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Optimization of Biodiesel Production from Waste Vegetable Oil: The Response Surface Methodology Approach

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ABSTRACT

This study investigated the optimization of biodiesel production from waste vegetable oil (WVO) using sodium hydroxide as a homogeneous catalyst, with a focus on enhancing yield and process efficiency. Process parameters including methanol-to-oil ratio, reaction temperature, reaction time, and catalyst loading were systematically optimized to achieve the highest biodiesel yield. The Response Surface Methodology (RSM) with a Box-Behnken Design was employed to model and analyze the interactions among these variables. Optimal conditions were determined as a methanol-to-oil ratio of 6.52:1, a reaction temperature of 60.79 °C, a reaction time of 54.78 minutes, and a catalyst loading of 0.85 wt%. These conditions yielded a biodiesel output of 91.42%, demonstrating significant process efficiency. The quadratic model used for optimization showed high predictive accuracy, with a coefficient of determination (R^2) of 0.97. This research underscores the importance of precise parameter optimization in biodiesel production, offering a pathway for improved cost-effectiveness and reduced waste, while ensuring high-quality biodiesel output from sustainable feedstock.

Keywords: Optimization, biodiesel, coefficient of determination, waste vegetable oil

INTRODUCTION

The global reliance on fossil fuels as the primary energy source has led to widespread environmental degradation, including greenhouse gas emissions, air pollution, and resource depletion [1, 2]. As the world's energy demands continue to rise, the search for sustainable and renewable alternatives has become imperative. Among the emerging renewable energy sources, biodiesel has proven to be a viable, eco-friendly substitute for petroleum-based diesel. Produced from renewable feedstocks such as vegetable oils, animal fats, and waste materials, biodiesel is biodegradable, non-toxic, and contributes significantly to reducing greenhouse gas emissions [1].

Its compatibility with existing diesel engines, without the need for major modifications, further underscores its practicality as a sustainable energy source.

Waste vegetable oil has emerged as a particularly attractive feedstock for biodiesel production due to its cost-effectiveness and environmental benefits. Unlike edible oils, the use of WVO does not compete with food resources, making it a sustainable option. Moreover, utilizing WVO addresses critical waste management challenges, as improper disposal of used cooking oil can lead to significant soil and water pollution [2]. WVO is readily available as a by-product from households, restaurants, and food processing industries, providing a high-potential feedstock for biodiesel production. The economic benefits are also notable, with studies indicating that the use of WVO can reduce biodiesel production costs by up to 70-80% [2, 3]. However, challenges such as the presence of impurities, free fatty acids, and water content necessitate pretreatment steps like filtration and esterification to improve feedstock quality [1].

Achieving optimal biodiesel yield and quality requires precise control and optimization of production parameters. Key variables such as methanol-to-oil molar ratio, reaction temperature, reaction time, and catalyst concentration significantly influence the efficiency of the transesterification process [4]. Response Surface Methodology has been widely adopted as a powerful statistical tool for modeling and optimizing these factors. Specifically, the Box-Behnken Design (BBD) facilitates the exploration of interactions among multiple variables while minimizing the number of experimental runs required [5]. Studies have demonstrated the reliability of BBD in predicting optimal production conditions, with high coefficients of determination (R^2) validating its accuracy [6].

[8]

Many previous studies lacked experimental validation of the optimum conditions or relied on single variable analysis. This study corrects that by using a statistically robust Box-Behnken Design and validating the optimized parameters experimentally, ensuring accuracy and reproducibility. The novelty of this work lies in the integration of response surface methodology and experimental verification with statistical optimization using BBD to achieve a high biodiesel yield from waste vegetable oil. This contributes to knowledge by establishing a reproducible and scalable biodiesel production process from low-cost, sustainable feedstock.

The purpose of this study is to optimize biodiesel production from WVO using sodium hydroxide as a homogeneous catalyst, employing RSM to refine critical parameters. By addressing

key challenges in feedstock preparation and process optimization, the research highlights the potential of WVO as a sustainable and economically viable feedstock. The findings contribute to global efforts to reduce reliance on fossil fuels, promote environmental sustainability, and advance renewable energy technologies [7]. This research supports the development of cleaner energy alternatives and underscores the importance of utilizing waste materials to achieve a circular economy

MATERIALS AND METHODS

Chemicals and reagents

All reagents such as ethylene glycol, sodium hydroxide, potassium hydroxide, sulphuric acid, benzene, ethanol, methanol, phenolphthalein indicator were all analytical grades [2].

Sample collection

The waste vegetable oil was obtained from a food vendor along DLA road, Asaba, Nigeria. It was filtered in order to remove suspended particles from it. After filtration, the oil was heated to remove any impurities such as water.

Characterization of Waste Vegetable Oil

Various tests and measurements were carried out to determine the physical and chemical properties of the oil. These are the flash point, specific gravity, acid value, and viscosity tests, which were conducted to check the eligibility of the oil in biodiesel production [9].

Acid value

The acid value, measured using ASTM D664, indicates the free fatty acid content of the oil. The process involved dissolving the oil sample in a benzene-ethanol mixture and titrating with a 0.05 N KOH solution using phenolphthalein as an indicator [10]

The acid value was calculated using the formula:

$$AV = \frac{MW \times N \times V}{W} \quad (1)$$

Where

MW ≡ Molecular weight of potassium hydroxide (56.1g).

N≡ Normality of potassium hydroxide solution (0.1 N).

V≡ Volume of potassium hydroxide solution used in titration.

W ≡ Weight of oil sample.

The free fatty acid (FFA) was calculated using the formula in Equation (2)

$$\%FFA = \frac{AV}{2} \quad (2)$$

Peroxide value

This test measures oxidation by quantifying peroxides in the oil. The sample was reacted with potassium iodide (KI), and the liberated iodine was titrated with sodium thiosulfate [11]

The peroxide value was calculated as shown in Equation 2:

$$PV = \frac{(S-B)\times N \times 1000}{weight.of.oil} \quad (3)$$

Where:

S = Sample titre value

B = Blank titre value

N = mol of thiosulphate

Iodine value

The iodine value, which measures unsaturation, was determined by reacting the oil with Hanus iodine solution, then titrating the unreacted iodine with sodium thiosulfate [12]

The formula used is shown in Equation 4:

$$I.V = \frac{126.9 \times c \times (b-v) \times 100}{m \times 1000} \quad (4)$$

Where

c = Normality of sodium thiosulphate ($Na_2S_2O_3$) used;

b = Vol of $Na_2S_2O_3$ used for the blank;

v = Vol of $Na_2S_2O_3$ used for sample;

m = mass of the sample.

126.9 = Equivalent weight of iodine

Specific gravity

The specific gravity was determined by weighing a density bottle filled with oil and comparing it with the weight of the bottle filled with water [13]. The specific gravity was calculated using Equation 5:

$$Sp.gr = \frac{W_1 - W_2}{W_2 - W_0} \quad (5)$$

:

Where

W_0 = weight of dry empty density bottle;

W_1 = weight of density bottle + oil;

W_2 = weight of density bottle + distilled water.

Saponification value

This value indicates the amount of alkali required to saponify the oil. The oil was refluxed with ethanolic KOH and titrated with hydrochloric acid [14].The saponification value was calculated as shown in Equation 6:

$$S.V = \frac{(b - s) \times 56.1 \times n}{w} \quad (6)$$

Where;

b = the volume of the solution used for blank test;

s = the volume of the solution used for determination;

n = Actual normality of the HCl used;

w = Mass of the sample.

Moisture content

The moisture content was determined using the oven-drying method, where the sample was weighed before and after drying [15]. The moisture content was calculated as shown in Equation 7:

$$\%moisture = \frac{W_m - W_d}{W_m} \quad (7)$$

Where;

W_m = weight of moist sample

W_d = weight of dry sample

Esterification of waste vegetable oil

This was done due to the high free fatty acid value (6.109%) found in the oil. Exactly 500 g of WVO in a round bottom flask glass reactor was esterified with 25wt% of methanol using 1.0wt%

H_2SO_4 as catalyst on a constant temperature magnetic stirrer set to heat at a constant temperature 60 ^{0}C for 1.5 hours.

Transesterification reaction

This was conducted in a 250 mL glass reactor with constant stirring at 450 rpm and atmospheric pressure. A mixture of 100 g esterified oil, methanol (23 wt%), and NaOH (1 wt%) was reacted at 60 °C for 60 minutes. Post-reaction, the mixture was separated into Fatty acid methyl ester (FAME) and glycerol layers using a separating funnel. The biodiesel was washed with distilled water and evaporated to yield a clear amber-yellow oil.

Characterization of biodiesel

The biodiesel was characterized to provide information on the quality of biodiesel that was produced. The characterization are as follows;

Flash point determination (ASTM D93)

The flash point of biodiesel was determined using the Pensky Martens Closed Cup method**.** The procedure involved filling the test cup with 75 ml of biodiesel, heating at a controlled rate of 50°C per minute**,** and stirring. An external flame was periodically introduced to the surface of the sample. When the sample ignited, the corresponding temperature was recorded as the flash point temperature [16].

Viscosity determination

The viscosity of biodiesel was measured using a Brookfield NDJ-5S rotary viscometer**.** The steps included; selecting the appropriate spindle number for the test sample, pouring 200 ml of biodiesel into a 250 ml beaker, placing the beaker in a 30°C water bath for 10 minutes to equilibrate, lowering the spindle and temperature sensor into the biodiesel and running the test until a stable viscosity value was obtained and recorded. These tests help assess the safety, handling properties, and engine performance of biodiesel [17].

Response Surface Methodology

The optimization of biodiesel production was carried out using the Response Surface Methodology, employing a Box-Behnken Design on the Design Expert v13.0 software for experimental modeling and process parameter evaluation. The process was designed to identify

the optimal conditions for biodiesel yield, focusing on the following variables: methanol-to-oil molar ratio, catalyst loading, reaction time, and reaction temperature [18].

The BBD approach was selected for its efficiency in analyzing quadratic response surfaces while minimizing the number of experimental runs. The design consisted of three levels (-1, 0, +1) for each parameter, enabling the evaluation of linear, quadratic, and interaction effects on biodiesel yield.

- Methanol-to-Oil Ratio: Varied within the range of 4:1 to 8:1.
- Reaction Temperature: Set between 50°C and 70°C.
- Reaction Time: Adjusted from 30 minutes to 90 minutes.
- Catalyst Loading: Tested within the range of 0.5% to 1.5% weight of oil.

Optimization procedure

Model development: A quadratic model was fitted to the experimental data, with biodiesel yield as the response variable.

ANOVA analysis: Analysis of variance was performed to assess the statistical significance of model terms, using a confidence level of 95% ($p < 0.05$).

Response surface analysis: Three-dimensional response surface plots were generated to visualize the interaction effects among variables.

Validation of optimum conditions: The optimum conditions predicted by the model were experimentally validated to ensure accuracy.

RESULTS AND DISCUSSION

Table 1 shows the physicochemical properties of biodiesel produced from WVO and compares them with ASTM standard values to assess its suitability as a diesel substitute.

Table 1: Biodiesel properties from waste vegetable oil

Properties	Values	Standard ASTM Values
Density @ 30 ^{o}C (g/ml)	0.87	0.88
Viscosity @ 30 ^{o}C (mPa.s)	4.97	1.9 – 6.0
Acid value (mgKOH/g)	0.34	<0.5

Free Fatty Acid (%)	0.17	NA
Flash point (^{o}C)	126	100 – 170
Calorific value (MJ/kg)	40.41	35>

The study assessed biodiesel produced from waste vegetable oil to evaluate its quality and compatibility as an alternative to fossil diesel. The findings, detailed in Table 1, show that the biodiesel aligns with ASTM specifications, ensuring compliance with international standards. Key parameters include a viscosity of 4.97 mPa.s, promoting efficient combustion and minimal engine deposits; a flash point of 126 ^{0}C, ensuring safety during handling and storage; and a specific gravity of 0.87 g/ml, providing optimal energy content and fuel efficiency [16]

The results confirm the produced biodiesel's suitability as a sustainable alternative to conventional diesel. The alignment with ASTM standards highlights it's potential for replacing fossil fuels in various applications without engine modifications. Furthermore, the use of waste vegetable oil as a feedstock demonstrates environmental benefits, offering a clean energy source while mitigating pollution.

The biodiesel yield obtained in this study (91.42%) compares well with previous studies such as Erma Hafiza Ibrahim et al. [24], who reported 90.8%, and Aliozo et al. [25] with 92.3%. The differences in yield can be attributed to variations in feedstock properties, reaction conditions, and catalyst concentration. Our study achieved a slightly higher accuracy due to the optimized methanol ratio and catalyst loading.

Table 2 presents the experimental design matrix generated using the Box-Behnken Design approach. It shows the effects of varying reaction temperature, time, methanol ratio, and catalyst loading on biodiesel yield.

Table 2: Box-Behnken optimization experimental design for transesterification reaction

Runs	A: Reaction Temperature (°C)	B: Reaction Time (minutes)	C: Methanol Ratio	D: Catalyst Loading (wt%)	Biodiesel Yield (wt%)
1	60	50	6	0.9	80.06
2	60	50	6	0.9	92.03
3	40	90	6	0.9	58.03
4	60	10	6	1.4	33.41
5	60	50	8	0.4	83.09
6	40	50	8	0.9	63.98
7	60	50	6	0.9	92.03
8	60	50	4	1.4	34.71
9	60	50	6	0.9	92.35
10	80	50	8	0.9	54.11
11	80	50	4	0.9	43.35
12	80	90	6	0.9	32.46
13	80	10	6	0.9	34.67
14	40	50	4	0.9	45.68
15	60	90	4	0.9	49.54
16	60	90	8	0.9	40.43
17	60	90	6	0.4	71.09
18	60	50	8	1.4	23.17
19	40	50	6	1.4	17.69
20	40	10	6	0.9	27.52
21	80	50	6	0.4	32.74
22	60	50	6	0.9	92.05
23	80	50	6	1.4	43.86
24	60	90	6	1.4	23.53
25	60	50	4	0.4	44.88

26	60	10	4	0.9	26.22
27	40	50	6	0.4	73.28
28	60	10	6	0.4	41.16
29	60	10	8	0.9	66.63

The software generated 29 runs for the four variables inputted. The design was followed and 29 different biodiesel samples were produced, the yields were recorded and inputted back into the software.

Analysis of variance (ANOVA)

Table 3 presents the ANOVA analysis results for the quadratic model, identifying the significance of each variable and their interactions on biodiesel yield.

Table 3: Biodiesel Yield ANOVA for Quadratic model

	Sum of Squares	Df	Mean Square	F-value	p-value	
Model	15244.38	14	1088.88	35.41	< 0.0001	Significant
A-Reaction Temp	168.68	1	168.68	5.49	0.0345	
B-Reaction Time	172.29	1	172.29	5.60	0.0329	
C-Methanol Ratio	631.19	1	631.19	20.53	0.0005	
D-Catalyst Loading	2404.65	1	2404.65	78.20	< 0.0001	
AB	267.65	1	267.65	8.70	0.0105	

AC	14.21	1	14.21	0.4622	0.5077	
AD	1112.56	1	1112.56	36.18	< 0.0001	
BC	613.06	1	613.06	19.94	0.0005	
BD	396.21	1	396.21	12.88	0.0030	
CD	618.77	1	618.77	20.12	0.0005	
A^2	3526.08	1	3526.08	114.66	< 0.0001	
B^2	4435.72	1	4435.72	144.24	< 0.0001	
C^2	1933.01	1	1933.01	62.86	< 0.0001	
D^2	3708.35	1	3708.35	120.59	< 0.0001	
Residual	430.52	14	30.75			
Lack of Fit	314.19	10	31.42	1.08	0.5133	not significant

The Model F-value of 35.41 implies the model is significant. There is only a 0.01% chance that an F-value this large could occur due to noise. Probability values (P-values) less than 0.0500 indicate model terms are significant. In this case A, B, C, D, AB, AD, BC, BD, CD, A^2, B^2, C^2, D^2 are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. If there are many insignificant model terms, not counting those required to support hierarchy, model reduction may improve the model. The Lack of Fit F-value of 1.08 implies the Lack of Fit is not significant relative to the pure error. There is a 51.33% chance that a Lack of Fit F-value this large could occur due to noise. Non-significant lack of fit is good as we want the model to fit [19] Table 4 summarizes the statistical model validation metrics, including R^2 values and adequate precision, confirming the model's reliability in predicting biodiesel yield.

Table 4: Fit Statistics

R^2	0.9725
Adjusted R^2	0.9451
Predicted R^2	0.8730
Adeq Precision	18.6323
Std. Dev.	5.55
Mean	52.20
C.V. %	10.62

The Predicted R^2 of 0.8730 is in reasonable agreement with the Adjusted R^2 of 0.9451; that is, the difference is less than 0.2. Adequate Precision measures the signal to noise ratio. A ratio greater than 4 is desirable. The ratio of 18.632 indicates an adequate signal. This model can be used to navigate the design space (19).

Figure 1 compares the predicted and actual biodiesel yields to validate the model's accuracy.

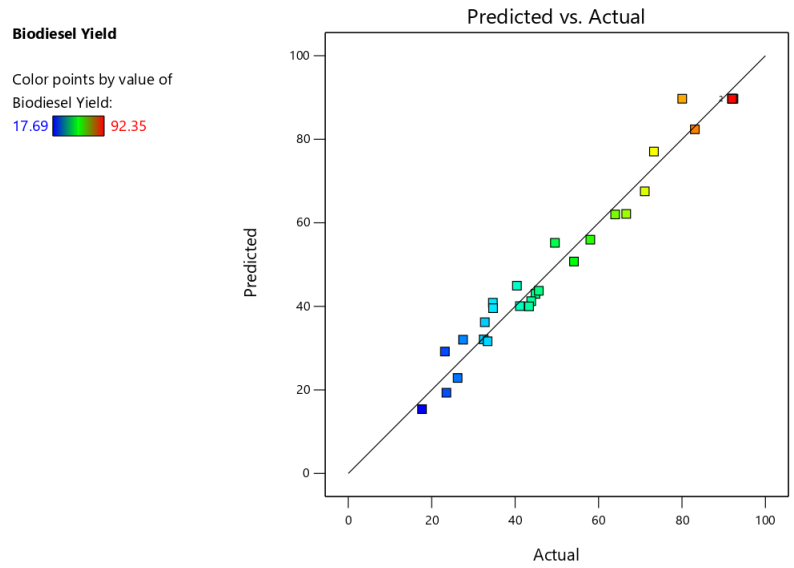

Figure 1: Scatter plot showing the predicted vs. actual values of biodiesel yield

The plot above accesses the accuracy of the predictive model by comparing the predicted biodiesel yields to the experimentally obtained biodiesel yields. A majority of the datapoints are close to the diagonal which indicates that the model performs well in predicting biodiesel yields [20]

Response Surface plots of biodiesel yield

Three-dimensional plots were used in the study to illustrate the response surface methodology (RSM) by displaying the response as a function of two variables while keeping the other variable constant [21].

Figures 2a to 2e illustrate the three-dimensional response surfaces showing how different combinations of process parameters affect biodiesel yield.

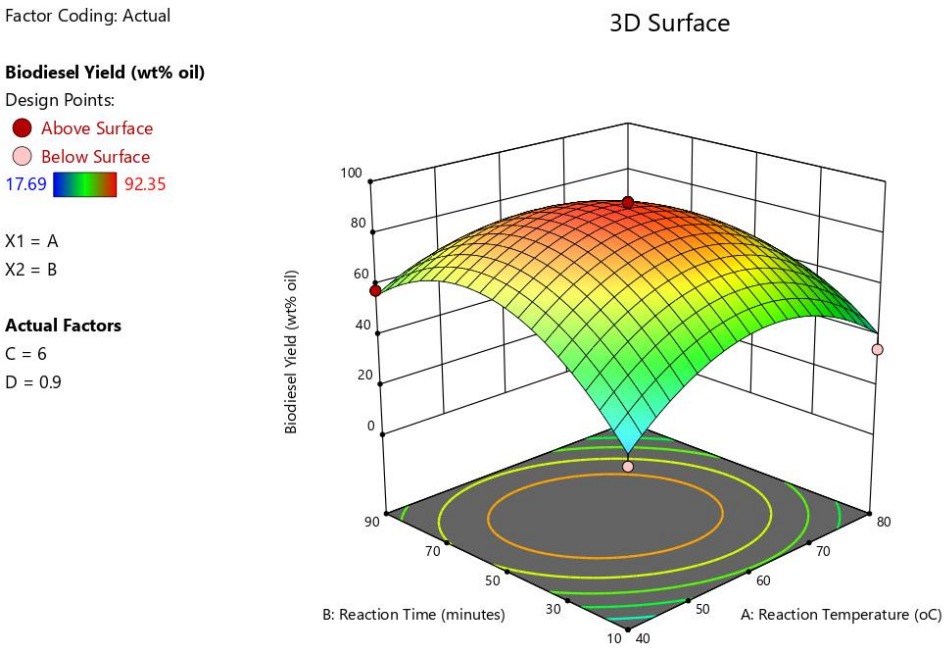

Figure 2a: Effect of reaction time and reaction temperature on biodiesel yield

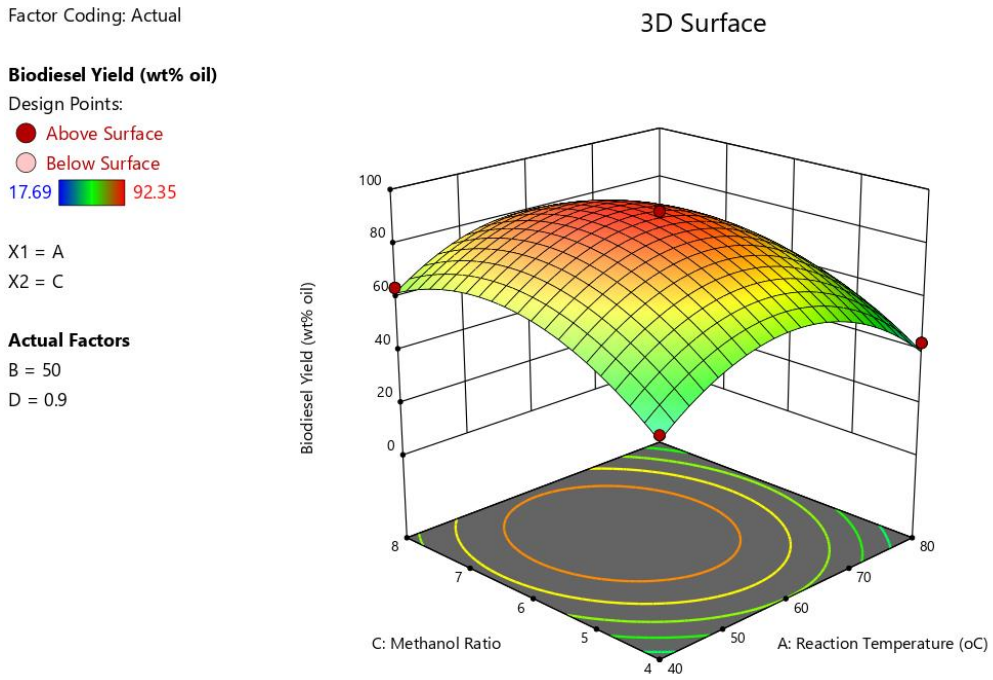

Figure 2b: Effect of methanol ratio and reaction temperature on biodiesel yield

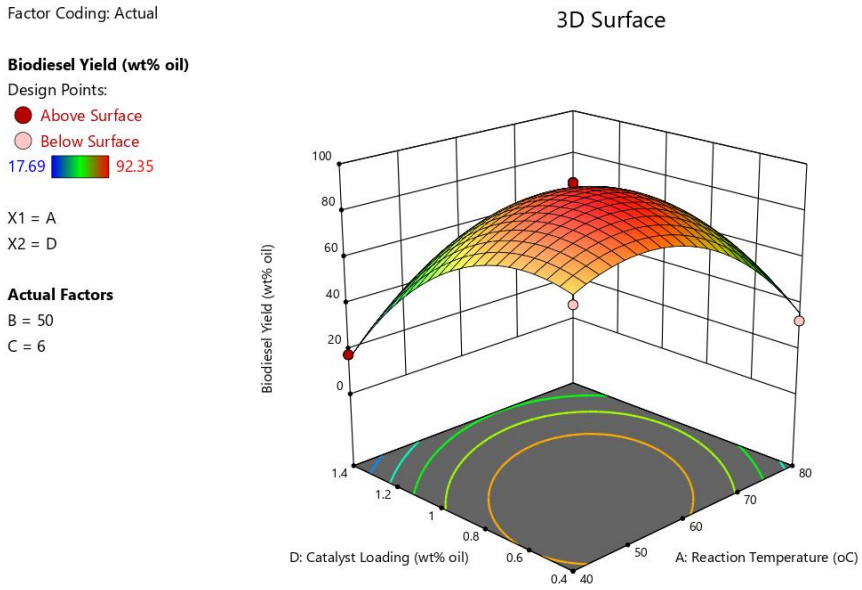

Figure 2c: Effect of catalyst loading and reaction temperature on biodiesel yield

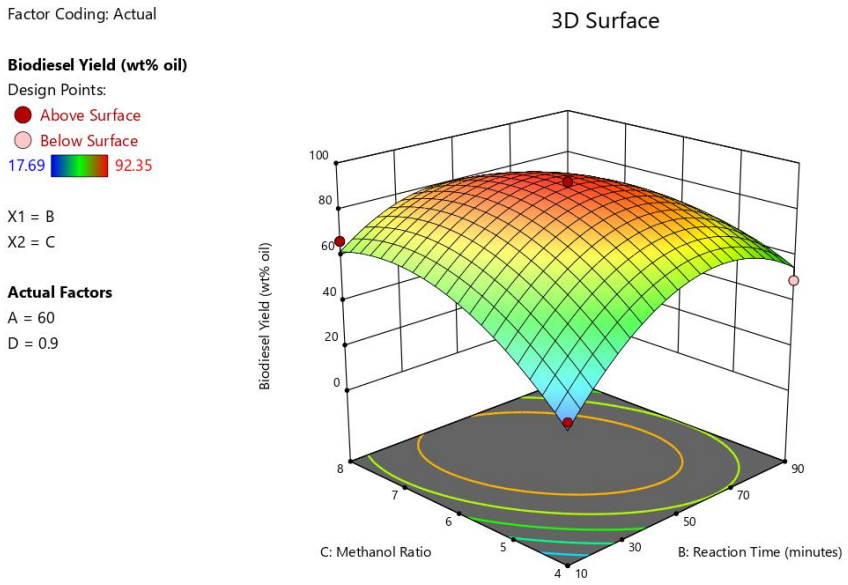

Figure 2d: Effect of methanol ratio and reaction time on biodiesel yield

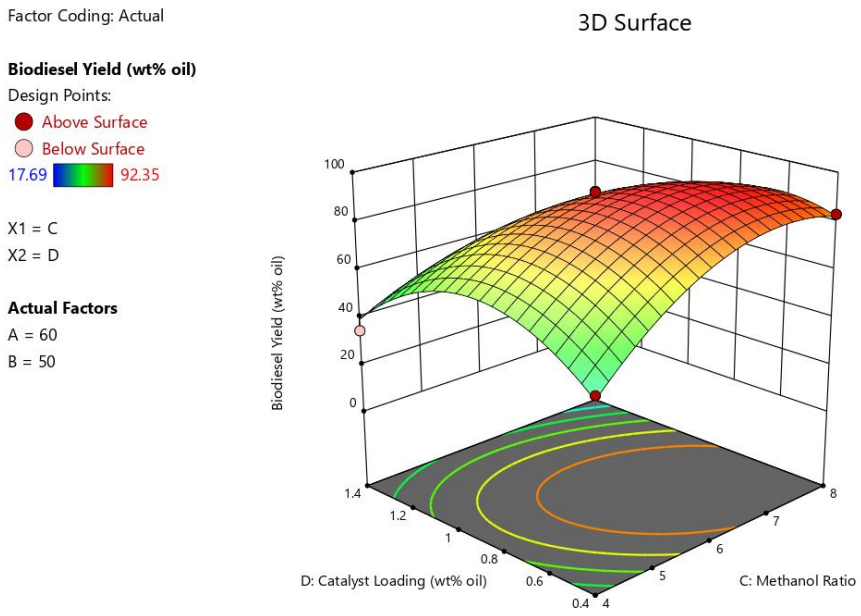

Figure 2e: Effect of catalyst loading and methanol ratio on biodiesel yield.

The results presented in Figures 2a to 2e demonstrate that reaction temperature, catalyst concentration, reaction time, and methanol ratio all had a direct impact on the production of biodiesel. The biodiesel yield in Figure 2a first rises with reaction temperature and time before peaking at a specific location and producing a curved surface. However, the yield starts to decrease

when the ideal parameters are exceeded. At moderate to high reaction temperatures and times, the maximum yield is achieved [21].

The plot in Figure 2b highlights how temperature and the methanol ratio work together, demonstrating the necessity of optimizing both at the same time. As these factors, methanol ratio and reaction temperature, become closer to their optimum values, the yield rises; however, at higher temperatures and with excessive methanol ratios, the yield diminishes. By providing active sites for transesterification, an adequate quantity of catalyst accelerates the reaction. While too much catalyst can cause viscosity or soap production, too little catalyst results in inadequate conversion. High temperatures can improve the process to some degree, but they can also be harmful if they are too high. The graph in Figure 2c makes it plainly evident that optimizing yield requires striking the right balance between these factors. As seen in Figure 2d, effective conversion demands a balance between reaction time and methanol ratio. The methanol ratio and catalyst loading are optimally balanced to maximize yield. As seen in Figure 2e, raising any parameter after this balance may actually reduce the yield rather than increase it.

All of the plots' trends highlight how crucial it is to keep the process parameters for biodiesel synthesis at optimal levels. This helps to direct experimental setups by constraining the ideal parameter range that maximize yield. [22]

Process optimization

As shown in Table 1, the measured biodiesel properties conform to ASTM standards. Table 2 outlines the design of experiments for parameter optimization. Table 3 provides ANOVA results validating model significance. Table 4 lists the fit statistics affirming model predictability. Figure 1 demonstrates the close agreement between predicted and actual yields. Figures 2a to 2e depict interactive effects of variables, and Figure 3 shows the optimal conditions for biodiesel yield.

The optimum production conditions for the FAME synthesis obtained utilizing Box Behnken design in a response surface methodology are illustrated in Fig 3 below. The optimum conditions were as follows: reaction temperature of 60.7873^0C, reaction time of 54.7816 minutes, methanol ratio of 6.52387 (approximately 7:1) and catalyst loading of 0.850367 wt%. The resulting biodiesel yield under these conditions is 91.415%, out of a possible range of 17.69% to 92.35%. This indicates that the conditions chosen lead to a high conversion efficiency from waste oil to biodiesel. [22, 23]

Desirability = 1.000 indicates that the combination of the chosen parameters results in an ideal outcome, ensuring the best possible biodiesel yield and process efficiency.

Solution 83 out of 100 shows that this specific solution is one of many potential combinations, but it ranks high in terms of achieving the optimum result [24].

Figure 3 displays the optimal transesterification conditions predicted by the Box-Behnken Design for maximum biodiesel yield.

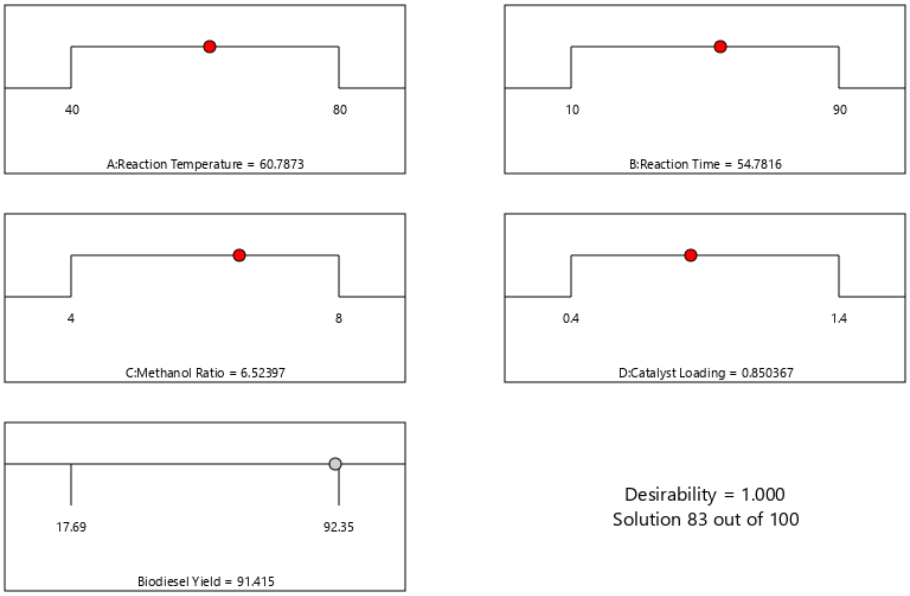

Figure 3: The Optimum Production Condition

As shown in Table 1, the measured biodiesel properties such as viscosity and flash point falls within American society of testing and material (ASTM) standards, confirming quality. Table 2 highlights the 29 experimental runs used for optimizing yield. Table 3 presents ANOVA results, indicating the model's significance. Table 4 provides statistical fit parameters, validating model. Figure 1 shows strong agreement between predicted and actual yields. Figure 2a-2e illustrate variable interactions, while figure 3 visualizes the optimum production conditions.

The maximum biodiesel yield in this study (91.42%) aligns with similar studies by Ermal Haliza et al [24] who reported 90.8%, and Aliozo et al. [25], who achieved 92.3%. The slight difference may be due to variations in feedstock compositions and reactor setup. Compared to Smith et al.

[18], whose yield was below 85%, our improved yield can be attributed to optimized menthanol ratio and catalyst loading [25]

CONCLUSION

This study successfully optimized the production of biodiesel from waste vegetable oil (WVO) using Response Surface Methodology (RSM) and a Box-Behnken Design. The optimal conditions methanol-to-oil ratio of 6.52:1, reaction temperature of 60.79 °C, reaction time of 54.78 minutes, and catalyst loading of 0.85 wt% yielded an impressive biodiesel output of 91.42%, with a strong predictive accuracy validated by a high R^2 value of 0.97.

This research underscores the efficacy of precise parameter optimization in biodiesel production, offering a sustainable and economically viable pathway for transforming waste materials into high-quality biodiesel. By leveraging WVO as a feedstock, the study not only addresses waste management challenges but also contributes to global efforts to mitigate reliance on fossil fuels and promote renewable energy alternatives. The findings provide a robust framework for scaling up biodiesel production processes, ensuring environmental sustainability and economic efficiency in energy development.

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