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Using Response Surface Methodology to Optimize Biodiesel Production from Sweet Almond (*Prunusamygdalus Dulcis*) and Jatropha (*Jatropha Curcas*) Seed Oils

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Abstract

The depletion of natural resources and the negative impact of oil on the environment have sparked interest in biodiesel as an alternative source of energy. Indigenous vegetable oils have the potential to be used as biodiesel feedstocks. Transesterification of vegetable oils produces biodiesel, which is regulated by numerous inputs factors, such as catalyst dosage, temperature, speed, and time while the density and specific gravity are outputs. Sweet almond (prunusamygdalus dulcis) and jatropha (jatropha curcas) seed oils were used to optimize conditions for the transesterification processes using the response surface methodology (RSM). The experimental matrix at different sodium hydroxide doses (0.3 – 1.5 wt %), intensity (500 – 1000 rpm), and time (20 – 60 min) in the presence of fixed molar ratio, and temperature were designed to optimize the biodiesel output variables (yield, specific gravity, and density).The analysis of variance (ANOVA) showed results for refined sweet almond biodiesel (RSAB) at catalyst (0.554 wt %), speed (750 rpm), time (40 min), giving the optimization solution with the specific gravity (0.995 g/cm3), density (1.230 g/cm3) with the yield of 83.304% for RSAB. Whereas the RJB had the optimum catalyst of (0.3 wt %,), speed (500 rpm), time (44.1 min), with the specific gravity (0.964 g/cm3), density (0.884 g/cm3), and the biodiesel yield of 96.4%. The estimated biodiesel yields vary by 13.096% under these reaction conditions. According to ANOVA statistics, the catalyst dose has a substantial effect on biodiesel yields, and these biodiesels could be employed as an environmentally friendly alternative to diesel.

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1. Introduction

Biodiesel is a diesel fuel made from animal or vegetable oils that emits less soot, carbon dioxide, and particulate matter during combustion, making it environmentally benign [1-2]. It is made from trans esterifying vegetable oil or animal fat and consists of long-chain mono-alkyl esters. One of the most intriguing ways to manufacture renewable liquid fuel for transportation is biodiesel [3]. Biodiesel is a domestically produced renewable alternative diesel fuel manufactured from a variety of fats and oils by a transesterification reaction. It is made up of the alkyl esters, mainly methyl esters, of the parent oil or fat's fatty acids [4]. Biodiesel has a heating value that is approximately 88 percent of standard petrodieselfuel. Biodiesel is a sustainable fuel that is geared toward agriculture. It is non-toxic and biodegradable. Non-edible vegetable oils like sweet almonds (*prunusamygdalus dulcis*) and jatropha (*jatropha curcas*) were chosen as feedstocks since they are not consumed by humans, the plants can grow in deficient soils, and their production is lower than that of other energy crops [5-7]. Compared to acid catalyzes, this route has shorter reaction times and lower catalyst costs. On the other hand, alkaline catalysis, on the other hand, has the disadvantage of being extremely sensitive to both water and free fatty acids in the oils. Base catalyzes the transesterification process and any strong base capable of deprotonating the alcohol (NaOH, KOH, sodium methoxide) can work, although sodium and potassium hydroxides are the best options [8]. The water promotes unfavorable base hydrolysis; the process must be kept completely dry. It's vital to remember that the acid or base isn't consumed during the transesterification reaction; thus, they're not reactants, but catalysts and they may be readily recovered by washing them off afterward [9]. The goal is to see how catalyst dosages, reaction speed, and time in the presence of a fixed molar ratio, mixing intensity, and reaction temperature affect the trans-esterification process as shown in Figure 1.

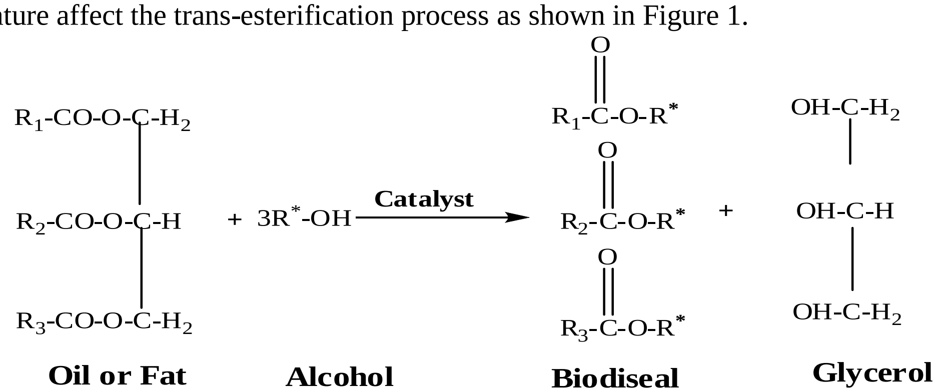


Figure 1: General chemical equation for biodiesel formation (trans-esterification).

2. Experimental Procedure

Standard compounds were obtained from Sigma Aldrich (Gillingham Dorset, UK) andLGC standards (Teddington Middlesex, UK. Fully matured two seeds of Sweet Almond (*Prunusamygdalus dulcis*) and Jatropha (*Jatropha curcas*) were collected from farms and markets in Kwara State. Sample collection and preparation, pretreatment, refinement and trans-esterification processes followed by the physicochemical properties of crude sweet almond oil (CSAO), refined sweet almond oil (RSAO), crude jatropha oil (CJO) and refined jatropha oil (RJO) carried out using Association of Official Analytical Chemists -AOAC [9] and American Society for Test and Material - ASTM (D6751-09) standard methods. Pre-treatment and refinement to ensure the levels of the free fatty acids (FFAs) of the two feedstocks were carried out to ensure the percentages of FFAs were <0.5% [10-11].

2.1. Using the Response Surface Method to Improve Biodiesel Stability

The RSM approach is used to establish correlations between independent and response variables. Box and Wilson [12] were the first to develop a model or optimal response for experimental data, but other ways to optimize processes have boosted its practical application.

2.2. Design of Experiments (DoE) for Biodiesel Optimization

The production variables for the production of biodiesels refined RSAO and RJO include NaOH catalyst doses, mixing speed, reaction temperature at fixed methanol to oil molar ratio, and time. The design of experiments (DoE) established input ranges were as provided in Table 1 to obtain the experimental outputs or responses (yield, specificgravit, and density) [10].

Table1: Design levels with independent variables.

Production independent factors	
Temperature (^{o}C)	60
Speed (rpm)	500, 750, 1000
NaOH Catalyst (%)	0.3, 0.9, 1.5
Molar ratio	1:6
Time (min)	20, 40, 60

2.3. Statistical Analysis

A response approach was used to evaluate the experimental data collected using the response surface methodology (RSM) ofthe Box-Behnken design. A multiple regression methodology is used to correlate the response variable with the independent variables using the polynomial equation developed by Design Expert Version 11 (stat-Ease Inc., USA), which is elevated to the second order. To determine the best conditions for the yield of methyl esters, statistical analysis of the data was carried out, including analysis of variance (ANOVA), regression analysis, and response surface plots of the interaction influence of variables. With ANOVA, the p-value can be computed for each of the models. The p-value of 0.05 for most process variables was beneficial when the values were less than 0.0500, indicating that model terms were significant.The statistical tool used was chosen because it has the three minimal categories of input and response variables, as well as projected and experimental values that are required for the adequacy assessment.

3. Results and Discussion

3.1Experimental effects of catalyst doses, speed, and time on output responses.

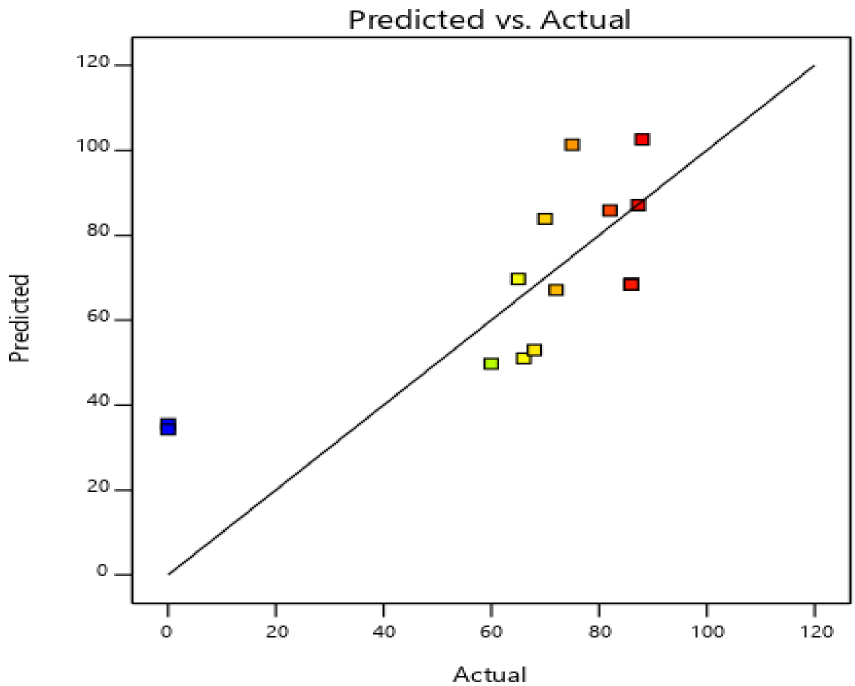
In this work, the experimental effects of catalyst doses on swalmond-based biodiesel, at a constant oil to themolar ratio (6:1), temperature (60^{o}C), but at different catalyst loading (0.3 – 1.5), speed (500 – 1000rpm), and time (20 – 60min) were studied The experimental results obtained for biodiesels yield, specific gravity and density were subjected to the analyses of variance (ANOVA) using Box–Behnken DoE design.

3.1.1. Sweet almond-basedBiodiesel Optimization Analyses

Table 2 shows the experimental outputs for yield, specific gravity, and density using Box–Behnken DoE design [20]. The Design-Expert software generated the maximum number of runs based on the three levels of inputs. Figure 2 shows the relationship between the actual values that were obtained experimentally (Table 2) and the predicted different models' values of yield (Figure 2a), specific gravity (Figure 2b), and density (Figure 2c).

Table 2: Design experimental matrix at different catalyst concentrations, speeds and times.

Run	**Factor 1**	**Factor 2**	**Factor 3**	**Response 1**	**Response 2**	**Response 3**
	A:Catalyst	B:Speed	C:Time	Yield	Specific Gravity	Density
	G	Rpm	min	%	g/cm^3, 30^{o}C	g/cm^3@ 40^{o}C
1	0.9	750	40	86	0.966	3.939
2	0.9	750	40	86	0.966	3.939
3	0.3	750	20	88	0.976	4.058
4	0.3	750	60	82	0.972	3.998
5	0.9	500	60	72	0.977	4.23
6	0.9	1000	20	65	0.926	3.872
7	0.9	500	20	70	0.972	4.801
8	0.3	500	40	75	0.969	4.336
9	0.9	750	40	86	0.966	3.939
10	1.5	1000	40	0	0	0
11	1.5	500	40	60	0.963	4.073
12	1.5	750	60	0	0	0
13	0.9	1000	60	68	0.977	4.888
14	0.3	1000	40	87.3	0.977	3.999
15	1.5	750	20	66	0.972	5.019
16	0.9	750	40	86	0.966	3.939
17	0.9	750	40	86	0.966	3.939


Predicted vs. Actual
Predicted
Actual
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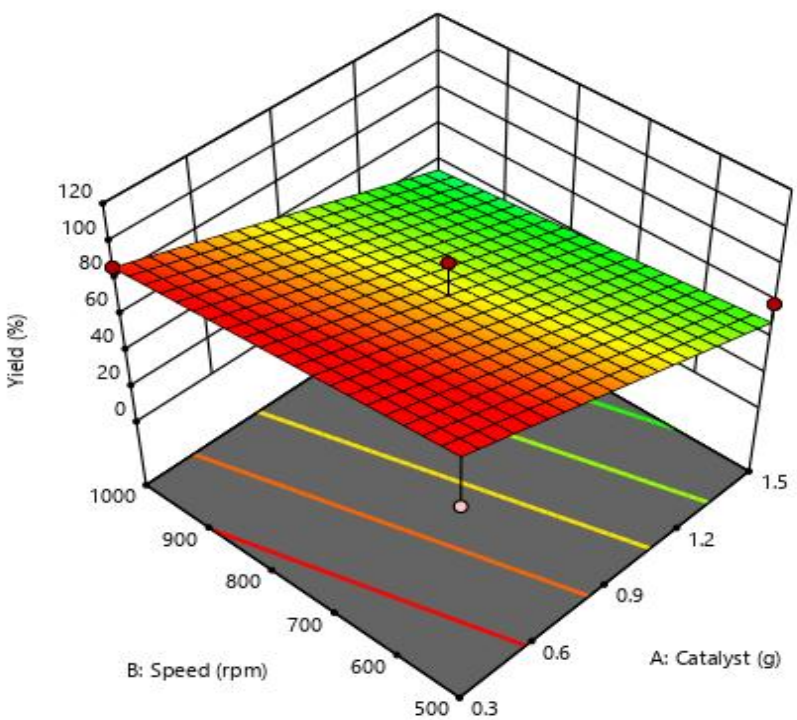
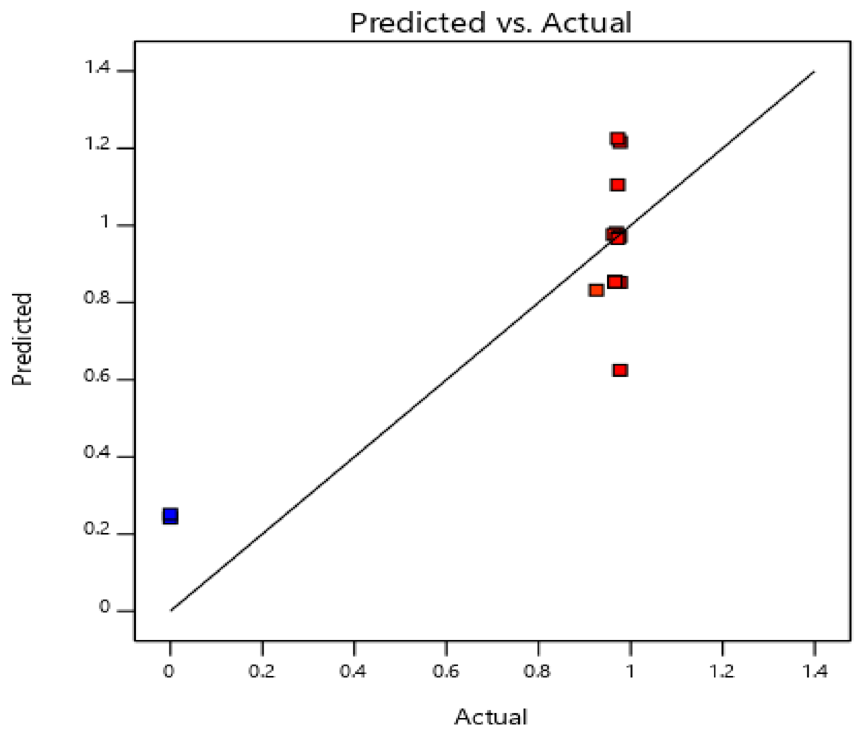
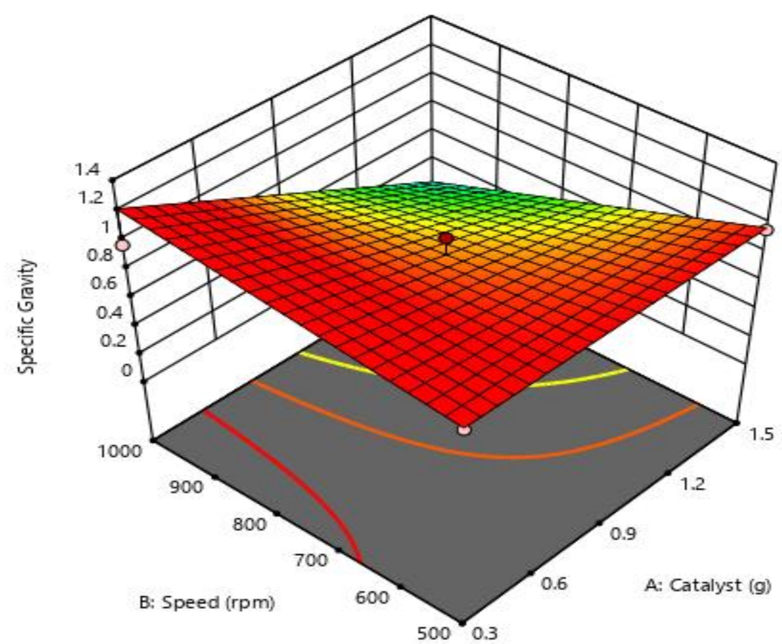
100
80
60
40
20
0
0
20
40
60
80
100
120

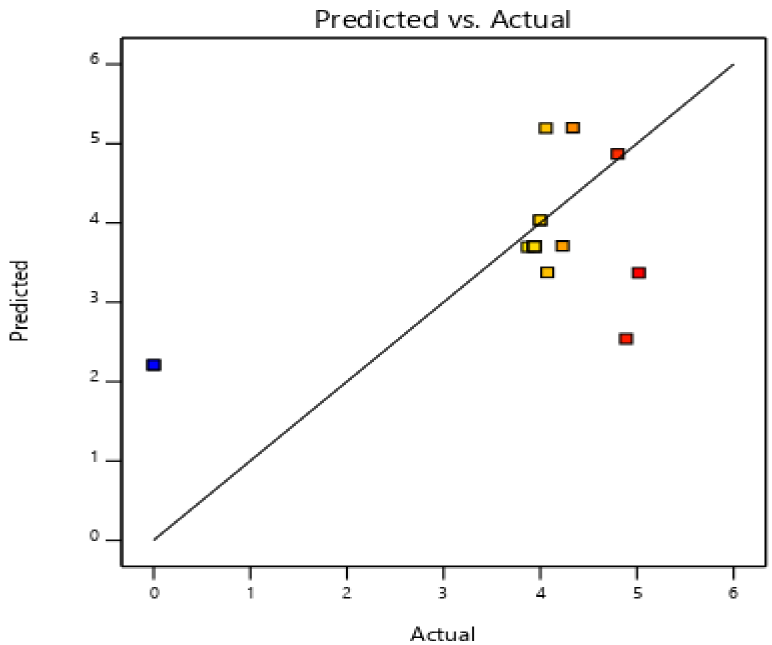

Yield (%)
120
100
80
60
40
20
0
1000
900
800
700
600
500
B: Speed (rpm)
0.3
0.6
0.9
1.2
1.5
A: Catalyst (g)

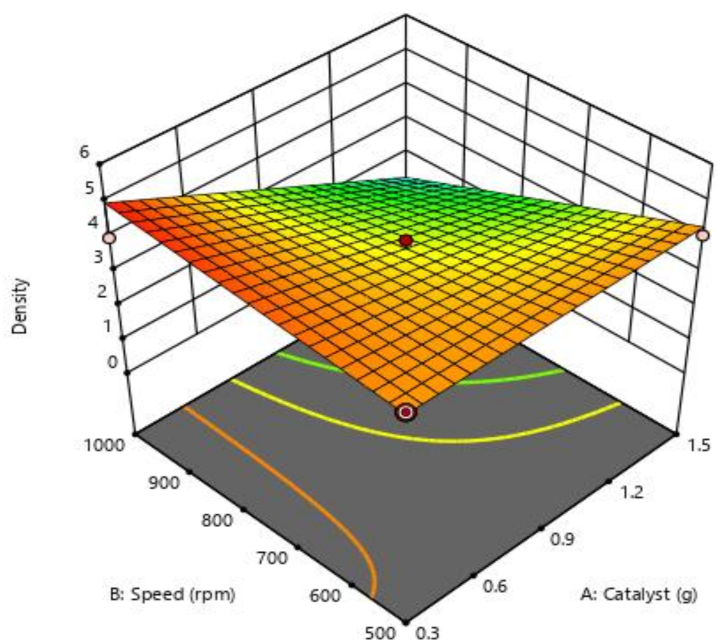
(a)Yield





(b) Specific Gravity





(c) Density

Figure 2: Scatter diagram: (a) yield; (b) specific gravity; (c) density with the corresponding 3D surfaces.

3.2. Analysis of Variance (ANOVA)

The equation shows the second polynomial function in terms of actual factors that were obtained to model yield, specific gravity, and density.Final equations in terms of actual factors:

$$\mathbf{Yield} = +145.12316 - 42.97917\mathbf{Catalyst} - 0.028350\mathbf{Speed} - 0.418750\mathbf{Time} \quad (1)$$

$$\mathbf{Specific\ Gravity} = +0.076901 + 1.61229\mathbf{Catalyst} + 0.000864\mathbf{Speed} + 0.010675\mathbf{Time} - 0.001618\mathbf{Catalyst} \times \mathbf{Speed} - 0.020167\mathbf{Catalyst} \times \mathbf{Time} + 2.30000\mathrm{E}-06 \quad (2)$$

$$\mathbf{Density} = +2.44475 + 7.28188\mathbf{Catalyst} + 0.000090\mathbf{Speed} + 0.004506\mathbf{Time} - 0.006227\mathbf{Catalyst} \times \mathbf{Speed} - 0.103312\mathbf{Catalyst} \times \mathbf{Time} + 0.000079\mathbf{Speed} \times \mathbf{Time} \quad (3)$$

The results obtained from Tables 3 - 7 using various input factors (NaOH catalyst, speed and, time) and the combination of processes that were studied, showing desirability functions from three different criteria. The optimization solutions found according to the biodiesel optimization scenario are shown in Table 7. In this work, the average input variables such asthe catalyst (0.554 wt.%), speed (750 rpm), time (40 min), gave the optimization solution with the specific gravity (0.995 g/cm^3), density (1.230 g/cm^3) and the biodiesel volume yield of 83.304%, with the selected overall desirability of 0.904. A statistical model was developed and used to forecast optimum conditions for methanol transesterification. The oil molar ratio, catalyst concentration, and reaction temperature were 10:1, 1%, and 45 °C, respectively, and a statistical model was developed and used to forecast optimum conditions for methanol transesterification. The reaction time and agitation speed were set to 60 minutes and 600 revolutions per minute, respectively. The highest biodiesel yield was estimated to be 96.12 % of the oil volume based on these parameters [13-17].

Table 3: ANOVA Table for the "Yield" linear model.

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	6282.95	3	2094.32	4.78	0.0186	**Significant**
A-Catalyst	5319.96	1	5319.96	12.14	0.0040	
B-Speed	401.86	1	401.86	0.9171	0.3557	
C-Time	561.13	1	561.13	1.28	0.2782	
Residual	5696.41	13	438.19			
Lack of Fit	5696.41	9	632.93			
Pure Error	0.0000	4	0.0000			
Cor Total	11979.36	16				

Table 4: ANOVA Table for the "Specific Gravity" 2FI model.

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	1.18	6	0.1969	4.17	0.0233	significant
A-Catalyst	0.4797	1	0.4797	10.15	0.0097	
B-Speed	0.1253	1	0.1253	2.65	0.1345	
C-Time	0.1058	1	0.1058	2.24	0.1654	
AB	0.2357	1	0.2357	4.99	0.0495	
AC	0.2343	1	0.2343	4.96	0.0501	
BC	0.0005	1	0.0005	0.0112	0.9178	
Residual	0.4724	10	0.0472			
Lack of Fit	0.4724	6	0.0787			
Pure Error	0.0000	4	0.0000			
Cor Total	1.65	16				

Table 5: ANOVA Table for the "Density" 2FI model.

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	22.35	6	3.72	3.44	0.0415	significant
A-Catalyst	6.66	1	6.66	6.14	0.0326	
B-Speed	2.74	1	2.74	2.53	0.1430	
C-Time	2.68	1	2.68	2.48	0.1466	
AB	3.49	1	3.49	3.22	0.1030	
+AC	6.15	1	6.15	5.67	0.0385	
BC	0.6296	1	0.6296	0.5809	0.4636	
Residual	10.84	10	1.08			
Lack of Fit	10.84	6	1.81			
Pure Error	0.0000	4	0.0000			
Cor Total	33.19	16				

Table 6: Constraints.

Name	Goal	Lower Limit	Upper Limit	Lower Weight	Upper Weight	Importance
A:Catalyst	minimize	0.3	1.5	1	1	4
B:Speed	is target 750	500	1000	1	1	3
C:Time	is target =40	20	60	1	1	3
Yield	is target =88	0	88	1	1	3
Specific Gravity	maximize	0	0.977	1	1	3
Density	minimize	0	5.019	1	1	3

Table 7: Optimization solutions found according to the biodiesel optimization scenario.

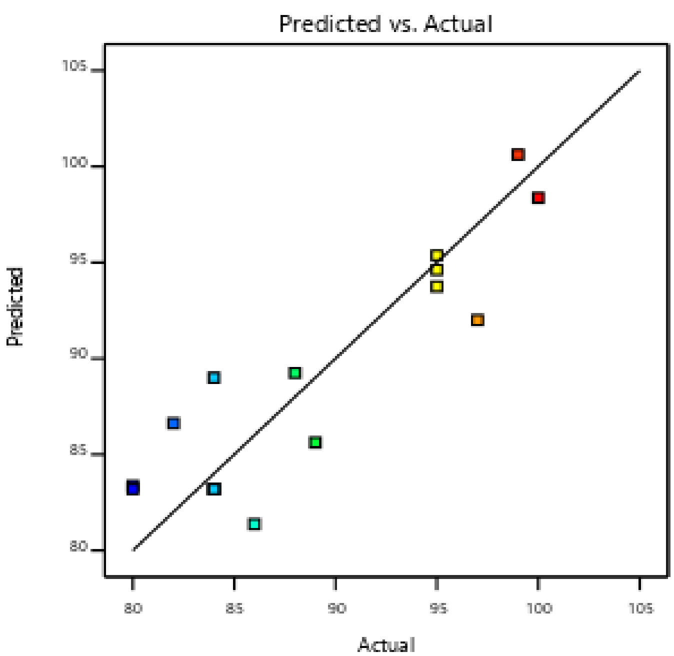
Number	Catalyst	Speed	Time	Yield	Specific Gravity	Density	Desirability	
1	**0.554**	**750.000**	**40.000**	**83.304**	**0.995**	**1.230**	**0.904**	**Selected**
2	0.558	750.002	40.000	83.113	0.993	1.224	0.904	
3	0.550	750.000	40.000	83.493	0.997	1.237	0.904	
4	0.545	749.996	39.999	83.701	0.999	1.244	0.904	
5	0.566	750.001	40.000	82.764	0.990	1.211	0.904	

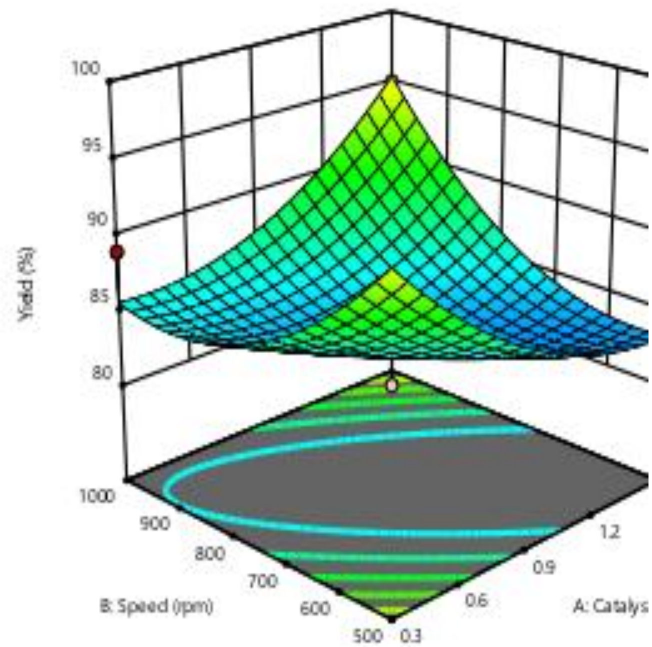
3.1.2. Jatropha based Biodiesel Optimization Analyses.

Table 8 shows the experimental outputs for yield, specific gravity, and density using Box–Behnken DoE design.The Design-Expert software generated the maximum number of runs based on the three levels of inputs. Figure 3 shows the relationship between the actual values and the predicted different model's values of yield (Figure 3a), specific gravity (Figure 3b), and density (Figure 3c).The results obtained from Tables 9 - 12 using various input factors (NaOH catalyst, speed, and time) and the combination of the process that were studied, show desirability functions from three different criteria. The optimization solutions found according to the biodiesel optimization scenario are shown in Table 13.The ANOVA showed results that indicated the important factors in the jatropha based biodiesel productionwith average input variables such as the catalyst (0.300 wt.%), speed (500 rpm), time (44.085 min), gave the optimization solution with the specific gravity (0.964 g/cm^3), density (0.884 g/cm^3) and the biodiesel volume yield of 96.4 %, with the selected overall desirability of 0.896. The Response Surface Methodology is used to optimize the production of jatropha biodiesel at an ideal temperature of 60 °C and a catalyst loading of 4 wt% for a reaction duration of 6 hours, both reactions provided 96 %yield and conversion. The findings imply that an optimization study of Jatropha curcas hydrolysate for yield and conversion of fatty acid methyl esters can be carried out using Design Expert 6.0.8's face-centered central composite design [18]. Similarly, the biodiesel was made from pure triglycerides in the presence of a 4-DBSA catalyst, utilizing methanol under microwave heating to speed up the transesterification step using Response Surface Methodology, they were able to optimize production and assessed the quality of papaya (Carica papaya) biodiesel [19].A transesterification process was used to produce methyl ester from jatropha algae oil, and the process was optimized using a response surface approach based on the BoxBehnken Design. An optimum biodiesel yield of 96% was obtained at a molar ratio of 1:10, a reaction temperature of 53°C, a catalyst concentration of 0.3 wt%, and a reaction time of 172 minutes [20-23].

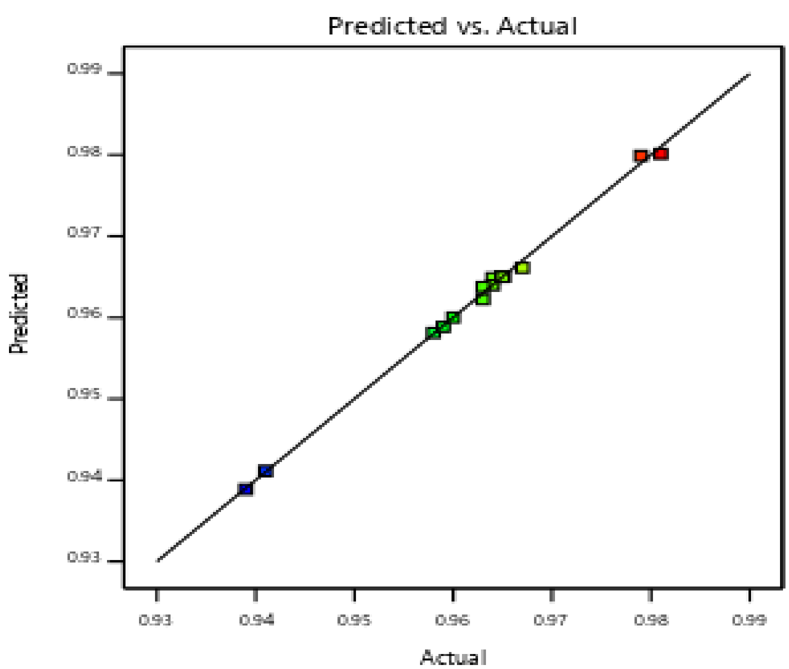
Table 8: Design experimental matrix at different catalyst concentrations, speeds, and times.

	Factor 1	**Factor 2**	**Factor 3**	**Response 1**	**Response 2**	**Response 3**
Run	**A:Catalyst**	**B:Speed**	**C:Time**	**Yield**	**Specific Gravity**	**Density**
	G	rpm	Min	%	g/cm^3, 30^{o}C	g/cm^3@ 40^{o}C
1	0.9	750	40	80	0.965	3.091
2	0.9	1000	60	82	0.96	3.192
3	0.9	500	20	86	0.964	2.704
4	0.3	500	40	95	0.967	2.915
5	0.9	750	40	84	0.965	3.091
6	1.5	500	40	80	0.958	0.327
7	0.9	750	40	84	0.965	3.091
8	0.9	750	40	84	0.965	3.091
9	1.5	750	20	88	0.959	2.993
10	0.3	750	20	84	0.964	3.091
11	1.5	750	60	97	0.981	2.888
12	0.3	1000	40	89	0.939	3.047
13	0.9	1000	20	100	0.963	2.73
14	0.3	750	60	95	0.941	2.93
15	1.5	1000	40	95	0.979	2.903
16	0.9	750	40	84	0.965	3.091
17	0.9	500	60	99	0.963	2.855




100
95
90
85
80
Yield (%)
1000
900
800
700
600
500
B: Speed (rpm)
0.3
0.6
0.9
1.2
A: Catalys

Yield


Predicted vs. Actual
Predicted
Actual
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0.95
0.96
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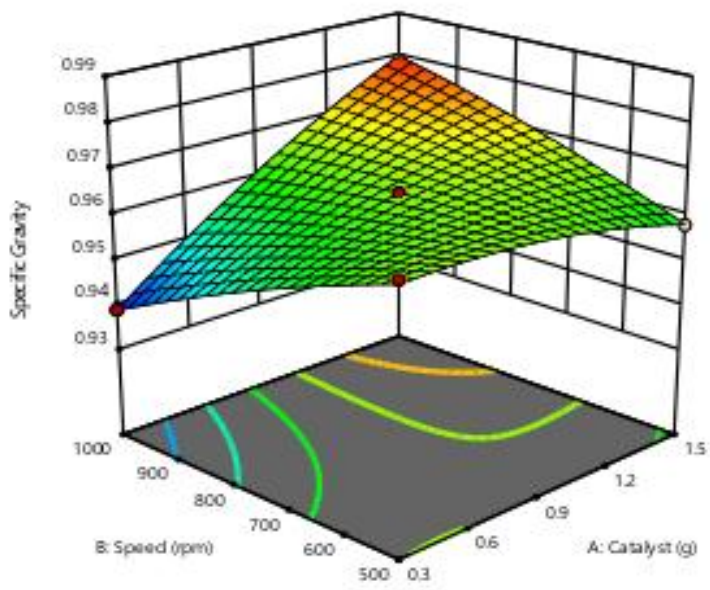
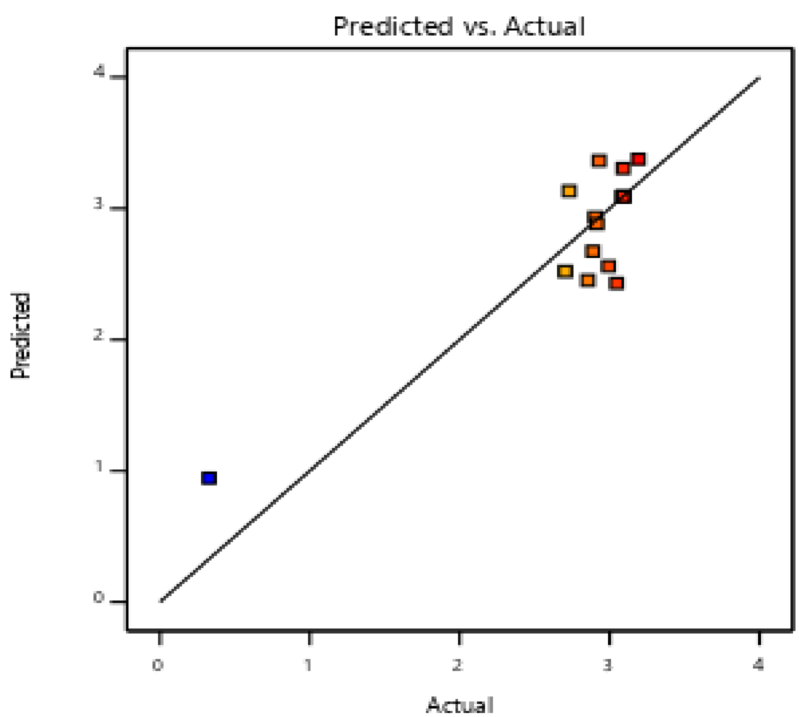
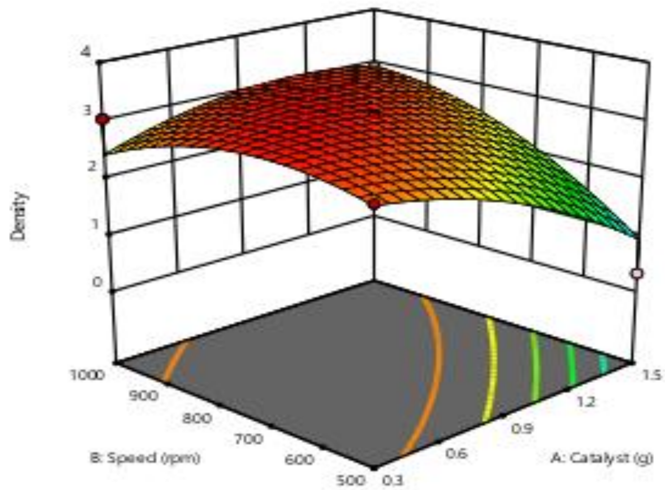
0.98
0.99



Specific gravity





Density

Figure 3: Scatter diagram: (a) yield; (b) specific gravity; (c) density with the corresponding 3D surfaces.

3.2. Analysis of Variance (ANOVA)

The equation shows the second polynomial function in terms of actual factors that were obtained to model yield, specific gravity, and density. Final Equation in Terms of actual factors are as follows:

$$\mathbf{Yield} = +112.36250 - 39.70833\mathbf{Catalyst} - 0.054100\mathbf{Speed} + 0.313750\mathbf{Time} + 0.035000\mathbf{Catalyst} \times \mathbf{Speed} - 0.041667\mathbf{Catalyst} \times \mathbf{Time} - 0.001550\mathbf{Speed} \times \mathbf{Time} + 8.05556\,\mathbf{Catalyst}^2 + 0.000058\,\mathbf{Speed}^2 + 0.012250\mathbf{Time} \quad (4)$$

$$\text{SpecificGravity} = +1.02019 - 0.071250\text{Catalyst} - 0.000039\text{Speed} - 0.000600\text{Time} + 0.000082\text{Catalyst} \times \text{Speed} + 0.000938\text{Catalyst} \times \text{Time} - 1.00000\text{E}-07\text{Speed} \times \text{Time} - 0.007639\text{Catalyst}^2 - 2.40000\text{E}-08\text{Speed}^2 - 2.50000\text{E}-06\text{Time}^2 \quad (5)$$

$$\text{Density} = +1.74678 - 1.98062\text{Catalyst} + 0.008027\text{Speed} - 0.056219\text{Time} + 0.004073\text{Catalyst} \times \text{Speed} + 0.001167\text{Catalyst} \times \text{Time} + 0.000016\text{Speed} \times \text{Time} - 0.955208\text{Catalyst}^2 - 7.18600\text{E}-06\text{Speed}^2 + 0.000571\text{Time}^2 \quad (6)$$

Table 9: ANOVA Table for the “Yield” Quadratic model.

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	599.07	9	66.56	3.40	0.0604	not significant
A-Catalyst	1.12	1	1.12	0.0575	0.8174	
B-Speed	4.50	1	4.50	0.2298	0.6463	
C-Time	28.12	1	28.12	1.44	0.2697	
AB	110.25	1	110.25	5.63	0.0494	
AC	1.0000	1	1.0000	0.0511	0.8277	
BC	240.25	1	240.25	12.27	0.0100	
A^2	35.41	1	35.41	1.81	0.2206	
B^2	56.09	1	56.09	2.87	0.1344	
C^2	101.09	1	101.09	5.16	0.0573	
Residual	137.05	7	19.58			
Lack of Fit	124.25	3	41.42	12.94	0.0158	significant

Pure Error	12.80	4	3.20			
Cor Total	736.12	16				

Table 10: ANOVA Table for the "Specific Gravity" Quadratic model.

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	0.0017	9	0.0002	314.77	< 0.0001	significant
A-Catalyst	0.0005	1	0.0005	896.82	< 0.0001	
B-Speed	0.0000	1	0.0000	24.91	0.0016	
C-Time	3.125E-06	1	3.125E-06	5.15	0.0576	
AB	0.0006	1	0.0006	988.65	< 0.0001	
AC	0.0005	1	0.0005	833.82	< 0.0001	
BC	1.000E-06	1	1.000E-06	1.65	0.2402	
A^2	0.0000	1	0.0000	52.45	0.0002	
B^2	9.474E-06	1	9.474E-06	15.60	0.0055	
C^2	4.211E-06	1	4.211E-06	6.93	0.0337	
Residual	4.250E-06	7	6.071E-07			
Lack of Fit	4.250E-06	3	1.417E-06			
Pure Error	0.0000	4	0.0000			
Cor Total	0.0017	16				

Table 11: ANOVA Table for the "Density" Quadratic model.

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	5.31	9	0.5896	2.54	0.1160	not significant
A-Catalyst	1.03	1	1.03	4.44	0.0730	
B-Speed	1.18	1	1.18	5.08	0.0588	
C-Time	0.0151	1	0.0151	0.0649	0.8063	
AB	1.49	1	1.49	6.44	0.0388	
AC	0.0008	1	0.0008	0.0034	0.9553	
BC	0.0242	1	0.0242	0.1042	0.7562	
A^2	0.4979	1	0.4979	2.15	0.1863	
B^2	0.8493	1	0.8493	3.66	0.0973	
C^2	0.2196	1	0.2196	0.9467	0.3630	
Residual	1.62	7	0.2320			
Lack of Fit	1.62	3	0.5413			
Pure Error	0.0000	4	0.0000			
Cor Total	6.93	16				

Table 12: Constraints.

Name	Goal	Lower Limit	Upper Limit	Lower Weight	Upper Weight	Importance
A:Catalyst	minimize	0.3	1.5	1	1	3
B:Speed	minimize	500	1000	1	1	3
C:Time	minimize	20	60	1	1	3
Yield	maximize	80	100	1	1	3
Specific Gravity	minimize	0.939	0.981	1	1	3
Density	minimize	0.327	3.192	1	1	3

Table 13: Solutionsfor optimization scenarios.

Number	Catalyst	Speed	Time	Yield	Specific Gravity	Density	Desirability	
1	**0.300**	**500.000**	**44.085**	**96.898**	**0.964**	**0.884**	**0.896**	**Selected**
2	0.300	500.000	43.879	96.773	0.964	0.883	0.896	
3	0.300	500.000	44.277	97.011	0.964	0.884	0.896	
4	0.300	500.000	44.751	97.307	0.963	0.886	0.896	
5	0.300	500.253	43.423	96.488	0.964	0.883	0.895	

4. Conclusions

The optimal parameters for biodiesel were studied using the surface response methodology of Box-Behnken Design. It demonstrated the use of the desirability package to combine production factors to generate optimal biodiesel production output variables in three separate combination and optimization scenarios of reaction catalyst, speed, and time. The accuracy of the anticipated methodology was tested using the outcomes of the biodiesel outputs generated from the two sets of combination variables with the optimized outputs of yield, specific gravity, and density quality data were gathered for the two biodiesels. The effects of sodium hydroxide catalyst, speed,and reaction timewere significant parameters that substantially influenced the yield of biodiesels produced from the RSAO and RJO, while the specific gravity and density varied very slightly. The multivariate analysis coefficients (R^2) were provided by the regression model as a variation of the mean, demonstrating that the models were capable of good desirability.

Conflict of Interest

No conflict of interest.

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