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Optimization and Production of Biodiesel from Castor Seed Oil Using Cocoa Pod Ash as a Catalyst

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Abstract

This research work uses a base catalyst made from cocoa pods to optimize process variables for the production of biodiesel from castor oil. After being thermally treated for 35 minutes at 600˚C, the cocoa pods were sieved to ensure homogeneity before being utilized in the transesterification process. Using Definitive Screen Design in Design Expert software 10.1, optimization was carried out. It was discovered that the ideal parameters for producing biodiesel using castor oil were 50˚C, 2.5 hours of reaction time, a 10:1 methanol-to-oil ratio, and 6% weight percentage catalyst loading, which produced a 88% yield. The catalyst demonstrated a 69.48 kJ/mol activation energy, an exponential factor of 421.257 s^{-1}, and four cycles of reusability. This study demonstrates the potential of catalysts made from cocoa pods for effective biodiesel production.

Keywords

Biodiesel, Castor Oil, Cocoa Pod Husk, Trans-Esterification, Optimization, Design Expert Software

1. Introduction

The depletion of the stratospheric ozone layer, exacerbated by greenhouse gas emissions from conventional petroleum diesel, presents a critical environmental challenge that necessitates the transition toward sustainable energy alternatives. Biodiesel has emerged as a promising solution due to its renewability, high combustion efficiency, and significantly lower emission profile compared to fossil

fuels. To mitigate the ethical concerns of using edible crops for fuel, this study investigates the use of non-edible castor oil (*Ricinus communis* L.) as a primary feedstock. Castor oil is particularly well-suited for biodiesel production because of its high ricinoleic acid content, low iodine value, and excellent low-temperature properties, offering a viable pathway to reduce environmental impact without compromising global food security [1]-[5].

Biodiesel is a promising alternative to fossil fuels derived from vegetable oils and animal fats according to [6]. The main components of vegetable oils and animal fats are esters of fatty acid or triglyceride attached to glycerol. Biodiesel is chemically produced by combining natural oil or fat with an alcohol such as methanol or ethanol. Methanol is the most commercially used alcohol for the commercial production of biodiesel. Different works on biodiesel have shown that the fuel from vegetable oil can be used properly on diesel engines [7]-[10]. To work with compression ignition engines, biodiesel can be used either pure or blended [11]. With advantages over fossil fuels like biodegradability, renewability, high combustion efficiency, low sulfur content, and low emissions. Biodiesel enhances the environment by producing less soot in the vehicle's exhaust and a pleasant, fruity smell, according to [12]. It also lessens engine wear, which prolongs the life of the fuel injection equipment. When used, biodiesel generates less particulate matter and noise during idle, and it is simple to start cold [13]. Similar findings were made by [14], who discovered that biodiesel is more lubricating than all other fuels, less poisonous, safer to handle, and produces lower emissions of hydrocarbons and carbon monoxide than diesel. However, a sizable portion of biodiesel is made from edible vegetable oil, which puts food supplies in direct competition. As such, attempts are underway to create biodiesel from non-food sources of oil in order to combat this catastrophic event [15]. In this study, the generation of biodiesel from non-edible oils was investigated. These included animal fat, castor oil, Jatropha, and leftover vegetable oil.

The tropical plant *Ricinus communis* L., also known as the castor bean, is a member of the Euphorbiaceae spurge group and genus Ricinus [16]. Castor bean seeds thrive in marginal soils and have a strong ability to adapt to various weather conditions. About 80% - 90% of the total fatty acid composition in castor oil is ricinoleic acid ($C_8H_{34}O_3$), which is the main fatty acid. The oil is non-edible and harmful since it contains 1% - 5% ricin, a toxic protein that can be eliminated through cold pressing and filtering. Its molecules include hydroxyl groups, making it highly polar in comparison to other vegetable oils. It is a suitable raw material for the manufacturing of biodiesel due to its properties, which include low iodine content, high viscosity, high molecular weight, low freezing point, very low solidification point (−12 to −18˚C), and low melting point [17]-[19].

Biodiesel production needs a catalyst because it lowers the activation energy by modifying the reaction's transition state. The type of catalyst employed in the transesterification reaction is critical in converting triglycerides to biodiesel. The catalyst used to catalyze the transesterification reaction might be homogeneous or

heterogeneous. Cocoa pod husk contains cellulose, lignin, and hemicellulose, which can break down into carbon following calcination. The potassium content of cocoa pods can be isolated as an element of K_2CO_3 [19], and it is used as activated carbon and a K_2CO_3 catalyst in biodiesel production.

Consequently, much work is centered on the advancement and optimization of the processes of biodiesel generation to meet the measures and details required for the fuel to be utilized commercially. Diverse research involving the optimization of biodiesel production from various animal fat oils and plant oils with response surface models has been reported [20], which also includes process parameters [21]. Several scientific Design of Experiments (DOE) procedures can be utilized to investigate which factors and at what level the factors will maximize a specific yield. These methods have been broadly utilized in all circles of the science of maximizing yield for a given input of resources [22]. The focus of this method is to optimize the response (biodiesel yield) that is influenced by several independent input variables. Definitive screen design was used to study the effects of the independent variables on the dependent variables. The careful use of this design of experiments and using suitable mathematical models developed from this design. It is convenient to predict the optimal process conditions with a minimum number of experiments thereby saving time and experimental cost. Hence, this research work describes the production of biodiesel from castor oil as feedstock. It discusses the optimum values at which the maximum yield is been achieved while varying the different process variables as discussed in the method. Finally, it also reveals the number of times the catalyst can be reused effectively.

2. Materials and Method

The laboratory process required the use of many pieces of equipment, including Castor seed, methanol (analytical grade), cocoa pod, mortar and pestle, a magnetic stirrer (Model 400, CGOLDENWALL, China), distilled water, and a weighing balance.

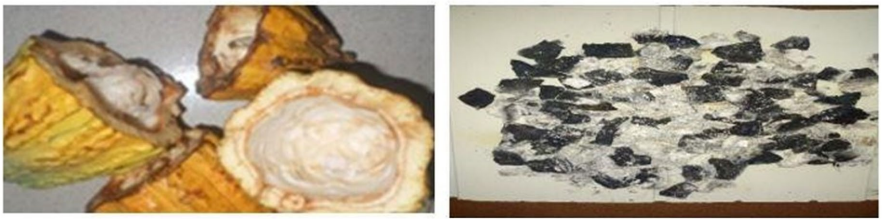
2.1. Seed Collection and Preparation

Castor seeds were collected from wild castor plants growing on moist marginal soil near Iluju in Ogbomosho, Oyo State, Nigeria. Ripe castor fruits were hand-cleaned and sun-dried for 4 - 5 days until the capsules burst open, revealing the seeds inside. The seed pods were then removed, and the shells and beans (cotyledons) were separated using tray-winnowing. Before extraction, the beans were ground into a paste with a mortar and pestle [23] [24].

2.2. Catalyst Synthesis and Characterization

The cocoa pods, obtained from a farm at Ilorin, Kwara State, Nigeria were sun-dried for 5 d before being reduced to ashes (**Figure 1(a)**) by placing crucibles containing the dried pods in a muffle furnace set to 600˚C for 35 min according to [24] [25] (**Figure 1(b)**). The ash was then sieved to achieve an average particle size

of 0.8 mm and then analyzed by atomic absorption spectroscopy to determine the metal composition for use as a transesterification catalyst [24] [25].



(a) (b)

Figure 1. CPH before (a) and after (b) sun-drying and calcination.

2.3. Synthesize and Characterize the Base Catalyst

2.3.1. Oil Extraction

For the purpose of extraction, castor seed paste (40 g) was wrapped in a clean muslin cloth and placed in the thimble, which was inserted at the center of the extractor. About 50 mL of hexane was weighed and poured into a round-bottom flask. The round bottom flask and a condenser were attached to the extractor to form the soxhlet extractor. The solvent in the extractor was then heated until it boiled and vaporized through the vertical tube into the condenser at the top. The liquid condensate is dropped into the cotton wool thimble at the center, containing the solid sample that retains the extract. The extract seeped through the thimble into the flask via the siphon. After extracting for 3 h, the sample was dried at 60˚C (to remove residual solvent) and weighed to determine the yield of oil extracted using Equation (1) [24].

$$\%\text{yield} = \frac{y_1 - y_2}{y_1} \times 100 \tag{1}$$

where y_1 and y_2 are the weights of castor beans before and after extraction, respectively.

2.3.2. Typical Experimental Run in Biodiesel Production

A small-scale laboratory glass reactor placed on a hot plate with a magnetic stirrer was used for the trans-esterification reaction. The Biodiesel sample was prepared using castor oil (**Figure 2**). Catalyst loading ranging from 3% to 6% weight (relative to oil) was used in the trans-esterification processes, with a methanol-to-oil ratio of 6:1 to 10:1. The reaction time and temperature ranged from 1 h to 4 h and 45˚C to 60˚C. A separating funnel separated the fatty acid methyl esters from the glycerol.

The yield of the biodiesel produced was then calculated using Equation (2).

$$\text{Biodiesel yield}\% = \frac{\text{weight of synthesized biodiesel}}{\text{weight castor oil}} \tag{2}$$

2.4. Optimization Studies

Definitive screen designs were used to study the main effects and the interactions

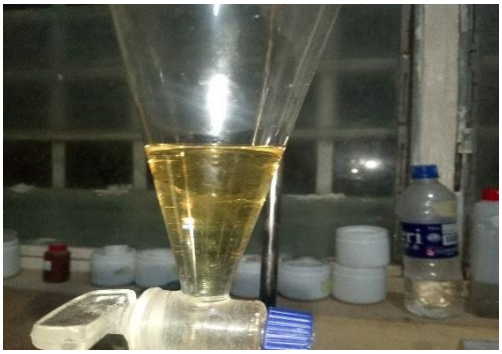


Figure 2. Biodiesel product.

between the experimental variables. The modeling of the trans-esterification process was also investigated using the four variables, which in turn produced 13 experimental runs for the optimization studies. The model equation produced was validated, and its level of significance was investigated using the R-squared and analysis of variance (ANOVA). Table 1 shows the experimental variables used in the design process.

Table 1. Experimental Variables and Levels Used for the Biodiesel Synthesis.

Reaction Variables	Units	Low code	Mid code	High code
Temperature	˚C	45	55	60
Time	H	1	2.5	4
M/O	w/w	5	7.5	10
Catalyst loading	Wt	3	4.5	6

2.5. Kinetic Studies

The kinetic study of the reaction at optimal conditions by the variation of the effect of the reaction time and temperature was investigated. The study was based on the following assumptions: the occurrence of a single-step transesterification reaction and that, since methanol was in excess, no reversible reaction occurred. Hence,

For transesterification reaction

$$-r = -\frac{\mathrm{d}[Castor\ oil]}{\mathrm{d}[t]} = k' \cdot [castor\ oil] \cdot [MtOH]^3 \tag{3}$$

k = Over all equilibrium constant, k' = Equilibrium constant, $[MtOH]$ is the concentration of Methanol.

$$-r = -\frac{\mathrm{d}[Castor\ oil]}{\mathrm{d}[t]} = k \cdot [Castor\ oil] \tag{4}$$

Knowing that

$$X_{castor\ oil} = 1 - \frac{[Castor\ oil\ conc]}{[initial\ castor\ oil\ conc]} \tag{5}$$

From Equation (4). we have

$$-\ln\left(1-X_{castor\ oil}\right)=k\cdot t \tag{6}$$

where,

$$X_{castor\ oil}=castor\ oil\ conversion.$$

From Equation (6), the values for "*k*" at four different temperatures of 45˚C, 50˚C, 55˚C, and 60˚C, was determined by plotting $-\ln\left(1-X_{castor\ oil}\right)$ against *t*, after which Equation (7).

Arrhenius equation was used to determine the activation energy by plotting $\ln k$ against $\frac{1}{T}$.

Hence, the slope and the intercept were used to determine the activation energy E_a and the frequency factor k_o

$$\ln k=\frac{-E_a 1}{RT}+\ln k_o \tag{7}$$

2.6. Reusability Test

After washing the deposited FAMEs on the active site of the used catalyst, the catalyst was dried at 40˚C until constant weight was achieved. This procedure was then repeated after each cycle. The catalyst was reused four times.

3. Result and Discussion Oil Extraction

After extraction, the yield of the extracted oil was calculated and found to be 42%. This is in agreement with the extraction method used in the literature [26]. During the process, hexane (solvent) was reused several times to maximize the aforementioned yield and minimize the cost of extraction.

3.1. Design Experiment

Table 2 presents the actual and predicted values. Equation 8 shows the resulting equation from the definitive screen design, a response surface methodology. The analysis of variance (ANOVA) results in an F-value of 354.40, which implies that the model terms are significant. In this case, *A*, *B*, *C*, *D*, *AB*, *AC*, *AD*, *BC*, *BD*, *CD*, and A^2 are the significant quadratic coefficients of the model.

$$\begin{aligned}Y_{CCPH}=&+72.82+0.29A+2.09B+18.66C+2.19D-8.58AB-2.11AC\\&-9.16AD+16.54BC-2.21BD-8.28CD-2.74A^2\end{aligned} \tag{8}$$

where the Yield of biodiesel (Y_{CCPH}), *A* is the temperature (˚C), *B* is the reaction time (h), *C* is the methanol/oil ratio, and *D* is the catalyst loading (wt%) was an effective factor considered. Also, based on the model, the methanol/oil ratio has the highest effect on the biodiesel yield due to the high positive coefficient. This was also confirmed by the ANOVA, which indicates the order of significance of the independent variables and denotes that methanol/oil is the most important variable affecting the biodiesel yield. In addition, a low lack of fittest of 4 was noted

Table 2. Actual and predicted values for the trans-esterification using CPA.

Run Order	*A*	*B*	*C*	*D*	Actual Value	Predicted Value
1	45	4	5	4.5	43.00	43.15
2	50	4	6	3	48.00	47.82
3	60	1	10	4.5	77.00	76.87
4	55	4	8	6	74.00	74.02
5	55	2.5	7.5	4.5	72.50	72.61
6	60	1	7.5	6	72.00	72.10
7	50	2.5	10	6	88.80	88.75
8	50	2.5	6	5	64.00	64.21
9	55	3	5	6	56.00	55.57
10	50	1	5	3	49.00	48.93
11	45	1	10	3	58.00	58.08
12	55	3	6	5	59.00	59.40
13	45	2.5	7.5	4.5	70.00	69.80

according to the ANOVA Table 3. This indicates that the model represents the actual relationship of all the parameters, which are all within the selected range. In actual fact, the P-value of 0.0414 and F-value of 354.40 of the model are indications of the significance of the model. The regression model adequately predicts the biodiesel yield within the design space, as the R^2 of 0.9997 is in reasonable agreement with the adjusted R^2 of 0.9969. At this R^2-value, the optimal condition that was achieved was 50˚C temperature, 2.5 reaction time, 10:1 methanol to oil ratio, and catalyst loading of 6 wt%. Also, the R^2 value correlates with the predicted and actual values, which is shown in Figure 3. This indicates that the model can be used to navigate within the design space.

Table 3. ANOVA for response surface reduced quadratic model.

Source	Sum of Squares	Df	Mean Square	F value	P-value Prob > F	
Model	2102.71	11	191.16	354.40	0.0414	Significant
A–Temp	0.042	1	0.042	0.078	0.8267	
B–4 Temp	3.60	1	3.60	6.67	0.2352	
C–m/o	122.78	1	122.78	227.64	0.0421	
D–Catalyst	1.45	1	1.45	2.68	0.3489	
AB	1.74	1	1.74	3.23	0.3231	
AC	0.36	1	0.36	0.67	0.5622	
AD	32.72	1	32.72	60.67	0.0813	
BC	37.08	1	37.08	68.74	0.0764	

Continued

BD	1.06	1	1.06	1.97	0.3938
CD	12.69	1	12.69	23.54	0.1294
A^2	0.81	1	0.81	1.50	0.4357
Residual	0.54	1	0.54		
Cor Total	2103.25	12			

3.2. Definitive Screen Design 3D Surface Expert

As shown in Figure 4(a), at a catalyst loading of 3 wt %, and temperature ranges between 48 - 55˚C the biodiesel yield increases from 30 - 98%. Also, at the catalyst loading of 6 wt %, the biodiesel yield attains 97% when the temperature is at 47˚C, but as the temperature increases yield of biodiesel decreases to 35%. Figure 4(b) displays the time and temperature profile in relation to biodiesel yield. At the time 1 h, temperature increases from 51 - 60˚C, the biodiesel yield remains constant. Then, between temperatures 45 - 57˚C, the biodiesel yield increases 35 - 80%. However, at 4 h, between 54 - 60˚C, the biodiesel yield also remained constant, but the biodiesel also attained a yield of 80% at a temperature 47˚C and remained constant till the temperature decreased to 45˚C. Figure 4(c), a methanol to oil ratio of 5 w/w, as the temperature slightly increases between 45 - 60˚C, the biodiesel yield remains slightly constant. However, with a methanol to oil ratio of 10 w/w, the biodiesel yield attains 98% when the temperature is 45˚C, but as the temperature increases, the yield of biodiesel tends to decrease slightly. At a catalyst loading of 6 wt %, as the heating time increases from 1 - 4 h according to the illustration in Figure 4(d), the biodiesel yield also increases from 41 - 72%. Furthermore, at a catalyst weight of 3, biodiesel attains 90% when the time is 1 h, but as the time increases, the yield of biodiesel decreases slightly. Figure 4(e) shows that, with a catalyst loading of 3 wt %, the methanol to oil ratio increases from 5 - 8 wt/wt, the biodiesel yield also increases from 30 - 98%, and then remains constant between 9 - 10 wt/wt. Also a catalyst loading of 6 wt%, the biodiesel yield remains constant between 6 wt/wt and 7 wt/wt, but later increases as the methanol to oil ratio increases. As represented in Figure 4(f), at 4 h, the methanol to oil ratio increases from 5 - 10 wt/wt, and the biodiesel yield increases significantly from 39 - 99%. Nevertheless, at 1 h, the biodiesel yield first remains constant and later attains 98%, when the methanol to oil ratio is 9.8 wt/wt.

3.3. Kinetic Studies of the Reaction

The kinetics studies of the reactions were carried out at optimal conditions. Figure 5 shows the conversion of biodiesel at different temperatures (45˚C, 50˚C, 55˚C, 60˚C) within the time limit of 1 - 4 h. These conversion values were employed to determine the rate constants for each temperature used, and the gradients 0.381, 0.138, 0.1019, and 0.132, each possessing an R-squared value of 0.987, 0.933, 0.934, and 0.970, as reported in Figure 6. The plots show that the reaction takes the path

of a first-order reaction. In order to determine activation energy and the exponential factor, the Arrhenius equation was followed, and the obtained values are 69.48 kJ/mol and 421.257 s^{-1}, respectively, as shown in Figure 7. The value of the activation energy and exponential factor depends on the types of feedstocks, catalysts, and the oil used.

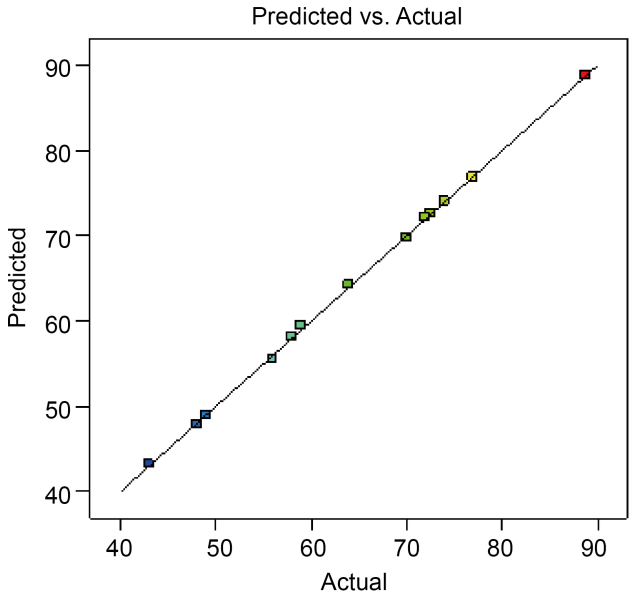
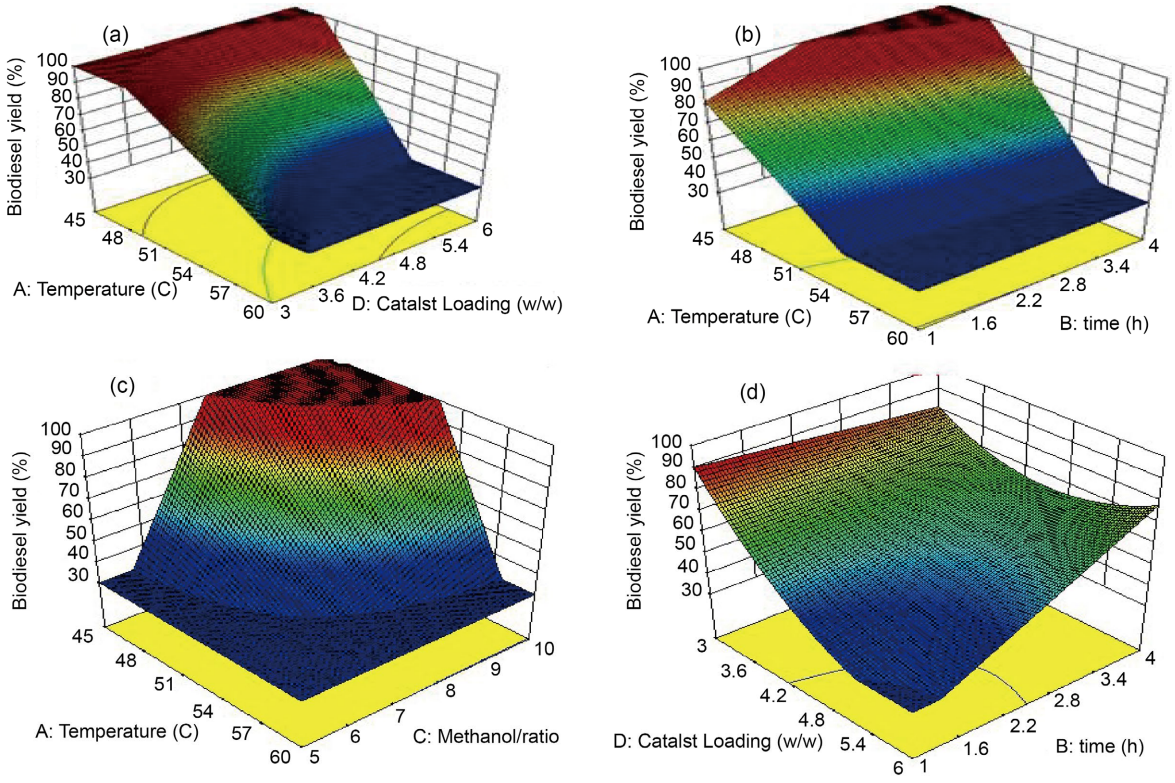


Figure 3. The graph of predicted and Actual value.



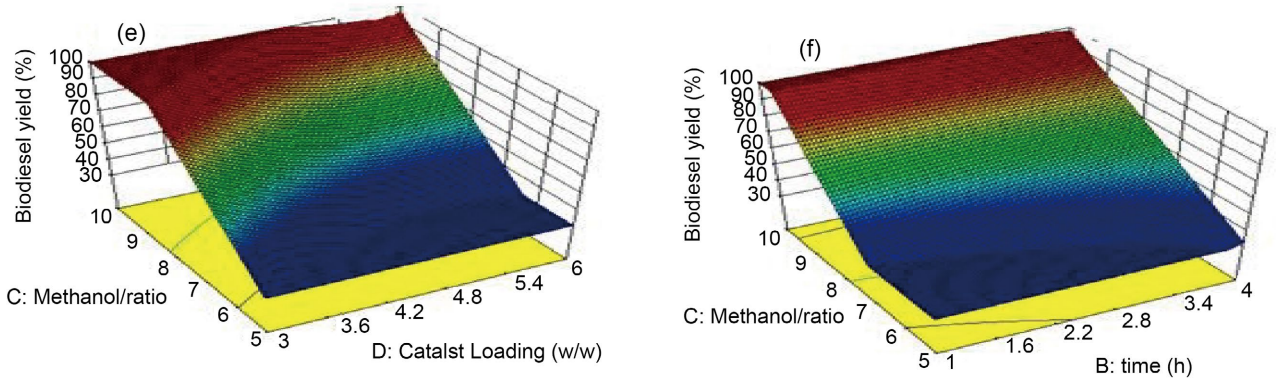


Figure 4. Response Surface Plot for the Design Variables and Biodiesel Yield.

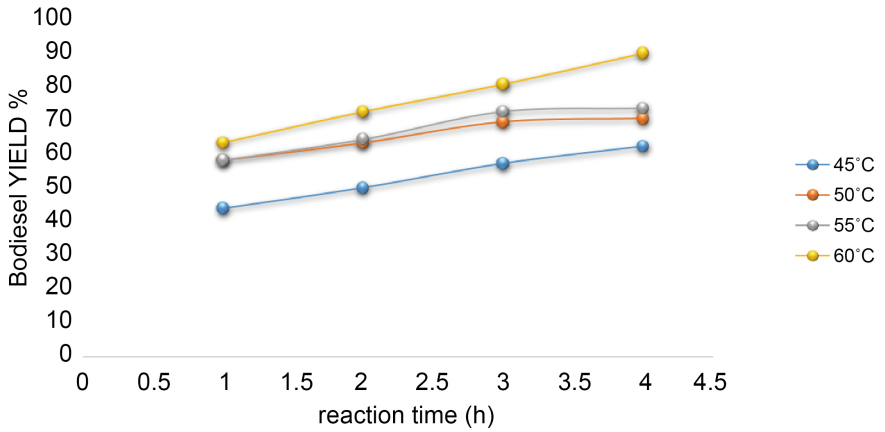


Figure 5. The plot of biodiesel conversion against time.

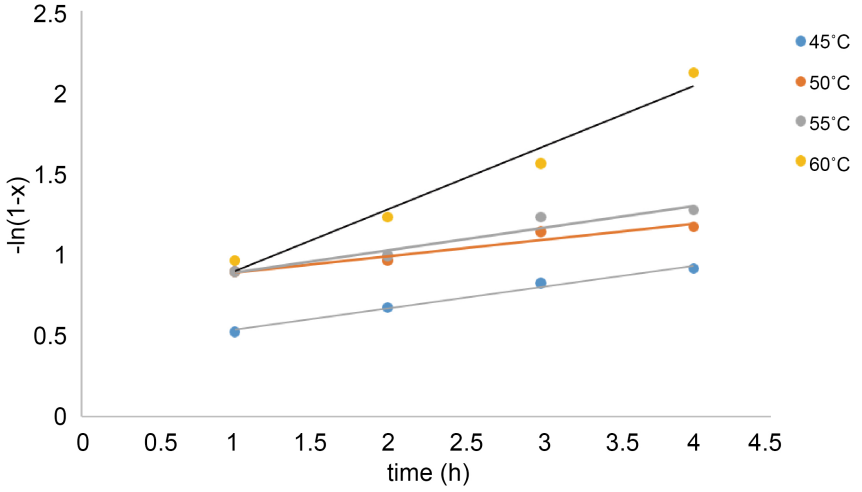


Figure 6. The plot of $-\ln(1-x)$ against t.

3.4. Reusability Studies of the Catalyst

In order to investigate the stability and reusability of the developed catalyst, the catalyst was examined under the optimum conditions of the reaction with a time

of 2.5 h, a temperature of 50˚C, a methanol-to-oil ratio of 10:1, and a catalyst loading of 6 wt%. The results showed that the catalyst may be reused for four times. It could be result of the leaching or deposition of glycerol on the active site of the catalyst. The chart in Figure 8. shows the biodiesel yield after four cycle usage. The reduction in activity is attributed to these phenomena based on theoretical expectations and observed trends.

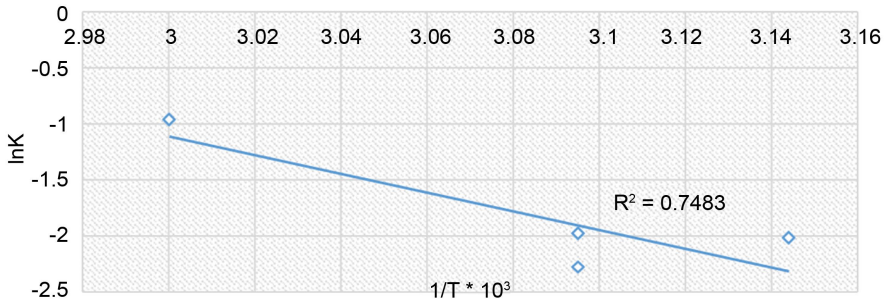


Figure 7. Arrhenius Plot of $\ln(K)$ against $1/T*10^3$.

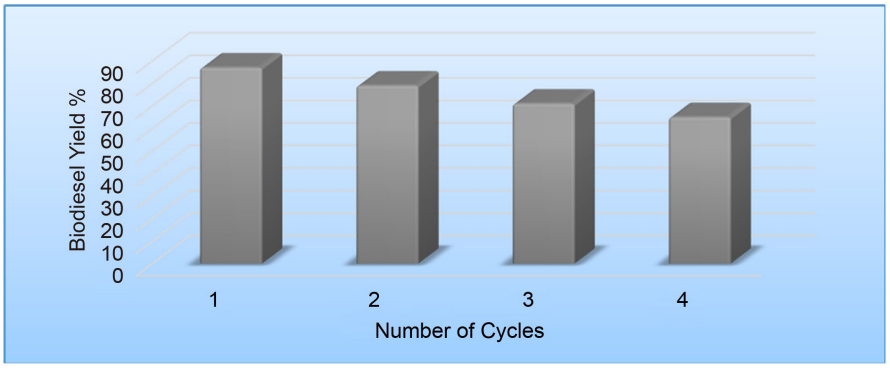


Figure 8. The reusability studies of the reaction.

4. Conclusions

Castor bean seeds contain a significant quantity of oil, making them ideal for biodiesel manufacturing. CPA has been demonstrated to be an excellent catalyst for the transesterification of castor oil. Definitive surface screening was used to maximize biodiesel yield, emphasizing critical parameters such as methanol/oil ratio, catalyst loading, temperature, and reaction duration. The ideal parameters were determined using a definite screen design: 50˚C, 2.5 hours of reaction duration, a methanol/oil ratio of 10:1, and a catalyst loading of 6 weight percent.

These conditions produced a high biodiesel production of 88%, demonstrating the effectiveness of the experimental design approach.

Furthermore, oil was extracted from the seeds using Soxhlet extraction, resulting in 42% castor oil. The catalyst was produced using appropriate thermal treatment and displays strong properties for trans-esterification. Moreover, the catalyst demonstrated reusability for up to four cycles, indicating a potential application in sustainable biodiesel synthesis.

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Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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Symbols Used

y1	Weight of castor beans before extraction
y2	Weight of castor beans after extraction

Abbreviations

ASTM	American Society for Testing and Materials
CPA	Cocoa pod ash
CPH	Cocoa pod husk
EN	European norm
FAME	Fatty acid methyl esters
HSC	Heterogenous solid catalyst
ICCO	International Cocoa Organization
SNI	Indonesian National Standard
RSM	Response Surface Model
ANOVA	Analysis of Variance