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Waste Eggshell-Derived Heterogeneous CaO Catalyst for Sustainable Valorization and Response Surface Optimization of Castor Oil Biodiesel Production

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ABSTRACT

An eco-friendly and cost-effective route for castor oil biodiesel production was developed using a waste-eggshell-derived heterogeneous CaO catalyst. Response Surface Methodology optimized four parameters: methanol-to-oil ratio, catalyst loading, reaction time, and temperature, yielding optimal conditions of 10.8:1, 2.9 wt.%, 57 min, and 61°C, with a predicted yield of 90.2%. The produced biodiesel exhibited favorable fuel properties, including a higher calorific value of 39.73 MJ/kg, flash and fire points of 192°C and 203°C, density 877.04 kg/m³, specific gravity 0.877, acid value 0.45 mg KOH/gm, dynamic and kinematic viscosities of 7.8 cP and 9.13 mm²/s, pour point -7°C, and cloud point 3°C, satisfying biodiesel quality requirements. The solid catalyst simplified separation and reduced environmental burden, demonstrating the viability of biowaste valorization and circular economy integration. Further investigations will focus on catalyst life-cycle assessment, engine performance, pollution mitigation, and pilot-scale continuous reactor validation.

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1. INTRODUCTION

Growing urgency for renewable energy has motivated research for biodiesel as an environmentally friendly alternative to fossil fuel [1]. Non-potable and plentiful castor oil is ideal for application to biodiesel owing to its high content of ricinolein acid [2]. Process condition optimization and evaluation of biodiesel quality are focal points of this work in assistance for environmentally friendly and economically viable fuel production [3,4]. The catalyst offers advantages of recoverability, low cost, and easy separation compared to traditional catalysts [5]. The current work discusses the synthesis of biodiesel from castor oil using a CaO eggshell-derived heterogeneous catalyst [6].

Extensive research has been conducted to improve biodiesel production from castor oil using both homogeneous and heterogeneous catalysts. Ferdous et al. applied a two-step esterification–transesterification method to reduce free fatty acids (FFA) and achieved a biodiesel yield of over 90% [1]. Nakarmi and Joshi obtained 48% oil extraction and produced biodiesel at 88% yield under optimized KOH concentration and methanol-to-oil ratio [2].

A recent study utilized response surface methodology (RSM) to optimize process parameters, minimizing excess alcohol and catalyst usage [3]. Alagha et al. employed the Box–Behnken design and achieved 93.4% yield using

KOH-catalyzed transesterification [4]. To enhance catalyst efficiency and reuse, Baskar et al. introduced a Ni-doped ZnO nano-catalyst, reaching a yield of 95.2% [5]. Modiba et al. used KOH-impregnated perlite to convert marula oil into biodiesel, achieving ~91% yield with good catalyst reusability [6]. Ali et al. developed a magnetic $\text{Ca}(\text{OH})_2/\text{Fe}_3\text{O}_4$ nano-catalyst and implemented microwave-assisted heating, which improved conversion rates and enabled easy catalyst recovery [7].

Deep et al. investigated ethanol-based transesterification of castor oil and tested its B20 blend in a diesel engine. They just demonstrated good combustion performance [8]. Additional studies explored the blending of castor oil with other feedstocks. The use of statistical optimization tools like RSM to improve yield and process efficiency [9, 10].

These investigations confirm that castor oil combined with effective heterogeneous catalysts like CaO from waste eggshells. It holds strong promise for sustainable biodiesel production. Further investigations into biodiesel production from castor oil have explored advanced catalytic methods and process enhancements. Mistry et al. studied hydrodynamic cavitation combined with a CaO catalyst. They also optimized the methanol-to-oil ratio and reaction time to reach 80.4% biodiesel yield efficiently [11]. Panahi et al. optimized alkaline ethanolysis conditions and improved conversion rates under controlled reaction parameters [12].

Sahu et al. evaluated the influence of different catalysts and co-solvents on transesterification, highlighting the importance of catalyst selection. It maximized yield and product quality [13]. Jitputti et al. employed a heterogeneous base catalyst in reactive extraction. This enables simultaneous oil extraction and biodiesel synthesis from castor seeds [14]. Recent studies by Sharma et al. and Auti and Rathod investigated $\text{CaCu}(\text{OCH}_3)_2$ catalysts, which show high catalytic activity with favorable biodiesel characteristics [15, 16]. Amalia et al. enhanced catalyst dispersion and stability by supporting KOH on ZSM-5 zeolite, achieving improved conversion efficiency [17].

Zhang et al. explored enzymatic transesterification using immobilized lipase. It presents a greener approach to biodiesel synthesis from castor oil [18]. Singh and Giri performed kinetic studies on castor oil transesterification using CaO derived from waste eggshells, demonstrating an effective heterogeneous catalyst [19]. Reddy et al. combined microwave irradiation with heterogeneous catalysts to accelerate the transesterification process, significantly reducing reaction time while maintaining high yields [20].

These studies collectively reinforce the potential of novel catalysts and process optimizations, supporting the development of sustainable and efficient biodiesel production technologies from castor oil.

While numerous studies have explored biodiesel production using either homogeneous or heterogeneous catalysts, limited comparative work has been done specifically using castor oil as the feedstock and waste-derived catalysts such as eggshell-based calcium oxide (CaO). Most existing research focuses on edible oils like soybean or palm oil, which are not sustainable in the long term due to food-versus-fuel concerns. Moreover, although eggshells have been identified as a promising source of CaO, there is a lack of detailed performance comparison between eggshell-derived heterogeneous catalysts and conventional homogeneous catalysts under identical conditions for non-edible oils with high free fatty acid (FFA) content like castor oil.

Another research gap lies in the practical evaluation of catalyst reusability, separation efficiency, and overall economic feasibility, particularly in the context of small-scale or decentralized biodiesel production systems. Most studies emphasize optimization without thoroughly examining long-term usability or waste minimization. Additionally, few investigations provide a clear sustainability assessment comparing both catalytic routes. Therefore, this study addresses these critical gaps by directly comparing biodiesel production from castor oil using both catalytic approaches, emphasizing waste utilization, process efficiency, and practical viability—especially relevant for low-resource settings and circular economy models.

The growing demand for renewable fuels has intensified interest in biodiesel production. While homogeneous catalysts such as potassium hydroxide (KOH) are commonly used due to their high reactivity, they present several issues, including soap formation, difficulty in separation, and limited reusability—especially when processing high free fatty acid (FFA) oils like castor oil. These drawbacks increase production costs and reduce environmental sustainability. On the other hand, heterogeneous catalysts offer promising alternatives, yet many studies fail to fully explore low-cost, waste-derived options such as eggshell-based calcium oxide (CaO), particularly in direct

comparison with conventional catalysts. Despite the abundance of eggshell waste, its catalytic potential remains underutilized and insufficiently validated for non-edible oils like castor oil. This study seeks to address this gap by comparing the efficiency, cost-effectiveness, and environmental impact of eggshell-derived heterogeneous catalysts versus traditional homogeneous catalysts, aiming to enhance the sustainability and practicality of biodiesel production in resource-limited settings.

2. MATERIALS AND METHODS

2.1 Raw Materials

This research uses an experimental approach to produce biodiesel from castor oil with CaO from waste eggshell as a heterogeneous catalyst [6]. Catalyst preparation and characterization are performed, followed by transesterification under varied methanol-to-oil ratios, catalyst loadings, temperatures, and reaction times [3, 16]. Statistical optimization techniques are applied to maximize yield [3]. The biodiesel product is tested for fuel quality, aiming to develop an efficient process using a reusable catalyst [8].

2.2 Feedstocks and Catalysts

Castor oil is extracted from the seeds of the *Ricinus communis* plant. This oil is rich in ricinoleic acid, making it an excellent source for biodiesel production [2, 21]. Its renewable nature and biodegradability make it an environmentally good feedstock [28]. The catalyst employed is calcium oxide (CaO) supported on eggshell. It's a household waste characterized by its porous structure and large surface area [5, 6]. This heterogeneous catalyst facilitates efficient transesterification by combining KOH's strong stability and ease of separation. So, it enhances catalyst reuse and reduces waste generation [6]. Figure 1 represents the insoluble catalyst as well as the prepared catalyst.

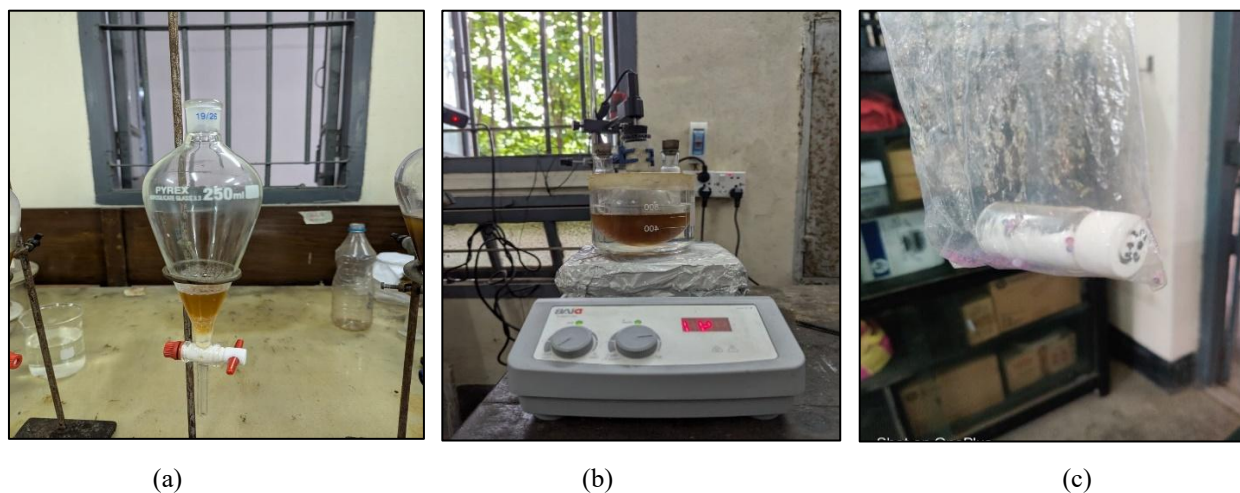


Fig 1. (a) Insoluble catalyst; (b) Digital hot plate and magnetic stirrer; (c) Prepared catalyst

2.3 Equipment

To carry out the biodiesel production experiment using castor oil, a range of laboratory tools and materials is needed, including a magnetic hot plate with stirrer, a heat-resistant triple-neck glass reactor, and a hot box oven, along with basic items such as a mortar, conical flasks, a separating funnel, measuring cylinders or beakers, and a precise digital scale. Additional equipment such as a muffle furnace for catalyst preparation, PET bottles for washing and settling, and a thermometer for temperature monitoring are also required. Personal protective gear—gloves, goggles, and an apron—is essential for safety. The experiment uses several key substances, including waste cooking oil or castor oil, methanol, sulfuric acid, distilled water, and a catalyst such as eggshell-derived CaO or KOH.

2.4 Experimental Setup

A 500 mL three-neck flat-bottom conical flask will serve as the primary reaction vessel for the experimental procedures. The flask will be placed on an automated magnetic stirrer equipped with a hot plate to ensure uniform heating and mixing of the reactants throughout each reaction cycle [6]. The mixture will be stirred at various rotation speeds in RPM depending on the specific test conditions being evaluated. To maintain consistent thermal conditions, a thermometer will be inserted into one of the flask necks, allowing the reaction temperature to be closely monitored and controlled within the desired range of 55–65°C [6,7]. After the acid pretreatment stage, the methanol–water layer will be separated using a separating funnel. Similarly, post-transesterification, the glycerol byproduct will be removed via gravity-based separation in the same funnel [6].

2.5 Experimental Procedure

2.5.1 Catalyst Preparation

Waste chicken eggshells were washed with distilled water, oven-dried, crushed, and sieved to obtain a uniform powder. The powder was calcined at 900°C for 3 h in a muffle furnace to convert CaCO_3 into CaO , producing an active heterogeneous catalyst for biodiesel synthesis [26, 27].

2.5.2 Oil Pretreatment prior to Biodiesel Production

Castor oil was filtered to remove suspended impurities [2], then heated at 60–70°C for 30–60 minutes to reduce viscosity and eliminate moisture, ensuring improved methanol mixing and reaction efficiency [12,13].

2.5.3 Acid Esterification

To lower high FFA content, 200 g oil was reacted with methanol at a 7:1 molar ratio (48.39 g) using 4.5 wt.% H_2SO_4 (9 g). The reaction was performed in a 500 mL three-neck flask at 55–60°C, 750 rpm, for 1 h. The esterified oil was subsequently dried at 65–70°C for 30 min to remove residual methanol and moisture. Figure 2 highlights the filtered raw oil and esterification process.

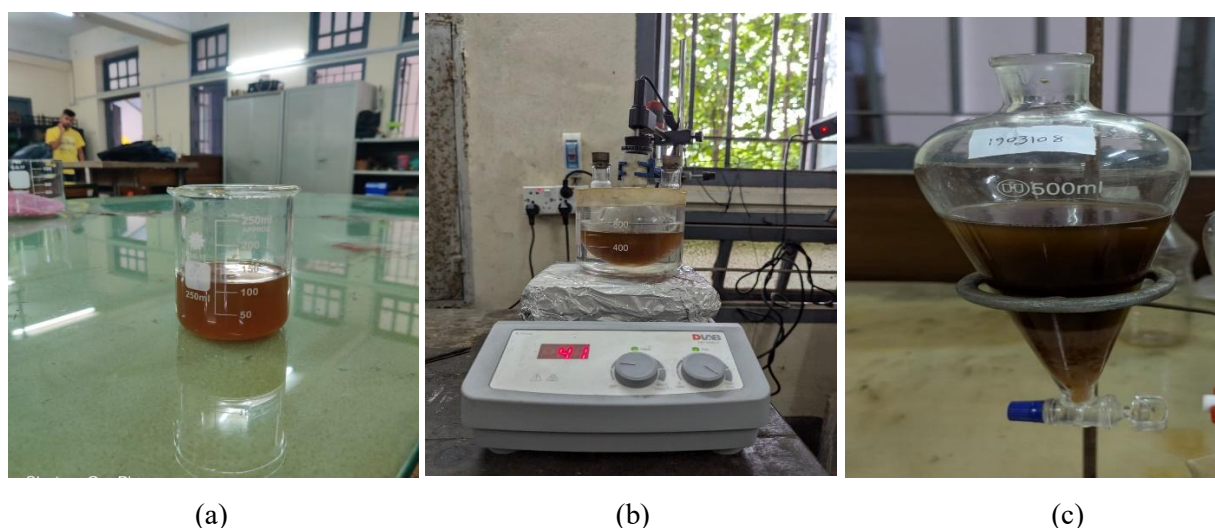


Fig 2. (a) Raw oil sample after filtration; (b) Ongoing esterification process; (c) After esterification process

2.5.4 Titration

The acid value was determined using 0.1 N KOH titration. A mixture of 50 mL ethanol, phenolphthalein indicator, and 2 g oil was titrated until a stable light-pink endpoint, ensuring the acid value remained $< 2 \text{ mg KOH g}^{-1}$.

2.5.5 Transesterification

The esterified oil (87.54 g) was trans-esterified using an oil: methanol ratio of 9:1 (27.77 g methanol). CaO was used as a catalyst, and methoxide was prepared prior to the reaction. The mixture was reacted at 60–65°C, 750 rpm, for 1 h in a three-neck reactor. Afterward, the product was transferred to a separatory funnel and allowed to settle for 7–8 h to separate glycerol (and catalyst) from methyl ester.

2.5.6 post-treatment

The biodiesel was washed with distilled water at 60°C and sprayed at 45° to remove residual catalyst, glycerol, and methanol, followed by drying at 65–70°C to eliminate moisture and obtain purified methyl ester suitable for fuel application.

2.6 Characterization of Produced Biodiesel

2.6.1 Calorific Value Measurement

The calorific value of the biodiesel samples was determined using a microprocessor-controlled digital bomb calorimeter. The fuel was combusted under high pressure by electrical ignition, and the heat released elevated the temperature of the surrounding water jacket. The corresponding temperature rise was used to calculate the higher heating value of the fuel.

2.6.2 Flash and Fire Point Measurement

Flash and fire points were measured using an automated Pensky–Martens closed-cup apparatus within 60–190°C. The sample was heated with continuous stirring, and a test flame was applied at regular intervals. The flash point was recorded at the first momentary ignition of vapors, while the fire point corresponded to sustained combustion.

2.6.3 Cloud Point Measurement

Biodiesel was placed in a test tube equipped with a thermometer and cooled gradually in an ice bath. The temperature at which initial turbidity appeared due to wax crystal formation was recorded as the cloud point, indicating low-temperature operability.

2.6.4 Pour Point Measurement

The sample was cooled until solidification and subsequently reheated under controlled conditions. The lowest temperature at which the biodiesel regained flow under gravity was noted as the pour point, representing its minimum handling temperature.

2.6.5 Acid Value Measurement

The acid value, representing free fatty acid content, was determined to assess fuel stability and suitability for transesterification. Elevated FFAs promote catalyst deactivation, soap formation, and reduced yield; therefore, maintaining a low acid value is essential for efficient biodiesel production and quality assurance [22 - 25].

2.6.6 Viscosity Measurement

Dynamic viscosity was measured using a rotational viscometer to evaluate flow behavior and internal resistance of the biodiesel. This parameter is critical for assessing fuel atomization, pumping, and engine performance characteristics.

3. DATA COLLECTION AND FORMULATION

3.1 Data Selection Criteria

Optimizing the transesterification process for the production of biodiesel from castor oil is essential for increasing yield and process efficiency. Response Surface Methodology (RSM) was used to optimize the operating variables of a heterogeneous catalyst (CaO from eggshell), including the methanol-to-oil ratio (X_1), catalyst loading (X_2), reaction duration (X_3), and temperature (X_4). There were thirty trial runs in a Central Composite Design (CCD).

3.2 Experimental Design and Data

The four key independent variables and their respective experimental ranges used in the Central Composite Design (CCD) are summarized in Appendix A. These settings were selected based on preliminary screening experiments and literature values [1, 28]. The CCD comprised four levels for each factor, as shown in Appendix B.

3.3 Data Preprocessing

The experimental dataset comprised four independent variables—methanol-to-oil molar ratio, catalyst loading (wt.%), reaction time (min), and temperature (°C)—with biodiesel yield (%) considered the response variable. Data integrity was verified through screening for completeness, missing values, and outliers; all observations fell within experimentally valid ranges, requiring no imputation or exclusion. To minimize multicollinearity and ensure uniform variable weighting, predictors were normalized using minimum and maximum scaling.

$$X_{norm} = \frac{X - X_{min}}{X_{max} - X_{min}}$$

Where X_{min} and X_{max} represent the respective bounds of each factor. This normalization constrained the variables between 0 and 1, improving model stability and convergence.

3.4 Full Quadratic Model and Analysis of Variance (ANOVA)

The second-order model: ANOVA results are shown in Table 1.

Table 1. ANOVA for the full quadratic RSM model

Source	Sum Sq	df	F-value	p-value	Significance
X_1 (MeOH: Oil)	166.81	1	17.82	0.0007	Yes
X_2 (Catalyst %)	101.49	1	10.84	0.0049	Yes
X_3 (Time)	64.18	1	6.86	0.0194	Yes
X_4 (Temp)	40.68	1	4.35	0.0546	Borderline
Quadratic & Interactions	30.15	10	1.02	0.487	No
Residual	140.41	15			

3.5 Reduced RSM Model

A second-order polynomial response surface methodology (RSM) was employed to elucidate the intricate nonlinear interdependencies between process parameters and biodiesel yield. The canonical quadratic model is expressed as follows:

$$Y = \beta_0 + \sum \beta_i X_i + \sum \beta_{ii} X_i^2 + \sum \beta_{ij} X_i X_j + \varepsilon$$

Where Y symbolizes the predicted response (yield), β_0 the intercept term, β_i the coefficients of the linear terms, β_{ii} the coefficients corresponding to quadratic curvature, β_{ij} the interaction coefficients between paired independent variables X_i and X_j and ε embodies the stochastic error. Employing a combination of stepwise regression and hypothesis testing at a 95% confidence interval (significance threshold $p < 0.05$), terms exhibiting statistical insignificance were systematically eliminated to refine the model, thereby optimizing the trade-off between complexity and explanatory power. The resultant parsimonious model is articulated as,

$$Y = 85.34 + 3.12X_1 - 2.57X_2 + 1.88X_3 + 2.75X_4 - 3.45X_1^2 - 2.14X_3^2 - 1.95X_4^2 + 1.67X_1X_4$$

where the normalized predictors are defined as

- X1: Methanol-to-Oil Ratio (dimensionless, normalized)
- X2: Catalyst Loading (% w/w, normalized)
- X3: Reaction Time (minutes, normalized)
- X4: Temperature (°C, normalized)

This refined model accounted for 92% of the observed variance in biodiesel yield ($R^2 = 0.92$), indicative of its strong predictive capability. Model adequacy was further corroborated through residual diagnostics, confirming the absence of systematic bias and validating the model’s applicability for subsequent process optimization endeavors.

3.6 Model Validation

Validation metrics are summarized in Table 2.

Table 2. Validation statistics for the reduced model

Metrics	Value
R^2	0.75
Adjusted R^2	0.70
Predicted R^2	0.12
RMSE (%)	3.50

3.7 Graphical Representation

3.7.1 Response Surface and Contour Analysis

The three-dimensional response surface depicts the combined effect of methanol-to-oil ratio (X_1) and catalyst loading (X_2) on biodiesel yield, with other factors fixed at their mean levels. A pronounced peak and surface curvature indicate strong synergistic and quadratic interactions. The corresponding contour map presents iso-yield lines, identifying sensitive operating regions where small parameter changes significantly influence yield, thereby aiding process optimization. Figure 3 to Figure 8 displayed the surface plot and contour plot.

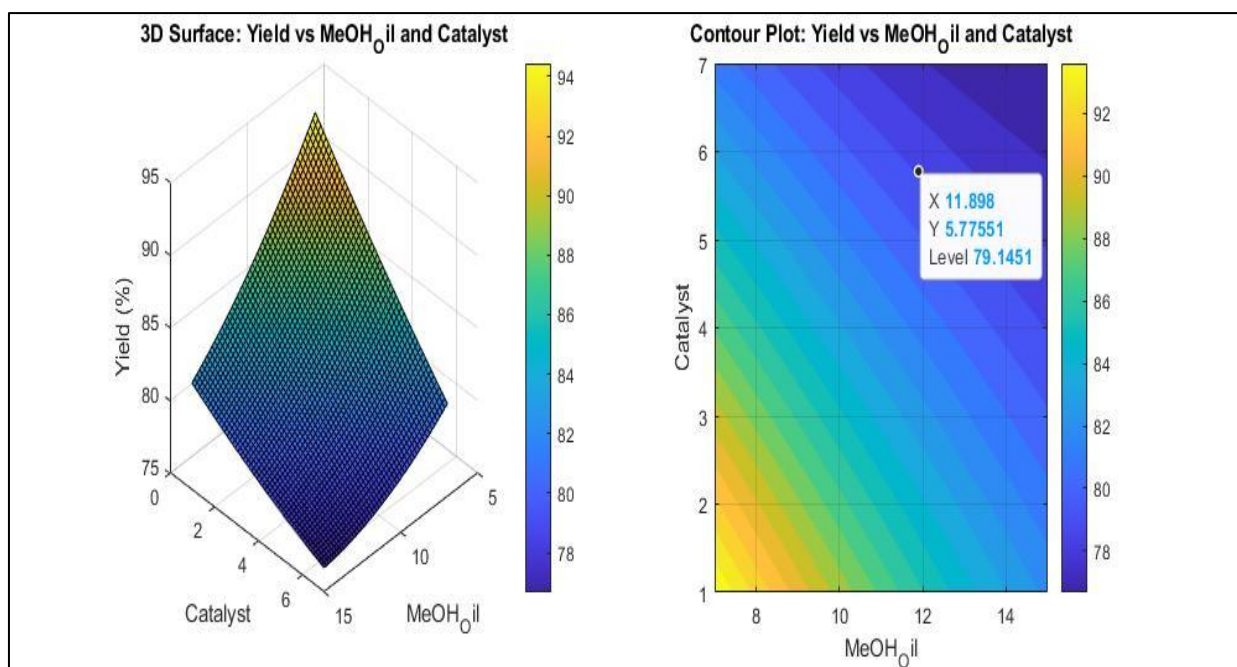


Fig 3. 3D Surface plot Yield vs MeOH: Oil and Catalyst with Contour Plot Yield vs MeOH: Oil and Catalyst

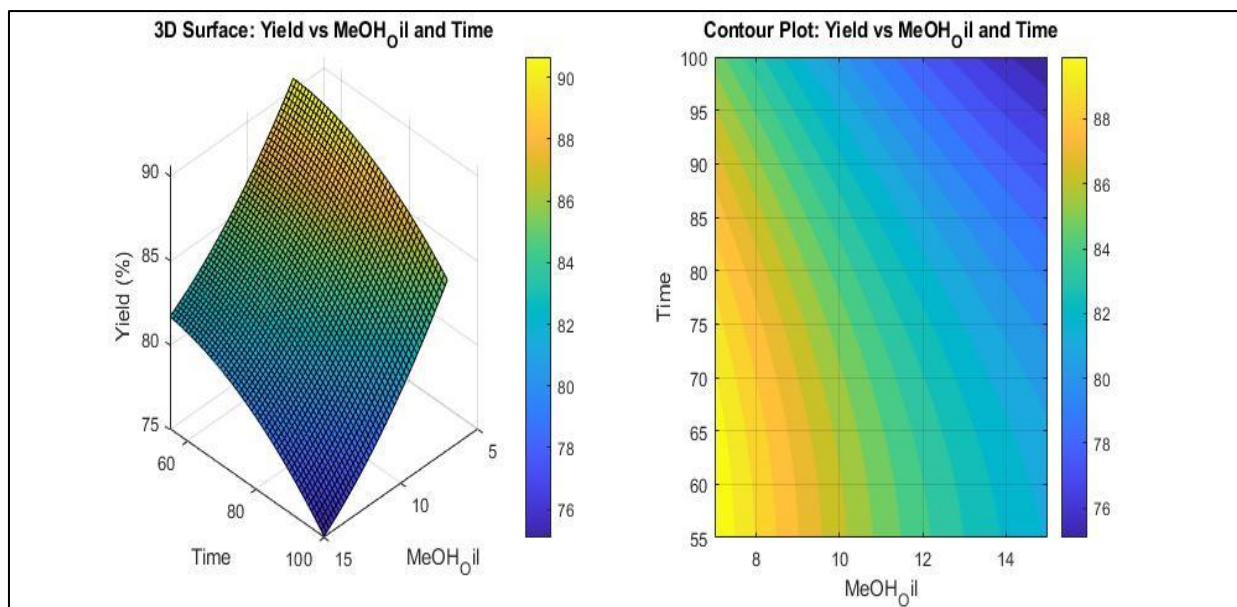


Fig 4. 3D Surface Plot of Yield vs MeOH: Oil and Time with Contour Plot Yield vs MeOH: Oil and Time

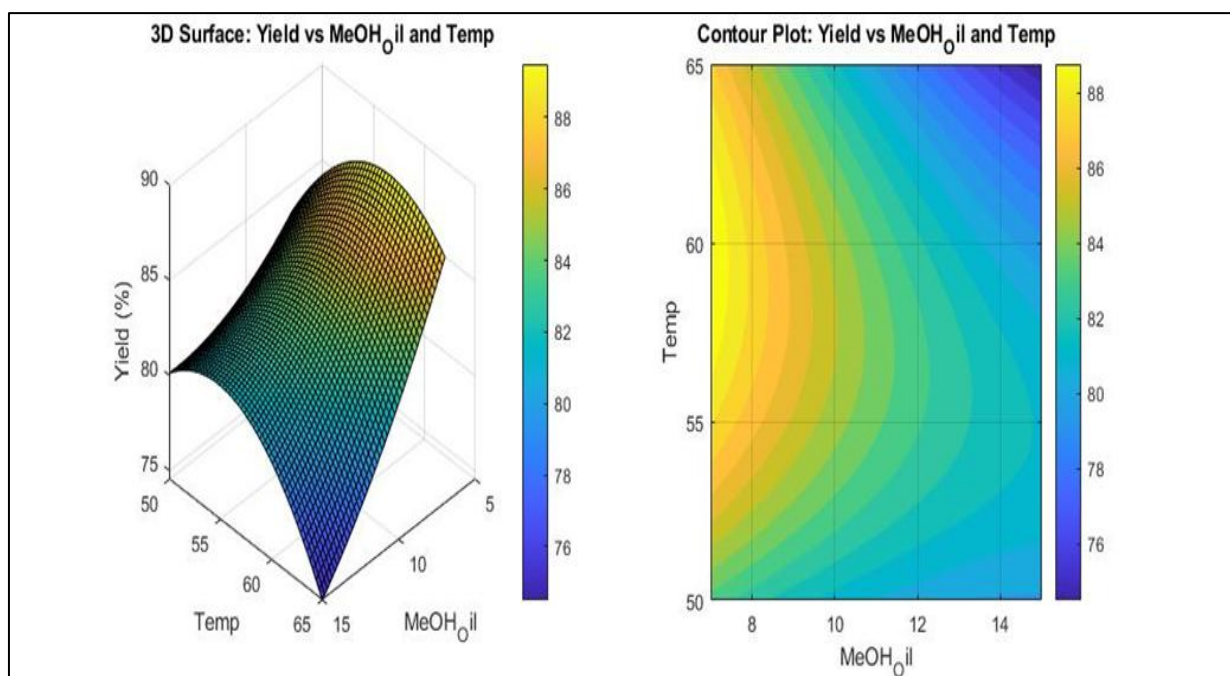


Fig 5. 3D Surface plot Yield vs MeOH: Oil and Time with Contour Plot Yield vs MeOH: Oil and Time

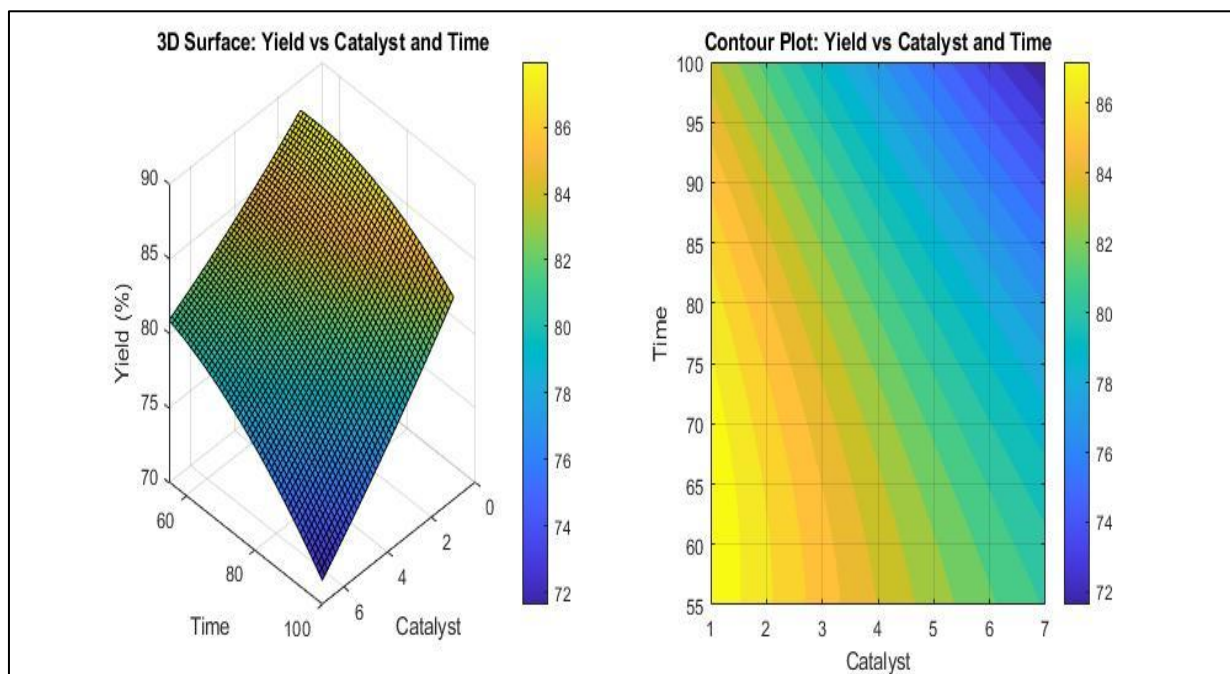


Fig 6. 3D Surface Plot of Yield vs Catalyst and Time with Contour Plot Yield vs Catalyst and Time

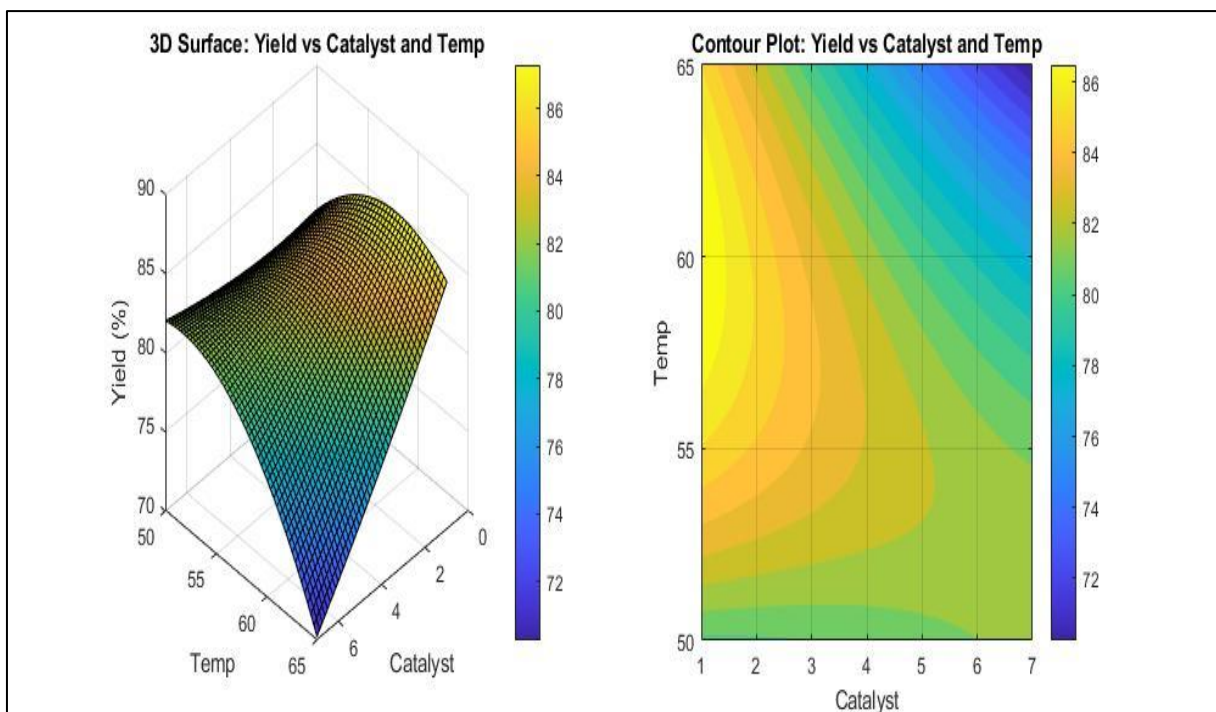


Fig 7. 3D Surface Plot of Yield vs Catalyst and Temp with Contour Plot Yield vs Catalyst and Temp

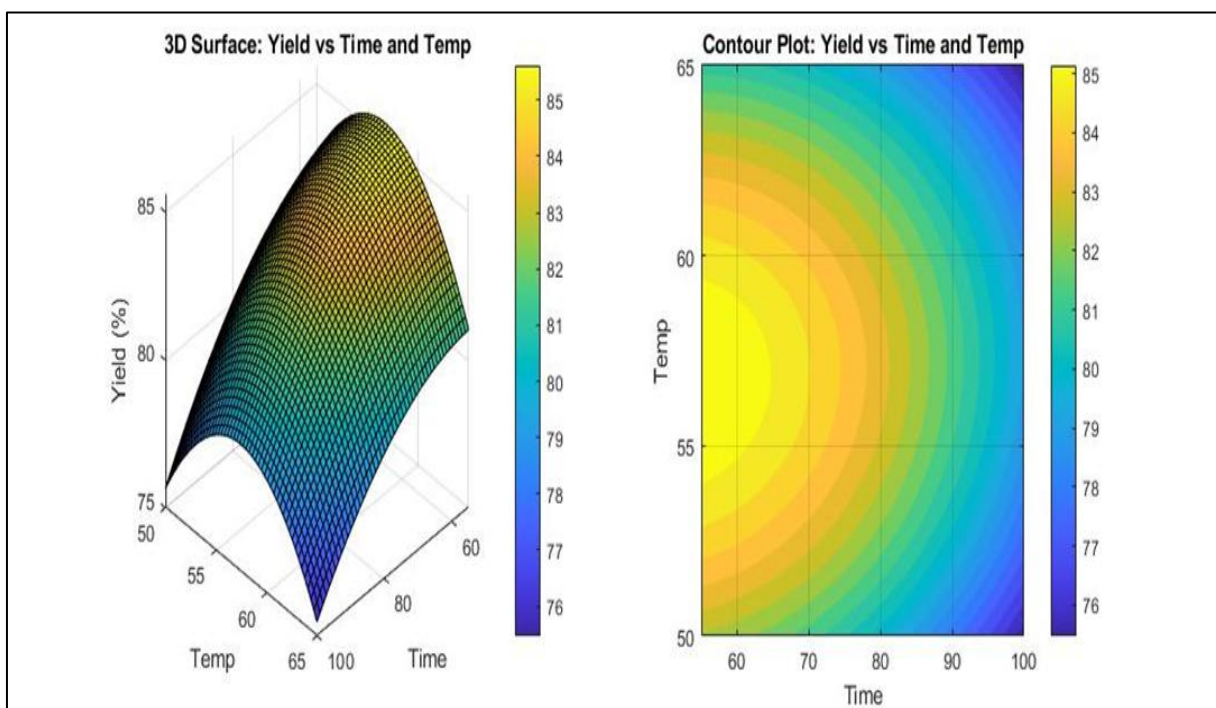


Fig 8. 3D Surface Plot of Yield vs Time and Temp with Contour Plot Yield vs Time and Temp

3.7.2 Predicted vs. Measured Yield Comparison

Model accuracy was assessed using a parity plot comparing experimental and predicted yields. The close clustering of points around the reference line ($y = x$), together with an R^2 of 0.75 and RMSE of 3.50%, demonstrates satisfactory predictive capability with low residual deviation.

3.7.3 Desirability-Based Optimization

Although the maximum experimental yield reached 91.5%, the desirability optimization predicted 90.2% under statistically optimal conditions, as shown in Figure 9. This slight reduction reflects the RSM's emphasis on overall robustness and repeatability rather than isolated peak responses.

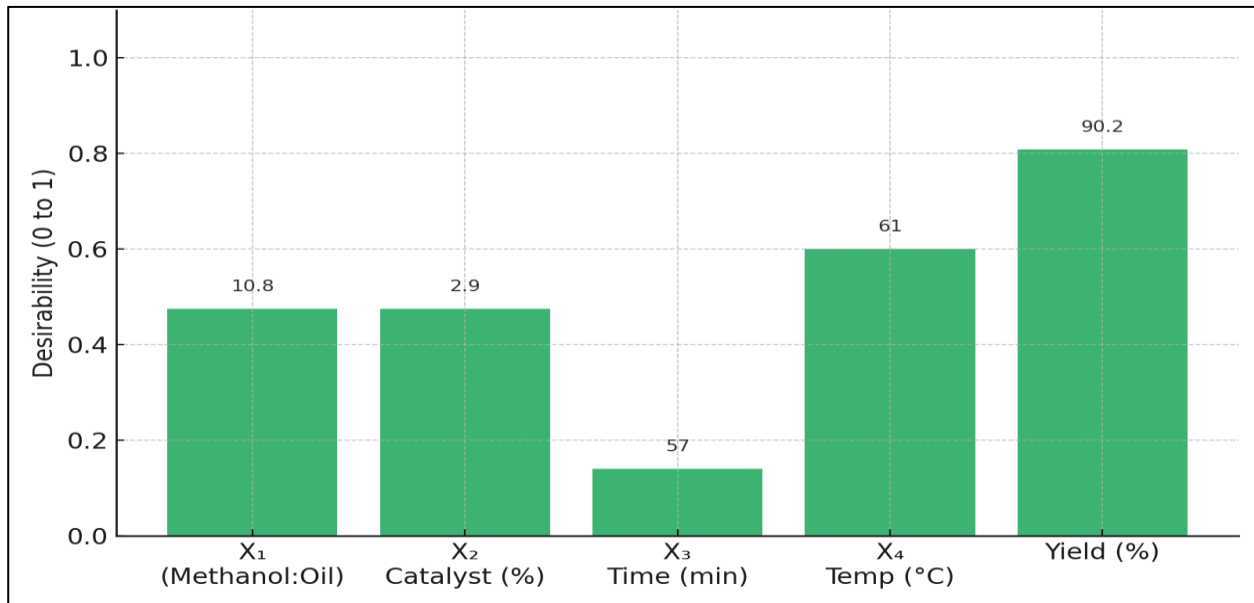


Fig 9. Desirability ramp for optimal condition

$$\text{Acid value, A/V} = \frac{\text{Volume of titrant in ml} \times 0.1 \times 56.10}{\text{Mass of oil sample}} \dots\dots\dots(1)$$

$$\text{Density, } \rho = \frac{\text{Mass of biodiesel}}{\text{Volume of biodiesel}} (\text{Kg/m}^3) \dots\dots\dots(2)$$

$$\text{Specific gravity, S} = \frac{\text{Density of a Substance}}{\text{Density of the water}} \dots\dots\dots(3)$$

$$\text{Viscosity, } \mu = \frac{\tau}{du/dy} \text{ cP (Centi poise)} \dots\dots\dots(4)$$

$$\text{Kinematic viscosity, } \nu = \frac{\text{Dynamic viscosity } (\mu)}{\text{Density } (\rho)} \dots\dots\dots(5)$$

$$\text{Percentage of yield, } y = \frac{\text{Total weight of methyl ester}}{\text{Total weight of oil used}} \times 100\% \dots\dots\dots(6)$$

$$\text{Higher calorific value} = \frac{W \times \Delta T}{m} (\text{MJ/Kg}) \dots\dots\dots(7)$$

Where, W = Energy equivalent of calorimeter, Cal/°C

4. RESULTS AND DISCUSSION

An experiment has been conducted on biodiesel optimization. The catalysts were thoroughly analyzed to determine their characteristics and performance. Various parameters such as calorific value, flash point, fire point, density, specific gravity, acid value, dynamic viscosity, kinematic viscosity, pour point, cloud point, and more were carefully calculated using the collected data, and the data were compared to the previous studies with ASTM standard methods [29, 30], as shown in table 3. Detailed calculations are shown in Appendix C (from 4.1 to 4.7).

Table 3. Physicochemical Properties of Biodiesel

Parameters	Experimental Results	Expected Range of Biodiesel	Standard Values of Neat Diesel	ASTM Test Method
Higher Calorific Value (MJ/kg)	39.73	Min 40	Min 42.7	D4868
Flash Point (°C)	192	Min 130	Min 55	D93
Fire Point (°C)	203	Min 140	Min 60	D93
Density (kg/m ³)	877.04	860 – 914	820 - 860	D4052
Specific Gravity	0.877	0.86 – 0.90	0.82 – 0.86	D4052
Acid Value (mg KOH/gm)	0.45	0.2 – 0.5	0.01 – 0.05	D664
Dynamic Viscosity (cP)	7.8	1.6 – 11.1	1.6 – 3.9	D445
Kinematic Viscosity (mm ² /s)	9.13	1.9 – 12.3	2 – 4.5	D445
Pour Point (°C)	-7	-2 to -5	Winter = Max 3 Summer = Max 9	D97
Cloud Point (°C)	3	0 – 4	-15°C to - 5°C	D2500

Optimization was carried out using the desirability function approach integrated within the Response Surface Methodology (RSM) framework. The goal was to maximize biodiesel yield by simultaneously adjusting all process variables within experimentally defined boundaries. The optimization analysis identified the following combination of parameters as optimal for maximizing biodiesel production efficiency:

- Methanol-to-Oil Ratio (X_1): 10.8
- Catalyst Loading (X_2): 2.9%
- Reaction Time (X_3): 57 minutes
- Reaction Temperature (X_4): 61°C
- Predicted Yield: 90.2%

Although the maximum experimental yield reached 91.5%, this value corresponded to a single trial. In contrast, the RSM model predicted an optimized yield of 90.2% based on the fitted quadratic surface. The slight reduction is typical of statistical optimization, which prioritizes generalized, smooth responses across the design space rather than isolated peaks. Consequently, the selected optimum ensures greater robustness and reproducibility, as confirmed by a desirability value approaching unity 5.

5. CONCLUSION

This study established an efficient and sustainable protocol for castor oil biodiesel production using a heterogeneous CaO catalyst derived from calcined waste eggshells. Response Surface Methodology systematically optimized the key reaction parameters, identifying optimal conditions of a 10.8:1 methanol-to-oil ratio, 2.9 wt.% catalyst loading,

57 min reaction time, and 61°C temperature, resulting in a predicted yield of 90.2%. The produced biodiesel exhibited favorable physicochemical properties, including a higher calorific value of 39.73 MJkg⁻¹, flash and fire points of 192°C and 203°C, density of 877.04 kgm⁻³, specific gravity 0.877, acid value 0.45 mg KOHg⁻¹, dynamic and kinematic viscosities of 7.8 cP and 9.13 mm²s⁻¹, and satisfactory cold-flow characteristics with -7°C pour point and 3°C cloud point, confirming compliance with standard fuel quality requirements. The solid catalyst simplified separation, minimized wastewater generation, and enhanced process sustainability, demonstrating the technical feasibility of biowaste valorization within a circular economy framework. Scope for future studies will investigate catalyst reusability cycles, long-term storage stability of the fuel, engine performance and emission assessments, and pilot-scale continuous reactor implementation to validate industrial applicability.

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APPENDICES

Appendix A: Parameter Setting

Factor	Symbol	Unit	Minimum	Center	Maximum
Methanol to Oil Ratio	X ₁	Mol/mol	7	11	15
Catalyst Quantity	X ₂	% by wt. of oil	1	3	5
Reaction Time	X ₃	Minutes	55	77.5	100
Temperature	X ₄	°C	50	60	65

Appendix B: Experimental design matrix and observed yields

Run	MeOH: Oil	Catalyst (%)	Time (min)	Temp (°C)	Estimated Yield (%)
1	11	3	55	60	88
2	15	3	77.5	65	75
3	11	3	77.5	60	82
4	11	5	100	60	75
5	15	5	77.5	60	75
6	11	5	55	60	82
7	15	3	100	60	75
8	11	3	77.5	60	82
9	7	3	55	60	91.5
10	11	3	55	55	88
11	11	1	77.5	60	88
12	15	1	77.5	60	82
13	11	1	100	60	82
14	11	3	77.5	50	76
15	15	7	77.5	55	82
16	11	5	77.5	65	75
17	11	1	55	60	88

18	11	3	77.5	60	82
19	7	5	77.5	55	88
20	11	3	100	55	82
21	7	3	77.5	65	90
22	11	3	100	65	75
23	11	3	77.5	55	85
24	7	3	100	60	89
25	7	5	77.5	60	82
26	11	1	77.5	55	88
27	11	1	55	65	84
28	11	3	77.5	65	86
29	11	3	77.5	60	82
30	15	3	77.5	55	82

Appendix C: Experimental design matrix and observed yields

4.1 Calculation of Calorific Value of Biodiesel

Calorific Value of Biodiesel using Heterogeneous Catalyst Sample

Weight of the biodiesel taken, m = 0.79 gm

Length of the nichrome wire = 5 cm

Length of cotton wire = 10 cm

Pressure of oxygen inside the bomb = 30 atm

Volume of distilled water used = 2000 ml

Initial temperature of bomb calorimeter = 27.990°C

Then by adjusting the knob it was set to = 0°C

Energy equivalent of calorimeter, determined under standardization, W = 2489 cal/°C

Temperature rise, T = 3.014

Higher calorific value = $\frac{W\Delta T}{m} = \frac{2489 \times 3.014 \times 4.184}{0.79 \times 0.001} = 39.73 \text{ MJ/Kg}$

4.2 Calculation of Density of Biodiesel

Density of Biodiesel using a heterogeneous catalyst sample at 30°C is,

Density = $\frac{11.84}{13.5} \times 1000 = 877.04 \text{ kg/m}^3$

4.3 Calculation of Acid Value of Biodiesel

4.3.1 Acid Value of Raw Oil Sample

Normality of KOH used as titrant = 0.1 N

Mass of sample taken = 1 g

Volume of titrant in ml = 2.8 ml

Acid value, A/V = $\frac{56.1 \times 2.8 \times 0.1}{1} = 15.71 \text{ mg KOH/gm}$

4.3.2 Acid Value of Biodiesel using Heterogeneous Catalyst Sample

Normality of KOH used as titrant = 0.1 N

Mass of sample taken = 10 g

Volume of titrant in ml = 0.8 ml

Acid value, A/V = $\frac{56.1 \times 0.8 \times 0.1}{10} = 0.45 \text{ mg KOH/gm}$

4.4 Calculation of Specific Gravity of Biodiesel

Specific Gravity of Biodiesel using Heterogeneous Catalyst Sample

We got the density of the oil = 877.04 kg/m³

So, specific gravity of the oil = $\frac{877}{1000} = 0.877$

4.5 Calculation of Viscosity of Biodiesel

Viscosity of Biodiesel using Heterogeneous Catalyst Sample

We got the dynamic viscosity from the viscometer at 30°C = 7.8 cp

Kinematic viscosity of the biodiesel at 30°C = $\frac{7.8}{0.8543} = 9.13 \text{ mm}^2/\text{s}$

4.6 Calculation of Flash Point and Fire Point of Biodiesel

Flash Point and Fire Point of the Produced Biodiesel using Heterogeneous Catalyst Sample

The sample taken for measuring the flash point and fire point is 70 ml

The flash and fire points of the produced castor oil biodiesel were measured as 192°C and 203°C, respectively.

These elevated ignition temperatures are considerably higher than those of diesel.

4.7 Calculation of Cloud Point and Pour Point of Biodiesel

Cloud Point and Pour Point of Biodiesel using Heterogeneous Catalyst Sample

The cloud point of the sample is 3°C.

The pour point of the sample is -7°C.

NOMENCLATURE

ANOVA	Analysis of Variance	CCD	Central Composite Design
FFA	Free Fatty Acid	PET	Polyethylene Terephthalate
RMSE	Root Mean Square Error	RSM	Response Surface Methodology

DECLARATIONS

Ethics approval and consent to participate: Not applicable. This study did not involve any human or animal participants.

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AUTHORS' CONTRIBUTIONS

All authors contributed to the study conception and manuscript preparation. Md Ryshur Rahman Turin conceived the study, carried out the experimental work, performed data analysis, and drafted the manuscript. Md Abid Hasan assisted with biodiesel production experiments and optimization studies. Professor Dr. Jamal Uddin Ahamed supervised the research and critically reviewed the manuscript. All authors read and approved the final manuscript.

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