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Production of Biodiesel from Caster Oil: Experimental and Optimization Study

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Keywords:

Biodiesel; Castor Oil; Optimization; Response Surface Methodology; Transesterification.

Highlights:

- BBD was used in the experimental design.
- The highest product yield was obtained at a high molar ratio.
- Biodiesel was checked by GC-MS.

Abstract: Biodiesel production provides a diversified and renewable energy source offering lower greenhouse gas emissions than traditional diesel. It also offers economic benefits by reducing dependence on imported fossil fuels. Castor oil transesterification is an essential process in the creation of biodiesel. In this experimental study, castor oil transesterified using methanol, and potassium hydroxide was the catalyst. The effects of various reaction parameters, including temperature, the molar ratio of methanol to oil, and catalyst concentration, on the biodiesel yield were studied and optimized by the conventional method followed by the statistically based Box-Behnken design method. The maximum yield was reached at a temperature of 65°C, a molar ratio of 12:1 methanol to oil, and a catalyst concentration of 1.5% by weight. The yield of biodiesel under these conditions was 93%. The optimized results of experiments showed increases in yield to 93.36% at 65°C temperature, 14.12:1 a molar ratio methanol to oil, and a 1.12% by weight catalyst concentration; hence, the optimal temperature was the highest achieved value. The fatty acid methyl ester composition analysis revealed that the major constituents of the biodiesel were ricinoleic acid methyl ester, linoleic acid methyl ester, and oleic acid methyl ester. The findings of this research highlight the significance of selecting the appropriate reaction conditions to maximize biodiesel yield. Also, it was found that castor oil had the potential to be an essential feedstock for biodiesel production.

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إنتاج وقود الديزل الحيوي من زيت الخروع: دراسة عملية والظروف المثلى

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الخلاصة

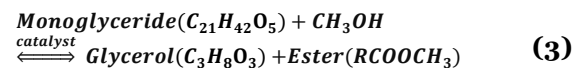
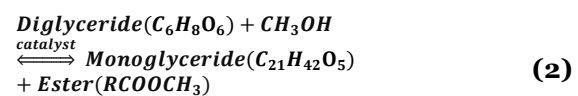
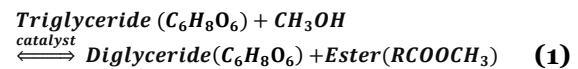
يوفر إنتاج وقود الديزل الحيوي مصدرًا متنوعًا ومتجددًا للطاقة يوفر انبعاثات أقل من غازات الاحتباس الحراري مقارنة بالديزل التقليدي. كما أنه يوفر فوائد اقتصادية من خلال تقليل الاعتماد على الوقود الأحفوري المستورد. تعتبر عملية التحويل إلى زيت الخروع عملية مهمة في إنتاج وقود الديزل الحيوي. استرة زيت الخروع هي عملية مهمة في صناعة وقود الديزل الحيوي، وهو بديل متجدد للوقود الأحفوري التقليدي. في هذه الدراسة التجريبية، تم إجراء الاسترة التبادلية لزيت الخروع باستخدام الميثانول وكان هيدروكسيد البوتاسيوم هو العامل المساعد. تم تحسين تأثيرات معاملات التفاعل المختلفة، بما في ذلك درجة الحرارة، والنسبة المولية للميثانول إلى الزيت، وتركيز المحفز، على إنتاجية وقود الديزل الحيوي بالطريقة التقليدية متبوعة بطريقة تصميم Box-Behnken. تم العثور على أقصى إنتاج عند درجة حرارة 65 درجة مئوية، ونسبة مولارية تبلغ 1:12 ميثانول إلى زيت، وتركيز محفز بنسبة 1.5٪ بالوزن. كان إنتاج وقود الديزل الحيوي في ظل هذه الظروف 93٪. أظهرت النتائج المثلى للتجارب زيادة في الإنتاجية إلى 93.36٪ عند درجة حرارة 65 درجة مئوية، و 1:14.12 نسبة مولارية ميثانول إلى زيت، و 1.12٪ بالوزن تركيز محفز، وبالتالي كانت درجة الحرارة المثلى هي أعلى قيمة يمكن تحقيقها. أظهر تحليل تركيبة إستر ميثيل الأحماض الدهنية أن المكونات الرئيسية للديزل الحيوي هي حمض الريسينوليك وإستر ميثيل وحمض اللينوليك وإستر ميثيل حمض الأوليك. تسلط نتائج هذا البحث الضوء على أهمية اختيار ظروف التفاعل المناسبة لزيادة مردود وقود الديزل الحيوي إلى الحد الأقصى، كما أن زيت الخروع لديه القدرة على أن يكون مادة بسيطة مهمة لإنتاج وقود الديزل الحيوي.

الكلمات الدالة: وقود الديزل الحيوي، الاسترة التبادلية، زيت الخروع، ومنهجية سطح الاستجابة.

1. INTRODUCTION

Using ordinary fuel oil, such as petroleum-based diesel or gasoline, harms the environment, human health, and sustainability [1]. It contributes to greenhouse gas emissions, air pollution, non-renewable resource depletion, environmental contamination, and health risks [2]. Transitioning to cleaner and more sustainable energy sources is crucial to mitigate these harmful effects [3,4]. Biodiesel is the most promising sustainable energy substitute for today's fossil-based fuels [5]. Additionally, due to high cetane numbers, low sulfur concentration, and aromatic content, diesel fuel is utilized in aviation and has strong combustion qualities in diesel engines [6]. The monoalkyl esters of vegetable or animal fats are known as biodiesel. Chemically, according to the alcohol (acyl acceptor) utilized during the process, the fatty acid alkyl esters (FAAEs) that compromise biodiesel come in the form of fatty acid methyl esters (FAMES) or fatty acid ethyl esters (FAEEs) [7]. Vegetable oils are biodiesel production's most commonly utilized raw materials [8]. Castor oil stands out because it has two intriguing characteristics as a biodiesel raw material: first, it does not compete with edible oils [9], and second, its growth does not need an extensive amount of inputs [10]. Castor oil is an essential raw material in the chemical and pharmaceutical industries. However, its application as fuel for internal combustion engines may be challenging due to its unusually high viscosity and water content [11]. Compared to the bulk of other widely-used oil seed crops (soybean; 15-20% (w/w), sunflower; 25-35% (w/w), rapeseed; 38-46% (w/w), and palm; 30-60% (w/w)), typically, 40-55% of the oil extracted from castor seeds is oil [12]. Typically, it is exceedingly viscous, pale yellow,

and has a slightly distinctive smell [13]. Castor oil contains 80-90% hydroxylated fatty acids, ricinoleic acid, and about 10% non-hydroxylated fatty acids, primarily oleic and linoleic acids. Because its viscosity is approximately seven times that of other vegetable oils, its utility for biodiesel generation is limited. To overcome this disadvantage, castor oil biodiesel has met effective parameters in standards when blended with petrodiesel [14]. Due to the presence of the group hydroxyl (-OH) connected to the chain of hydrocarbons in the ricinoleic acid of the molecule, castor oil is chemically different from other oils. The solubility of alcohol, reducing the melting point with improved oxidation stability, is another distinctive quality. Castor oil has a very high solubility in alcohol, making it possible to transform it into biodiesel at low temperatures [15]. Transesterification of animal or vegetable fats is the most commonly used technique [16]. In this reaction, short-chain alcohols and triglycerides combine in the presence of a catalyst to form diglycerides, subsequently combining to form monoglycerides. Then, because of the monoglycerides' reaction with the alcohol, ester and glycerol are produced as follows:



The two most widely utilized alcohols are methanol and ethanol. Ethanol is preferred over methanol for biodiesel because it is carbon dioxide neutral, less toxic environmentally, and can be made from agricultural feedstocks. Methanol is toxic and more volatile than ethanol. However, methanol is frequently utilized in practice since it is inexpensive and benefits in both physical and chemical terms [17]. The catalyst used, depending on how soluble the catalyst is in the reactant, can be homogeneous, including alkaline catalysts, such as sodium hydroxide (NaOH) and potassium hydroxide (KOH); acidic catalysts, such as sulfuric acid (H₂SO₄) or hydrochloric acid (HCl); heterogeneous, including solid base catalysts, such as calcium oxide (CaO), magnesium oxide (MgO), and sodium silicate (Na-SiO₂); and solid acid catalysts, such as sulfated zirconia (ZrO₂/SO₄) and zeolites, or enzymatic, often derived from lipases or other enzymes [18-20]. Homogeneous catalysts are preferred over heterogeneous catalysts in biodiesel production due to higher reaction efficiency, faster reaction rates, better control over reaction kinetics and parameters, and

simplified catalyst separation from the reaction mixture [21]. Castor oil transesterification with homogeneous catalysts has been the subject of numerous studies and sustainable studies for different engineering applications. The optimal conditions and yields of some studies of alkali-catalyzed transesterification are shown in Table 1. To create methyl esters of fatty acids from castor oil and use a catalyst, potassium hydroxide, this research aimed to produce experimental data on these processes employing response surface methodology, considering reaction conditions' impact on product yield and quality. Ultimately, this research contributes to developing more sustainable and environmentally friendly energy sources through biodiesel production from renewable feedstocks. Overall, the differences between this work and others are process parameters, catalyst selection, and research objectives of each study. Understanding these differences is crucial for evaluating biodiesel production's unique aspects and potential benefits from different feedstocks.

Table 1 The Optimal Conditions and Yields of Some Alkali-Catalyzed Transesterification Castor Oil Studies.

Temperature in ° C	Molar ratios	Catalyst type	Catalyst concentrations	Yield	Reference
60	9:1	KOH	1%	95 %	[22]
70	3:1	NaOH	0.5 %	96.2%	[23]
65	6:1	KOH	1%	83.41%	[24]
60	12:1	KOH	1.25%	94.9%	[25]
65	7:1	KOH, NaOH, CH ₃ ONa, CH ₃ OK	1.5%	99%	[11]
64	5.4:1.21	KOH	0.73%	97.82%	[26]
45	18.8:1	CH ₃ OK	1%	97%	[27]
40	25:1	NaOH	1%	93%	[28]
40	5:1	CH ₃ ONa	1%	99%	[29]

2. MATERIALS AND METHODS

2.1. Materials

Commercial-grade castor oil, methanol (99% (v/v) purity, and potassium hydroxide pellets (purity > 99% (w/w) are supplied from Sisco Research Laboratories Pvt Ltd (SRL) - India).

2.2. Experimental Procedure

A 250 ml glass three-necked batch reactor, a reflux system, and a hot plate magnetic stirrer (Heidolph MR Hei-Standard) were used to conduct the transesterification processes. Fig. 1 shows the equipment used. Each experiment was conducted using 30 g of castor oil. KOH was combined with methanol to create CH₃OK. Castor oil was heated until the appropriate temperature was obtained. The transesterification process then started when the oil was mixed with a mixture of methanol and catalyst. The stirrer was operated at 650 rpm for one hour to avoid the mass transfer limits [25]. The parameters used were as

follows: temperature (35 to 65 °C), methanol to oil molar ratio (6:1 to 18:1), and KOH (0.5 to 1.5 % w/w of oil). After transesterification, the solution was removed from a hot plate and cooled to room temperature. After that, it was moved to the separating funnel for a few hours. Two layers were created: the top layer was made of biodiesel, while the bottom layer was made of unreacted catalysts, extra methanol, or other contaminants. The two layers are shown in Fig. 2. Glycerol was then separated, and the upper methyl ester phase was washed with deionized water to remove the acid catalyst and residual alcohol and then dried by heating at 110 c for 30 min in a drying oven. Fig. 3 shows the process diagram in the laboratory. The following equation determines yield by weighing purified biodiesel [25].

$$\text{Biodiesel yield \%} = \frac{\text{Weight of biodiesel produced}}{\text{weight of castor oil}} \times 100 \quad (4)$$

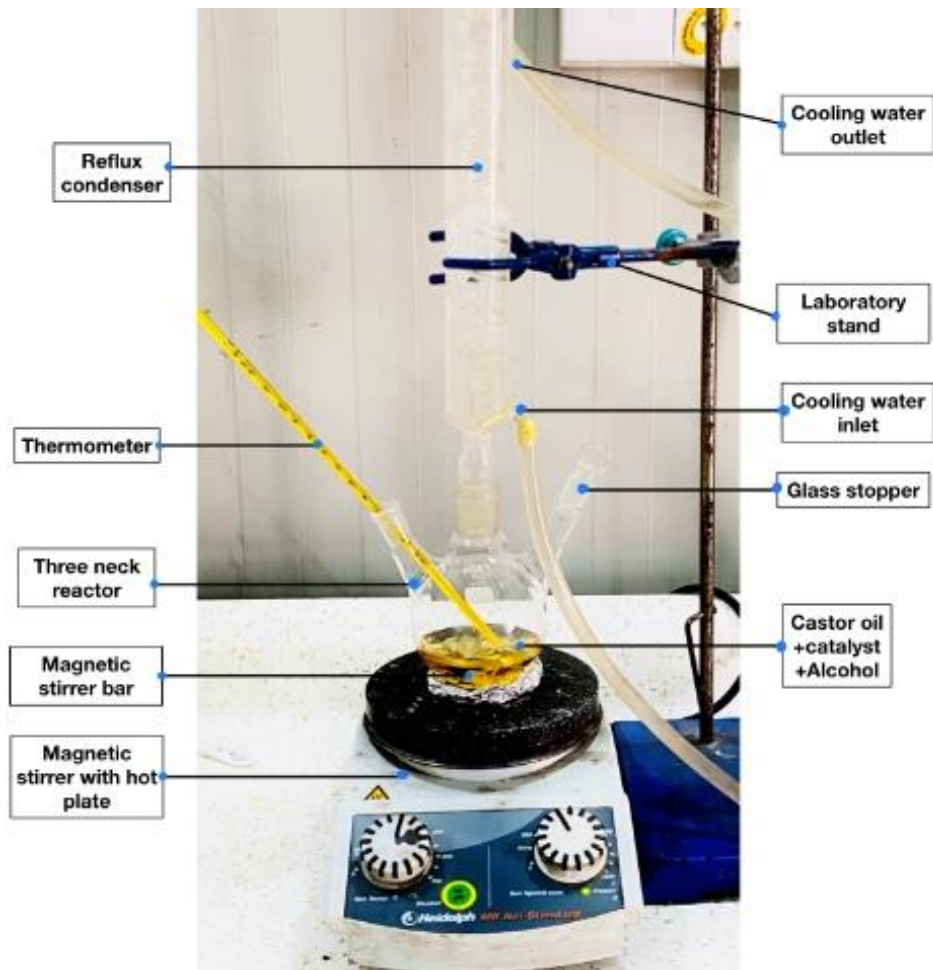


Fig. 1 The Batch Reactor System.

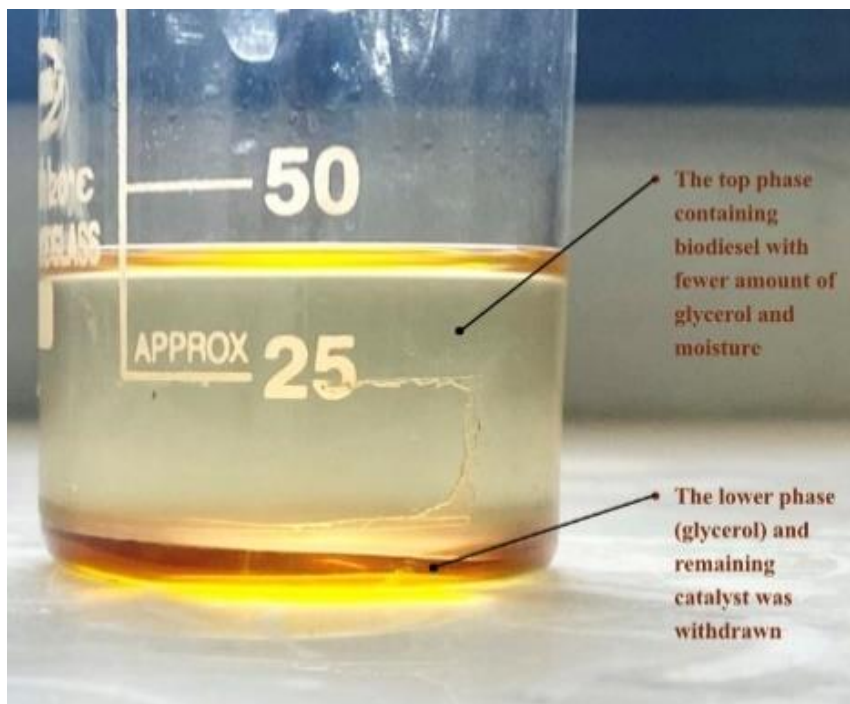


Fig. 2 Two Layers: The Upper Methyl Ester and the Lower Glycerol.

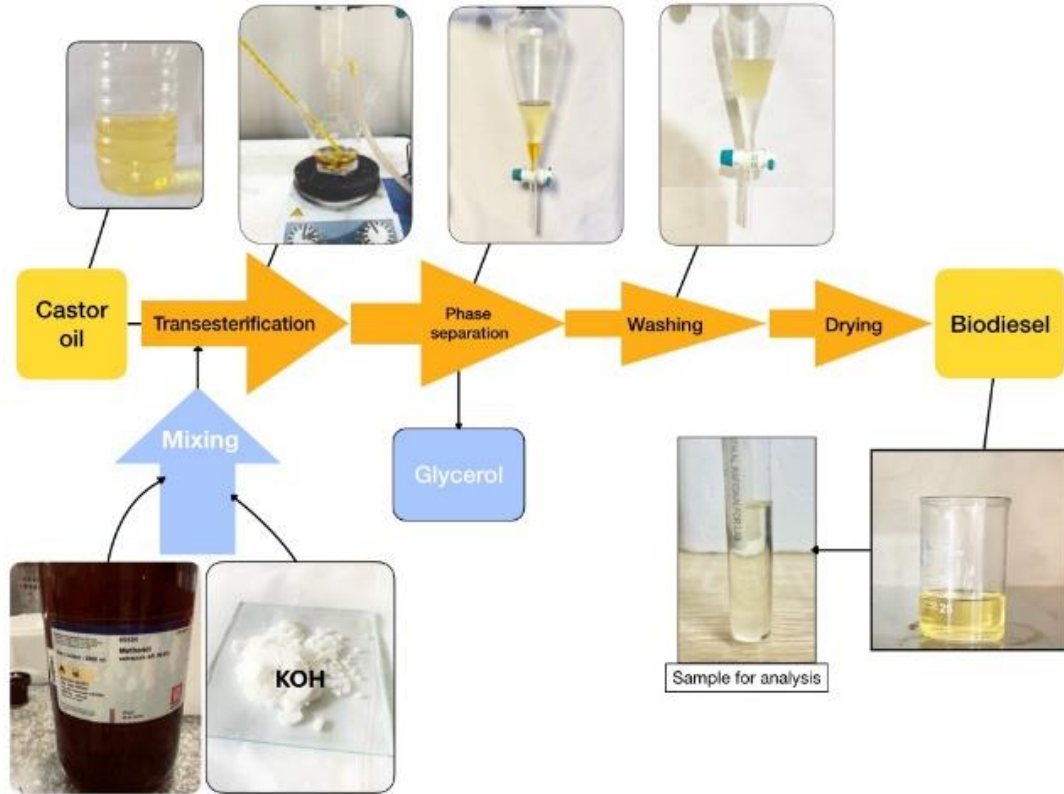


Fig. 3 Scheme of the Transesterification Process.

2.3. Optimization of the Transesterification Process:

Response surface methodology (RSM) and Box-Behnken design (BBD) were the statistical techniques utilized to optimize the biodiesel production from castor oil by the statistical software Minitab 18. Applying this methodology requires an appropriate selection of responses, factors, and levels. The factors were temperature, catalyst concentration, and methanol-to-oil molar ratio. The upper and lower levels are listed in Table 2. According to the BBD matrix, fifteen experimental runs were conducted. Higher regression coefficient R² values and results from the analysis of variance (ANOVA) are indicators of a model's quality [30]. The coefficient b₀ represents the result in the center, whereas the other coefficients quantify the average effects and interactions of the coded factors X_i on the response Yield [31]:

$$\text{Yield} = b_0 + \sum b_i X_i + \sum b_{ii} X_i^2 + \sum b_{ij} X_i X_j \quad (5)$$

Table 2 Process Variables with their Levels.

Name	Code	Low (-1)	Middle (0)	High (+1)
Temperature °C	A	35	50	65
Methanol to oil	B	6	12	18
Molar ratio				
Catalyst concentration %	C	0.5	1	1.5

2.4. Gas Chromatography and Mass Spectrometry (GC-MS) Analysis

Quantitative analysis of fatty acid methyl ester content in biodiesel was performed by gas chromatography and mass spectrometry

Agelint (5977E) USA with analytical column Agelint HP-5ms Ultra Inert (30 m length × 250 μm inner diameter × 0.25 μm film thickness), as shown in Fig. 4. The analysis was done in Industrial Research and Development Authority / Ministry of Industry and Minerals. Instrument Conditions were 250 °C GC inlet line temperature, 300 °C oven temperature, 250 °C injector temperature, and 11.933 psi pressure. The program had a set rate of 8 °C/min and ranged from 70 to 280 °C hold to 3 min. The result is shown in Table 4.



Fig. 4 Gas Chromatography and Mass Spectrometry Device.

3.RESULTS AND DISCUSSION

Analyzing the factors' impact on the FAME yield helps optimize the process conditions for the transesterification of castor oil to FAME, which is a vital step in biodiesel production. Based on the BBD experimental design, Table 3 lists these experimental parameters and their yields. For all experimental responses, product yields ranged from 88 to 93% (w/w). According to the results of the present study, the temperature had more effect on product yield than the molar ratio of methanol:oil and the catalyst concentration. The highest product yields were obtained using 65 °C, 12 :1 molar ratio, and 1.5% catalyst concentration (mean of 93% (w/w)).

3.1.Fatty Acid Profile (GS–MS) Analysis

The castor oil biodiesel was checked by GC-MS to ensure that the oil was converted into its fatty acid methyl esters (FAME) and other compounds. Table 4 shows a list of the FAME found using GC-MS analysis. According to GC-MS analysis, 9-Octadecenoic acid 12-hydroxy [R-(Z)] methyl ester (Ricinoleic acid), 9-Octadecenoic acid (Z) methyl ester (oleic acid), 9-12- Octadecadienoic acid (Z, Z) methyl ester (linolenic acid), and Hexadecanoic acid methyl ester (palmitic acid) were the primary fatty acids in the biodiesel made from castor oil. As shown in Fig. 5, the GC-MS findings for castor biodiesel revealed distinct peaks of retention times taken by the detector's component compared to the area covered by fatty acid molecules.

3.2.Effect of Temperature

Reaction temperature is an essential factor that impacts biodiesel production. The optimum temperature for an experiment was 65 °C. As shown in Fig. 6, the biodiesel production increased directly as temperature increased from 35 °C to 65 °C. Increased temperature speeds up the process and increases yield, which may be related to a decrease in oil viscosity as temperature rises, leading to better mixing of oil with alcohol and quicker separation of glycerol from biodiesel [32]. Furthermore, a reflux condenser was utilized in the tests to reduce methanol losses because as the reaction temperature approached or was above the boiling point of methanol (64.7 C°), the methanols vaporized and generated a significant number of bubbles, inhibiting the process [33].

3.3.Effect of the Catalyst Concentration

The quantity of methyl ester of castor oil produced decreased with decreasing catalyst concentrations of KOH; however, biodiesel yields are insignificantly affected by increasing concentrations of KOH. Fig. 7 shows that increasing KOH content positively affected biodiesel yield from 0.5 to 1.12%. However, at higher concentrations, it insignificantly impacted biodiesel yield. A high catalyst concentration reportedly reduced the conversion of fatty acids to butyl esters due to secondary reactions, such as saponification reactions. Sánchez et al. [34] showed that the best biodiesel yield at the optimum catalyst value was 1.2 wt.%.

Table 3 Experimental Runs Variables with Results of Biodiesel Yield Using BBD.

Runs	Temperature	Molar Ratios	Catalyst Concentrations %	Yield %
1	35	6	1.0	88.1
2	65	6	1.0	91.6
3	35	18	1.0	90.0
4	65	18	1.0	92.4
5	35	12	0.5	87.0
6	65	12	0.5	92.5
7	35	12	1.5	90.6
8	65	12	1.5	93.3
9	50	6	0.5	88.6
10	50	18	0.5	90.4
11	50	6	1.5	89.6
12	50	18	1.5	92.3
13	50	12	1.0	91.8
14	50	12	1.0	92.0
15	50	12	1.0	91.5

Table 4 FAME Composition of Biodiesel Using GC-MS.

Fatty Acid	Systematic Name	Carbon Number	Wt%
Palmitic acid	Hexadecanoic acid methyl ester	C 16:0	2.19
Linoleic acid	9,12-Octadecadienoic acid methyl ester	C 18:2	0.27
Stearic acid	Methyl stearate	C 18:0	0.68
Linolenic acid	9,12-Octadecadienoic acid (Z,Z) methyl ester	C 18:3	6.40
Oleic acid	9-octadecenoic acid (Z) methyl ester	C 18:1	5.15
Ricinoleic acid	9-Octadecenoic acid 12-hydroxy [R-(Z)] methyl ester	C 18:1-OH	85.32

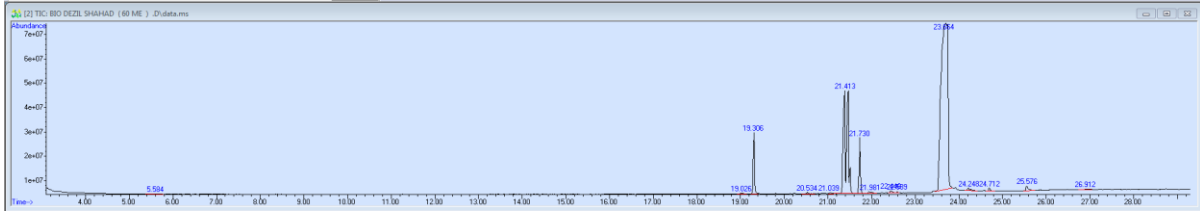


Fig. 5 GC-MS Chromatogram of Biodiesel.

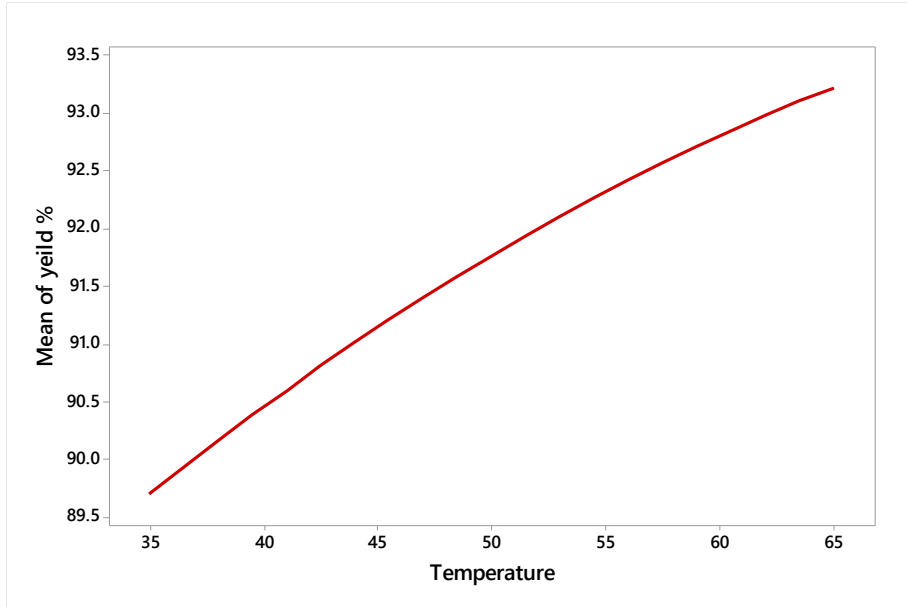


Fig. 6 The Effect of Temperature on the FAME Yield.

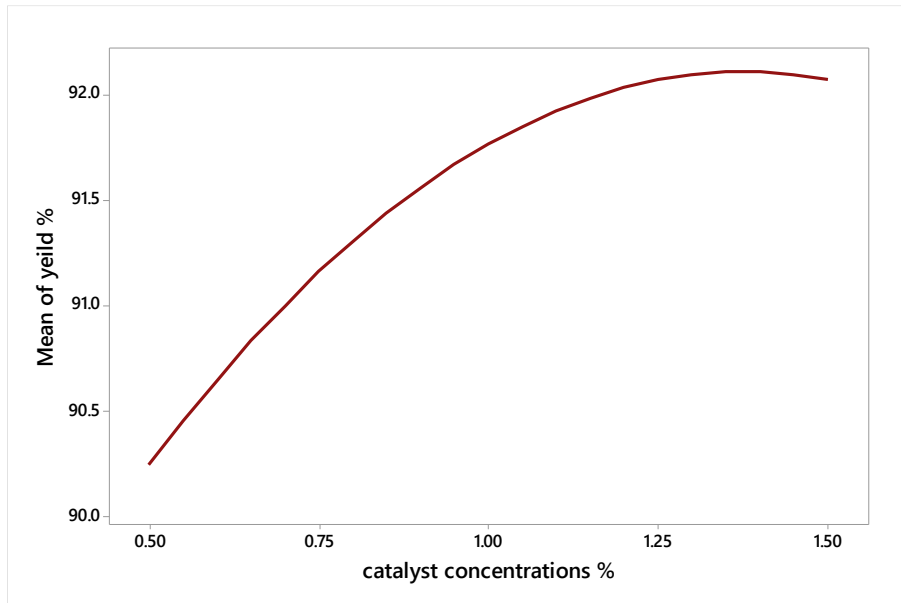


Fig. 7 The Effect of Catalyst Concentration on the FAME Yield.

3.4. Effect of Methanol to Oil Molar Ratio

To produce 1 mole of glycerol and 3 moles of methyl ester, the transesterification reaction's stoichiometry ratio requires 3 moles of methanol. To move this reaction to the right, since it is reversible, more methanol can be used [35]. Fig. 8 shows a considerable rise in biodiesel yield when the methanol/oil molar ratio increased from 6:1 to 12:1; however, there was a decrease in biodiesel yield. When the methanol/oil molar ratio increased beyond 14:1. Because of the increased solubility, a

larger alcohol molar ratio interfered with glycerol separation [36]. Furthermore, since some of the glycerol was still in the alkyl esters phase, the yield of alkyl esters decreased. Additionally, compared to transesterification reactions conducted using low molar ratios. The conversion of di- to monoglycerides with excess alcohol appeared to be preferred, and glycerol and alkyl esters slightly recombined into monoglycerides because of the reaction's increasing concentration [17].

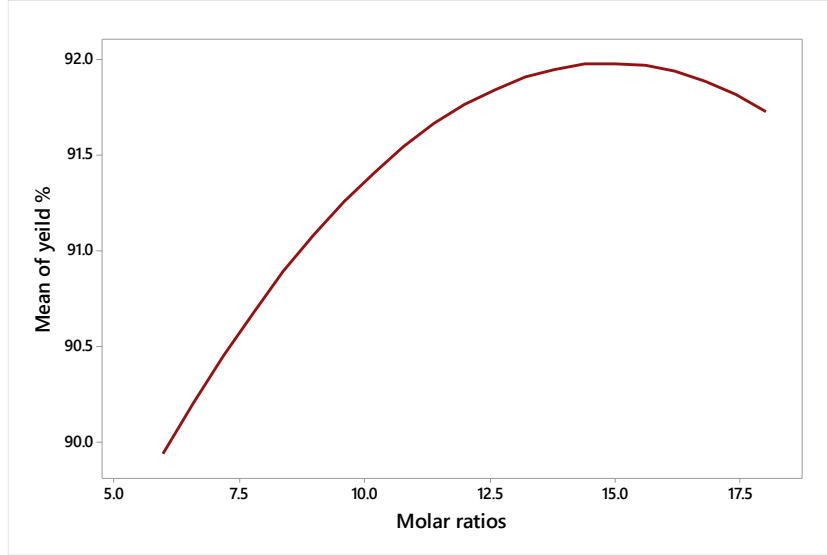


Fig. 8 The Effect of Methanol: Oil Molar Ratio on the FAME Yield.

3.5. Optimization of the Transesterification Process by BBD and ANOVA Analysis

Multiple regression analysis was used to create the statistical model using experimental data. The resulting polynomial Eq. (6) gave the FAME yield (Y) due to the reaction temperature (A), the methanol:oil molar ratio (B), and the KOH concentration (C):

$$\text{Yield \%} = 67.07 + 0.385 A + 0.850 B + 10.46 C - 0.00137 A^2 - 0.02593 B^2 - 2.43 C^2 - 0.00306 AB - 0.0933 AC + 0.0750 BC \quad (6)$$

Analysis of Variance (ANOVA) was used to assess the model's statistical significance, and the results showed that the model was statistically significant with a p-value of 0.003 and an R² value of 96.82%, indicating that it could accurately anticipate a better response. According to Table 5, the quadratic term molar ratios of oil to methanol (BB) (p = 0.021), the

linear terms of temperature (A) (p = 0.0), catalyst concentration (C) (p = 0.005), oil:methanol molar ratio (B) (p = 0.005) and interaction terms of temperature and catalyst (AC) (p = 0.05) substantially impacted on the FAME production. Indicating that the model was correctly registering most of the experimental data, the lack-of-fit value of the model was found to be 0.126 (not significant). R² and R²_{adj}, which quantify the accuracy of the model fitting, were determined to have values of 0.9682 and 0.911, respectively. Fig. 9 shows the operational variable's ideal values; when D equals 1, the results obtained are accurate. The yield increased to 93.36% with a rise in temperature, a 14.12:1 molar ratio, and a concentration of 1.12% catalyst; hence, the optimal temperature was the highest value that could be achieved. The optimal conditions obtained can be used to improve the efficiency and cost-effectiveness of the process.

Table 5 Analysis of Variance (ANOVA) of Biodiesel Yield Using BBD.

Source	Degrees of freedom	Adjusted sum of squares	Adjusted mean squares	F-Value (ratio of two variances)	P-Value (probability)
Model	9	44.8898	4.9878	16.92	0.003
Linear	3	37.9925	12.6642	42.95	0.001
Temperature (A)	1	24.8512	24.8512	84.29	0.000
Molar ratios (B)	1	6.4800	6.4800	21.98	0.005
catalyst concentrations % (C)	1	6.6612	6.6612	22.59	0.005
Square	3	4.4323	1.4774	5.01	0.057
Temperature*Temperature (A ²)	1	0.3510	0.3510	1.19	0.325
Molar ratios*Molar ratios (B ²)	1	3.2164	3.2164	10.91	0.021
Catalyst concentrations %*catalyst concentrations % (C ²)	1	1.3664	1.3664	4.63	0.084
2-Way Interaction	3	2.4650	0.8217	2.79	0.149
Temperature*Molar ratios (AB)	1	0.3025	0.3025	1.03	0.358
Temperature*catalyst concentrations % (AC)	1	1.9600	1.9600	6.65	0.050
Molar ratios*catalyst concentrations % (BC)	1	0.2025	0.2025	0.69	0.445
Error	5	1.4742	0.2948		
Lack-of-Fit	3	1.3475	0.4492	7.09	0.126
Pure Error	2	0.1267	0.0633		
Total	14	46.3640			

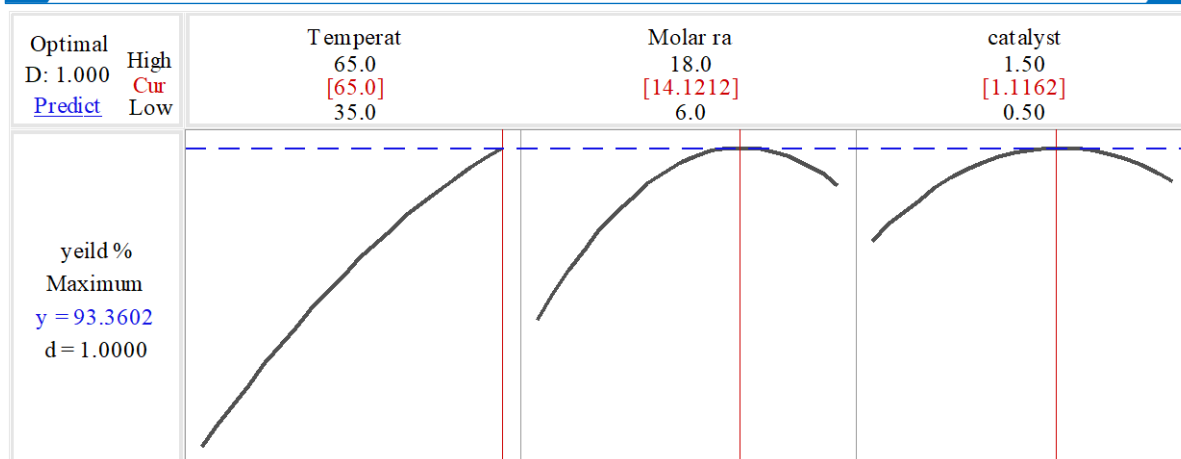


Fig. 9 The Transesterification Process Operating Variables Optimal Values.

4. CONCLUSIONS

The experimental transesterification of castor oil research indicated that castor oil might be a useful feedstock for biodiesel production using methanol and potassium hydroxide as catalysts. Experimental results from the Box-Behnken design method demonstrated that the optimal conditions for the transesterification reaction were 65 °C reaction temperature, 1.16% (w/w) KOH catalyst, and a molar ratio of 14.12:1 methanol to oil to obtain 93.36% FAME content. ANOVA statistics revealed that the model was highly significant, with an R^2 value of 0.968, indicating that the experimental data correlates well with the predicted model. GC-MS analysis identified ricinoleic acid methyl ester, linolenic acid methyl ester, oleic acid methyl ester, and palmitic acid methyl ester as the most prevalent esters in FAME. These findings suggest that castor oil has a high potential as a feedstock for its production of biodiesel. That optimization of reaction conditions can result in high yields of high-quality biodiesel and provide insights that can contribute to the development of more efficient and sustainable biodiesel production methods. Overall, the results of this study contribute to the growing body of knowledge on the use of non-edible feedstocks for biodiesel production, which can reduce the dependence on fossil fuels and promote sustainable energy production.

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