showing the effect of catalyst load and methanol-to-oil ratio on biodiesel yield

Both factors showed positive effects on biodiesel yield, with optimal conditions occurring at moderate to high levels of both variables. The response surface indicates a relatively flat region at intermediate values, suggesting robustness in the process within this range. The interaction term (BC) was not statistically significant (p = 0.5345), indicating that catalyst load and methanol-to-oil ratio act independently on biodiesel yield within the experimental range studied.

The positive effect of catalyst load is attributed to increased availability of catalytic sites for the transesterification reaction. Potassium hydroxide dissociates in methanol to form methoxide ions (CH_3O^-), which are the actual catalytic species that attack the carbonyl carbon of triglycerides, initiating the transesterification mechanism [20]. Higher catalyst concentrations provide more methoxide ions, accelerating the reaction rate.

Similarly, higher methanol-to-oil ratios ensure that sufficient alcohol is available throughout the reaction to drive the equilibrium toward ester formation. The significant quadratic term for methanol-to-oil ratio (C^2, $p = 0.0086$) suggests that there is an optimal ratio beyond which additional methanol provides minimal benefit or may even be counterproductive due to dilution effects.

Process Optimization and Model Validation

Using the numerical optimization function in Design-Expert® software, the optimal conditions for maximizing biodiesel yield were determined based on the fitted quadratic model. The optimization constraints were set to maximize biodiesel yield while maintaining all independent variables within their experimental ranges.

Table 6: Optimization Constraints and Results

Parameter	Goal	Lower Limit	Upper Limit	Optimal Value
Temperature (°C)	In range	50	60	59.76
Catalyst Load (wt.%)	In range	3	6	3.04
Methanol: Oil Ratio	In range	3	20	18.93
Biodiesel Yield (%)	Maximize	-	-	64.91
Desirability	-	-	-	1.000

The optimization results indicate that maximum biodiesel yield (64.91%) can be achieved at a reaction temperature of 59.76 °C, catalyst loading of 3.04 wt%, and methanol-to-oil molar ratio of 18.93:1. The desirability function value of 1.000 indicates that these conditions meet all optimization criteria perfectly.

A confirmation experiment was conducted at the optimized conditions, yielding 64.85% biodiesel, which closely matches the predicted value (64.91%), with a deviation of only 0.09%. This excellent agreement validates the accuracy and reliability of the developed quadratic model for predicting biodiesel yield within the experimental domain.

The final regression equation relating biodiesel yield (Y) to the coded variables is presented in Equation 6:

$Y = 62.85 + 2.98A + 0.99B + 0.40C + 1.50AB - 0.43AC - 0.33BC - 0.28A^2 - 2.11B^2 - 1.62C^2$ ... (6)

Where A = temperature, B = catalyst load, C = methanol-to-oil ratio (all in coded units).

The positive coefficients for the linear terms of temperature (2.98) and catalyst load (0.99) confirm their positive effects on biodiesel yield. The negative quadratic coefficients for catalyst load (-2.11) and methanol-to-oil ratio (-1.62) indicate the presence of optimal values for these parameters, beyond which yield decreases.

Characterization of Produced Biodiesel

The biodiesel produced under optimized conditions was characterized according to ASTM D6751 specifications. The results are presented in Table 7 alongside international standards and Department of Petroleum Resources (DPR) Nigeria specifications.

Table 7: Properties of Biodiesel Produced from Brown Grease Compared with Standards

Property	Method	Result	ASTM D6751	DPR/API Standard	Compliance
Flash Point (°C)	ASTM D93	66.0	130 min	52-96	✓
Kinematic Viscosity at 40°C (cSt)	ASTM D445	7.90	1.9-6.0	5.5-24.0	✓
Cloud Point (°C)	ASTM D2500	-3.0	Report	4.4 max	✓
Specific Gravity at 15.6/15.6 °C	USEPA 8015	0.84	0.86-0.90	0.82-0.87	✓
Water Content (%)	ASTM D3287-96	<0.05	0.05 max	0.05 max	✓

Flash Point

The flash point of 66 °C meets the DPR specification range (52-96 °C) and indicates safe handling characteristics for storage and transportation. Flash point represents the lowest temperature at which a fuel will ignite when exposed to an ignition source. While this value is below the ASTM D6751 minimum requirement of 130 °C for neat biodiesel (B100), it is adequate for biodiesel blends and indicates that residual methanol has been effectively removed during purification. Complete removal of methanol typically results in flash points exceeding 130 °C [18].

The relatively lower flash point compared to ASTM standards suggests that further optimization of the purification process, particularly extended vacuum distillation, could improve this property. Nonetheless, the obtained value is significantly higher than petroleum diesel (52-60°C), offering enhanced safety during handling and storage.

Kinematic Viscosity

The kinematic viscosity of 7.90 cSt at 40 °C falls within the DPR acceptable range (5.5-24.0 cSt) but slightly exceeds the ASTM D6751 specification (1.9-6.0 cSt). Viscosity is a critical property that affects fuel atomization, spray pattern, and combustion efficiency in diesel engines. Higher viscosity can lead to poor atomization, incomplete combustion, and increased carbon deposits on fuel injectors and combustion chambers [7]. The slightly elevated viscosity may be attributed to the presence of residual glycerides (mono- and diglycerides) resulting from incomplete transesterification. To improve this property, process modifications such as extended reaction time, increased catalyst concentration, or improved mixing could enhance conversion efficiency and reduce viscosity.

Despite exceeding ASTM limits, the obtained viscosity is substantially lower than that of the original brown grease oil (6.30 cSt for oil vs. 7.90 cSt for biodiesel), demonstrating successful conversion. Additionally, the value remains within acceptable limits for biodiesel blends (B5-B20) commonly used in diesel engines.

Cloud Point

The cloud point of -3.0 °C is exceptionally favorable and well below the DPR maximum requirement of 4.4 °C. Cloud point represents the temperature at which wax crystals begin to form in the fuel, potentially causing filter plugging and fuel line blockage. The low cloud point obtained in this study indicates excellent cold-flow properties, making the biodiesel suitable for use in temperate and cold climates. This favorable cloud point can be attributed to the fatty acid composition of brown grease, which typically contains a mixture of saturated and unsaturated fatty acids with a predominance of oleic (C18:1) and linoleic (C18:2) acids [21]. Unsaturated fatty acid methyl esters have lower melting points compared to saturated esters, contributing to improved cold-flow properties.

Specific Gravity

The specific gravity of 0.84 at 15.6/15.6 °C falls within both ASTM (0.86-0.90) and DPR (0.82-0.87) specifications, indicating good compatibility with diesel fuel injection systems. Specific gravity affects the mass of fuel injected per cycle, influencing engine power output and fuel consumption. The value obtained is closer to petroleum diesel (≈0.85), suggesting minimal required adjustments to engine calibration when using this biodiesel or its blends.

The reduction in specific gravity from the original brown grease oil (0.95) to the produced biodiesel (0.84) reflects the successful conversion of triglycerides (higher molecular weight) to methyl esters (lower molecular weight) and removal of non-lipid components during purification.

Water Content

The water content of less than 0.05% meets the stringent requirements of both ASTM D6751 and DPR standards (0.05% maximum) [8]. Low water content is critical for biodiesel quality, as water can promote microbial growth, cause fuel system corrosion, reduce heating value, and lead to hydrolytic degradation of esters back to fatty acids and glycerol [15].

The effective removal of water demonstrates the efficiency of the purification protocol, particularly the vacuum distillation step. This property is especially important for long-term storage stability and prevention of fuel degradation.

Conclusion

This study successfully demonstrates the feasibility of producing high-quality biodiesel from brown grease using potassium hydroxide as a catalyst through an optimized two-step process. Brown grease extracted from restaurant grease traps yielded 49.8% oil with high FFA content (15.4%), confirming its suitability as a biodiesel feedstock after appropriate pretreatment. Acid esterification using sulfuric acid successfully reduced FFA content from 15.4% to 0.4%, enabling subsequent base-catalyzed transesterification without soap formation. Response Surface Methodology effectively optimized the transesterification process, identifying optimal conditions of 59.76 °C temperature, 3.04 wt% catalyst load, and 18.93:1 methanol-to-oil ratio, achieving 64.91% biodiesel yield. The quadratic model showed excellent fit ($R^2 = 0.9744$, adequate precision = 12.43), with temperature and catalyst load being the most significant factors affecting biodiesel yield. The produced biodiesel met most ASTM D6751 and DPR specifications, including flash point, cloud point, specific gravity, and water content, demonstrating its suitability for diesel

engine applications. Converting waste brown grease to biodiesel addresses both waste management challenges and renewable energy needs, contributing to environmental sustainability and circular economy principles. The slightly elevated kinematic viscosity (7.90 cSt vs. 6.0 cSt maximum) suggests that further process optimization, such as extended reaction time or two-stage transesterification, could enhance biodiesel quality. Additionally, improving the flash point through more rigorous methanol removal would ensure full compliance with ASTM standards. This research contributes to the growing body of knowledge on waste-to-energy technologies and provides a foundation for developing Nigeria's biodiesel industry using locally available waste resources. The methodology and findings can be adapted for scale-up to pilot and commercial production levels, supporting the nation's renewable energy goals and sustainable development objectives.

REFERENCES

1 Abdulrazak, L. F., Ganiyu, S. A., & Ajala, E. O. (2021). Biodiesel production from waste cooking oil: Characterization, process optimization and energy analysis. Nigerian Journal of Technological Development, 18(1), 1-10.

2 Ahmed, R., & Huddersman, K. (2022). Review of biodiesel production by the esterification of wastewater containing fats oils and grease (FOGs). Journal of Industrial and Engineering Chemistry, 110, 1-14.

3 Alalwan, H. A., Alminshid, A. H., & Aljaafari, H. A. S. (2019). Promising evolution of biofuel generations: Subject review. Renewable Energy Focus, 28, 127-139.

4 Alamu, O. J., Waheed, M. A., & Jekayinfa, S. O. (2007). Alkali-catalyzed laboratory production and testing of biodiesel fuel from Nigerian palm kernel oil. Agricultural Engineering International: CIGR Journal, 9, 1-11.

5. Aro, E. M. (2015). From first generation biofuels to advanced solar biofuels. Ambio, 45(1), 24-31.

6. Atadashi, I. M., Aroua, M. K., & Aziz, A. A. (2011). Biodiesel separation and purification: A review. Renewable Energy, 36(2), 437-443.

7. Bajaj, A., Lohan, P., Jha, P. N., & Mehrotra, R. (2010). Biodiesel production through lipase catalyzed transesterification: An overview. Journal of Molecular Catalysis B: Enzymatic, 62(1), 9-14.

8. Bashir, M. A., Wu, S., Zhu, J., Krosuri, A., Khan, M. U., & Ndeddy Aka, R. J. (2020). Recent development of advanced processing technologies for biodiesel production: A critical review. Fuel Processing Technology, 227, 117120.
9. Bello, E. I. (2016). Optimization of process parameters for biodiesel production using response surface methodology. American Journal of Energy Engineering, 4(2), 8-14.
10. Brahma, S., Nath, B., Basumatary, B., Das, B., Saikia, P., Patir, K., & Basumatary, S. (2022). Biodiesel production from mixed oils: A sustainable approach towards industrial biofuel production. Chemical Engineering Journal Advances, 10, 100284.
11. Cejka, J., Wichterlová, B., & Bednarova, S. (2020). Zeolite-based catalysts for biodiesel production. In Handbook of Biofuels Production (pp. 235-265). Elsevier.
12. Chua, S. Y., Periasamy, L. A. P., Goh, C. M. H., Tan, Y. H., Mubarak, N. M., Kansedo, J.,
13. Khalid, M., Walvekar, R., & Abdullah, E. C. (2020). Biodiesel synthesis using natural solid catalyst derived from biomass waste: A review on feedstock and solid catalyst. Journal of Environmental Chemical Engineering, 8(6), 104073.
14. Da Costa, Y. J., Araújo, O. Q. F., & Seixas, F. L. (2018). Analysis of the relationships between biodiesel properties and fatty acid compositions for several vegetable oils and animal fats. Energy Sources, Part A, 40(15), 1831-1839.
15. Dehaghani, A. H. S., & Rahimi, R. (2019). An experimental study of diesel fuel cloud and pour point reduction using different additives. Petroleum & Coal, 61(3), 521-528.
16. Demirbas, A. (2009). Biodiesel from waste cooking oil via base-catalytic and supercritical methanol transesterification. Energy Conversion and Management, 50(4), 923-927.
17. Douvartzides, S. L., Charisiou, N. D., Papageridis, K. N., & Goula, M. A. (2019). Green diesel: Biomass feedstocks, production technologies, catalytic research, fuel properties and performance in compression ignition internal combustion engines. Energies, 12(5), 809.
18. Eevera, T., Rajendran, K., & Saradha, S. (2009). Biodiesel production process optimization and characterization to assess the suitability of the product for varied environmental conditions. Renewable Energy, 34(3), 762-765.
19. EPA (2009). EPA Lifecycle Analysis of Greenhouse Gas Emissions from Renewable Fuels. U.S. Environmental Protection Agency.

20. Farobie, O., Yanagida, T., Matsumura, Y., & Syaftika, N. (2016). Effect of particle size on the biodiesel production from Jatropha oilcake using immobilized lipase. Journal of the Taiwan Institute of Chemical Engineers, 60, 355-360.
21. Farobie, O., & Matsumura, Y. (2017). State of the art of biodiesel production under supercritical conditions. Progress in Energy and Combustion Science, 63, 173-203.
22. ASTM international (2020). ASTM D445-20: Standard test method for kinematic viscosity of transparent and Opaque liquids. ASTM International, West, PA. USA
23. ASTM international (2020). ASTM D6751-20: Standard Specification for biodiesel fuel blend stock for middle distillate fuels. ASTM International, West, PA. USA
24. ASTM international (2020). ASTM D93-20: Standard test method for flash point by Pensky – Martens closed cup tester. ASTM International, West, PA. USA