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A Study of Modeling and Optimization of Jatropha Oil Biodiesel Synthesis and Its Fuel Quality Characterization

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Abstract
Statistical modeling approach was employed to optimize biodiesel production from Jatropha oil in this work. Precisely, response surface methodology was applied, and the effects of four variables, viz. reaction temperature, catalyst amount, reaction time, and methanol/oil molar ratio, and their attendant interactions were determined in the first step. Central composite rotatable design (CCRD) was used to generate a total of 30 individual experiments, which were designed to study the effects of these variables during acid-catalyzed esterification step of Jatropha oil. A statistically significant quadratic model i.e. $p<0.05$, predicted the lowest acid value of the Jatropha oil to be 1.02 mgKOH g^{-1} with reaction temperature of 60°C, catalyst amount of 1.05 wt.%, and methanol/oil molar ratio of 30 with a reaction time of 60 min. For the alkali-catalyzed transesterification step, the effect of three variables (methanol/oil molar ratio, reaction time and catalyst amount) and their attendant interactions were determined. CCRD was used to generate a total of 21 individual experiments, which were designed to study the effects of these variables during alkali-catalyzed methanolysis of the pretreated Jatropha oil. A statistically significant quadratic model ($p<0.05$) predicted the highest Jatropha Oil Biodiesel (JOB) yield to be 93.76% at reaction time of 30 min, catalyst amount of 0.56 wt.%, and methanol/oil molar ratio of 0.25. Using these variables under experimental condition in four independent replicates, an actual biodiesel yield of 93.03% was achieved. The fuel properties of biodiesel produced were found to be within the ASTM D6751 and DIN EN 14214 biodiesel standard specifications.

Keywords: Biodiesel, *Jatropha curcas*, Transesterification, Esterification, Optimization, Response surface methodology

Introduction

With the exception of hydroelectricity and nuclear energy, the majority of the world energy needs are supplied through petrochemical sources, coal and natural gas. All of these sources are finite and at current usage rates, will be consumed in the future[1]. The depletion of world petroleum reserves and the increased environmental concerns have stimulated recent interest in alternative sources for petroleum-based fuels[2]. Biodiesel, derived from vegetable oil or animal fats by transesterification with alcohol like methanol and ethanol, is recommended for use as a substitute for petroleum-based diesel mainly because biodiesel is an oxygenated, renewable,

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biodegradable and environmentally friendly biofuel with similar flow and combustion properties and also low emission profile[3].

Despite the food vs. fuel challenge facing the world, both edible and non-edible vegetable oils have been successfully used to produce biodiesel with superior qualities to diesel. These feedstocks include soybean oil[4], moringa oil[5], sunflower oil[6], rapeseed oil[7], palm oil[8], Jatropha oil[9], camelina oil[10], neem oil[11,12], and sesame oil[13]. The ideal vegetable oil for biodiesel must be readily available, its plant should be easy to cultivate, and its composition must include a high percentage of monounsaturated fatty acids (C16:1, C18:1), a low proportion of polyunsaturated acids (C18:2, C18:3) and a controlled amount of saturated fatty acids (C16:0, C18:0)[14]. Transesterification of oil or fat to produce a high yield of biodiesel is typically investigated by optimizing the following reaction variables: alcohol/oil molar ratio, catalyst amount, reaction temperature, and reaction time involved in the process. Equally, the effects of the variables and their attendant interactions have been evaluated using response surface methodology (RSM). RSM is a practical statistical tool, which has been applied in research for optimizing various

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processes including pretreatment of the *Jatropha curcas* fruit hull for enzymatic hydrolysis[15], citric acid production[16], complex variable processes[17] and transesterification reaction of vegetable oils.

RSM was used to optimize the transesterification of various types of oil to biodiesel: *Jatropha curcas* (Tiwari et al.[9]), *Moringa oleifera* (Rashid et al.[5]), waste rapeseed oil (Yuan et al.[7]), and animal fat (Jeong et al.[18]). Also, Zhang et al.[19] applied RSM for the optimization of *Zanthoxylum bungeanum* seed oil transesterification to biodiesel using CaO as catalyst. Optimization of transesterification variables for biodiesel production from cottonseed oil using RSM has also been reported[20]. *Jatropha curcas* is a non-edible oil bearing plant widespread in arid, semi-arid and tropical regions of the world. *Jatropha curcas* is a drought-resistant perennial tree that grows in marginal lands and can live over 50 years[21]. *Jatropha curcas* has several benefits, such as its stem can be used as a natural toothpaste and toothbrush, latex from the stem can be used as a natural pesticide and to heal wounds, while its leaves are used as fodder for silkworms[21]. The oil content in *Jatropha curcas* seed is reported to be in the range from 30 to 50% by weight of oilseeds [22,23] and from 45 to 60% by weight of the kernel itself[24].

In this current study, an effort was made to optimize the process conditions for esterification and transesterification of Jatropha oil from a Nigerian cultivar to biodiesel. RSM with central composite rotatable design (CCRD) was applied, and the effect of four variables (catalyst amount, reaction time, reaction temperature and methanol/oil molar ratio) at five levels and their attendant interactions were evaluated in the first step while the effect of three variables (catalyst

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amount, reaction time, and methanol/oil molar ratio) at five levels and their attendant interactions were determined in the second step.

Materials and Methods

Oil and reagents

The Jatropha oil used for this study was obtained from a firm in Lagos. All the chemicals like methanol, potassium hydroxide, anhydrous calcium chloride, sulphuric acid, sodium thiosulphate were of analytical grades.

Two–step acid – base catalyzed transesterification

Crude Jatropha oil when transesterified using KOH catalyst produced a significant amount of soaps from saponification side reaction. This was due to the high level of free fatty acids and small quantity of moisture in the crude Jatropha oil. Therefore, a two-step process, acid-catalyzed esterification followed by alkali-catalyzed transesterification was employed according to the method of Berchmans and Hirata[24].

Acid pretreatment (acid-catalyzed esterification)

The crude oil was weighed, heated at 100°C for about 12 min and mixed with 30 ml of methanol. To the mixture was added 1.5 wt. % of concentrated H_2SO_4. The resulting mixture was then stirred on magnetic hot plate for 1 h at 60°C, after which it was allowed to settle for 2 h in a separating funnel. The pretreated oil was decanted from the methanol-water phase at the top.

Experimental design of biodiesel production

In this work, the central composite rotatable design (CCRD) was employed to optimize the esterification of Jatropha oil in order to reduce its FFA content. A five-level-four-factor design was used to generate 30 experimental runs. Selected variables for the esterification process of pretreatment of the Jatropha oil were methanol/oil molar ratio (X_1), reaction time (X_2), catalyst amount (X_3) and reaction temperature (X_4). The coded levels of the independent variables are given in Table 1. The CCRD was also employed to optimize the transesterification of the pretreated Jatropha oil to biodiesel. A five-level-three-factor design was used to generate 21 experimental runs. These included 14 factorial points, 4 axial points, and 3 central points to provide information regarding the interior of the experimental region, making it possible to evaluate the curvature outcome. Selected variables for the transesterification process from the Jatropha oil were methanol/oil molar ratio (X_1), reaction time (X_2) and catalyst amount(X_3). The coded levels of the independent variables are given in Table 2.

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Table 1 Independent variables used for central composite design in esterification of Jatropha oil

Variables	Symbol	Coded factor level				
		-2	-1	0	1	2
Methanol/Oil molar ratio	X_1	20	30	40	50	60
Reaction Time (min)	X_2	15	30	45	60	75
Catalyst Amount (v/v)	X_3	0.9	0.95	1	1.05	1.1
Reaction Temperature (^oC)	X_4	30	40	50	60	70

Table 2 Independent variables used for central composite design in transesterification of pretreated Jatropha oil

Variable	Symbol	Coded factor levels				
		-1.68	-1	0	1	1.68
Methanol/Oil molar ratio	X_1	0.18	0.20	0.23	0.25	0.27
Reaction Time (min)	X_2	26.59	30.00	35.00	40.00	43.41
Catalyst amount (wt.%)	X_3	0.55	0.56	0.58	0.60	0.61

The experiments were randomized to minimize the effects of unexplained variability in the observed response due to extraneous factors.

Jatropha biodiesel production procedures

The alkali-catalyzed reaction was carried out according to the work of Schinas et al.[25]. The transesterification reaction of pretreated Jatropha oil was carried out in a one-neck glass reactor (250-ml capacity). The reactor was charged with a known weight of the pretreated oil sample, which was preheated to the desired temperature. A known weight of KOH pellets (catalyst) was dissolved in a specific volume of methanol and was transferred into the preheated oil on the hot plate with a magnetic stirrer. The resulting mixture was transferred into a separating funnel for glycerol and methyl ester separation. The glycerol was allowed to separate by gravity for 24 h. Two phases separated clearly, the less dense methyl ester at the top and the denser phase of glycerol at the bottom. The glycerol was tapped off the bottom of the separating funnel, leaving behind methyl ester in the separating funnel. The methyl ester was washed with distilled water to remove residual catalyst, glycerol, methanol, and soap. The methyl ester was further dried over a heated $CaCl_2$ powder. The final product was the Jatropha Oil Biodiesel (JOB), and the yield was calculated gravimetrically as described in Equation 1:

$$JOB\ yield\ (wt.\%) = \frac{weight\ of\ biodiesel\ produced}{weight\ of\ jatropha\ oil\ used} \quad (1)$$

Statistical data analysis

Jatropha biodiesel production data were analyzed statistically using response surface methodology, in order to fit the quadratic polynomial equation generated by the Design Expert

[25] Schinas, P., Karavalakis, G., Davaris, C., Anastopoulos, G., Karonis, D., Zannikos, F., Stournas, S., Lois, E. (2009) Pumpkin (*Cucurbita pepo* L.) seed oil as an alternative feedstock for the production of biodiesel in Greece. *Biomass Bioenerg* 33:44–49.

software version 8.0.3.1 (Stat-Ease Inc., Minneapolis, MN, USA). To correlate the response variable to the independent variables, multiple regressions were used to fit the coefficient of the polynomial model of the response. The quality of the fit of the model was evaluated using a test of significance and analysis of variance (ANOVA). The fitted quadratic response model is described in Equation 2:

$$Y = b_0 + \sum_{i=1}^{k} b_i X_i + \sum_{i=1}^{k} b_{ii} X_i^2 + \sum_{i<j}^{k} b_{ij} X_i X_j + e \quad (2)$$

Where Y is the response variable (JOB yield), b_0 is the intercept value, b_i (i = 1, 2,..., k) is the first-order model coefficient, b_{ij} is the interaction effect, and b_{ii} represents the quadratic coefficients of X_i, and e is the random error.

Jatropha oil and biodiesel characterization

Fuel properties, namely, moisture content, density at 15°C, kinematic viscosity at 40°C, iodine value, acid value, higher heating value, flash point, pour point, cloud point, cetane number, diesel index, API, and aniline point of both Jatropha oil and biodiesel, were determined following standard methods and compared with American and European standards (ASTM D 6751 and DIN EN 14214).

Results and Discussion

Properties of the Jatropha oil

The analysis of the Jatropha oil showed that it had a density of 0.89 kg/m^3, specific gravity of 0.88 and kinematic viscosity of 3.30 mm/s^2. These results are within earlier reported values in the literature[14].

Optimization of Jatropha biodiesel production

The coded factors considered in this work with experimental results, predicted values, as well as the residual values obtained for the acid and alkali-catalyzed stages are shown in Tables 3and 4, respectively.

Eq. 3 was the model equation, which correlates the response (free fatty acid) to the independent variables in terms of actual values.

$$Y = 1.22 - 0.24X_1 - 0.64X_2 - 0.15X_3 - 0.02X_4 - 0.09X_1X_2 + 0.38X_1X_3 - 0.03X_1X_4 + 0.05X_2X_3 + 0.18X_2X_4 + 0.12X_3X_4 + 0.001X_1^2 - 0.06X_2^2 - 0.06X_3^2 - 0.04X_4^2 \quad (3)$$

where Y is the acid value, X_1 is the methanol/oil ratio, X_2 is the reaction time, X_3 is the catalyst amount and X_4 is the temperature.

Table 3 CCRD arrangement and responses for esterification of Jatropha oil

Standard order	X_1	X_2 (min)	X_3 (wt.%)	X_4 (^oC)	Free Fatty Acid (%)	
					Experimental value	Predicted value
1	-1.00	-1.00	-1.00	-1.00	3.27	2.73
2	-1.00	-1.00	1.00	1.00	2.14	1.65
3	0.00	0.00	0.00	0.00	0.99	1.35
4	1.00	-1.00	1.00	-1.00	2.26	0.97
5	0.00	0.00	0.00	2.00	1.97	2.39
6	1.00	-1.00	1.00	1.00	0.96	1.79
7	0.00	-2.00	0.00	0.00	5.02	0.76
8	-1.00	1.00	1.00	-1.00	1.07	0.86
9	1.00	1.00	1.00	1.00	0.56	2.88
10	0.00	0.00	2.00	0.00	0.85	1.80
11	-1.00	-1.00	-1.00	1.00	2.14	1.27
12	-2.00	0.00	0.00	0.00	1.24	0.89
13	0.00	0.00	0.00	0.00	1.30	2.37
14	0.00	2.00	0.00	0.00	0.79	1.77
15	0.00	0.00	0.00	0.00	1.18	0.51
16	-1.00	1.00	-1.00	-1.00	1.35	0.61
17	-1.00	1.00	-1.00	1.00	1.30	1.35
18	2.00	0.00	0.00	0.00	0.79	0.37
19	0.00	0.00	0.00	0.00	1.41	4.02
20	-1.00	1.00	1.00	1.00	0.96	1.48
21	0.00	0.00	0.00	-2.00	1.18	1.42
22	-1.00	-1.00	1.00	-1.00	1.92	0.81
23	0.00	0.00	0.00	0.00	1.02	1.47
24	0.00	0.00	0.00	0.00	1.41	1.38
25	1.00	1.00	-1.00	-1.00	1.69	1.22
26	1.00	1.00	1.00	-1.00	0.79	1.22
27	0.00	0.00	-2.00	0.00	1.69	1.22
28	1.00	-1.00	-1.00	1.00	1.97	1.22
29	1.00	1.00	-1.00	1.00	0.56	1.22
30	1.00	-1.00	-1.00	-1.00	0.40	1.22

Table 4 CCRD and experimental, predicted, and residual values for five-level-three-factor response surface analysis (transesterification step)

Standard order	X_1	X_2 (min)	X_3 (wt.%)	Experimental value (wt.%)	Predicted value (wt.%)	Residual value
1	-1.00	-1.00	-1.00	87.41	86.65	0.76
2	1.00	-1.00	-1.00	92.74	93.77	-1.03
3	-1.00	1.00	-1.00	83.31	80.63	2.68
4	1.00	1.00	-1.00	87.00	87.75	-0.75
5	-1.00	-1.00	1.00	91.00	88.52	2.48
6	1.00	-1.00	1.00	92.08	93.04	-0.96
7	-1.00	1.00	1.00	87.81	85.06	2.75
8	1.00	1.00	1.00	90.54	89.58	0.96
9	-1.68	0.00	0.00	75.51	79.84	-4.33
10	1.68	0.00	0.00	91.51	89.62	1.89
11	0.00	-1.68	0.00	91.43	91.35	0.08
12	0.00	1.68	0.00	80.85	83.38	-2.53
13	0.00	0.00	-1.68	90.00	90.16	-0.16
14	0.00	0.00	1.68	90.99	93.27	-2.28
15	0.00	0.00	0.00	86.55	84.84	1.71
16	0.00	0.00	0.00	84.60	84.84	-0.24
17	0.00	0.00	0.00	83.79	84.84	-1.05
18	0.00	0.00	0.00	84.00	84.84	-0.84
19	0.00	0.00	0.00	84.11	84.84	-0.73
20	0.00	0.00	0.00	87.21	84.84	2.37
21	0.00	0.00	0.00	84.06	84.84	-0.78

Table 5 shows the results of the test of significance for every regression coefficient. The results revealed that the p-values of the model terms were significant, i.e., $p<0.05$. In this particular case, the two linear terms (X_1, X_2) and the one quadratic terms (X_3^2) were all remarkably significant model terms at 95% confidence level. Based on the large F-values and low corresponding p-values, all the linear terms had very strong effects on the biodiesel yield. However, the interaction between methanol/oil molar ratio and reaction time X_1X_2 (p-value<0.0001) and the quadratic function X_1^2 (p-value<0.0001) are the most significant variable.

Table 5 Test of significance for every regression coefficient of CCRD

Source	Sum of Squares	Df	Mean Square	F-value	p-value
X_1	115.63	1	115.63	18.07	<0.0001
X_2	76.69	1	76.69	11.98	0.0053
X_3	11.69	1	11.69	1.83	0.2037
X_1X_2	0.00	1	0.00	<0.0001	0.9989
X_1X_3	3.39	1	3.39	0.53	0.4817
X_2X_3	3.26	1	3.26	0.51	0.4900
X_1^2	0.02	1	0.02	<0.0001	0.9525
X_2^2	11.89	1	11.89	1.86	0.2001
X_3^2	88.62	1	88.62	13.85	0.0034

In order to minimize error, all the coefficients were considered in the design. The model also proved suitable for adequate representation of the real relationship among the selected independent variables (X_1, X_2, X_3).

Table 6 ANOVA of regression equation

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	308.35	9.00	34.26	5.35	0.0057
Residual	70.39	11.00	6.40		
Lack of Fit	58.87	5.00	11.77	6.13	0.0236
Pure Error	11.52	6.00	1.92		
Totals corrected for the mean	378.75	20.00			

R^2 = 93.02% and R^2 (adj) = 80.05%

Results of the ANOVA of the regression equation are presented in Table 6. The model F-value of 5.35 with a low probability value (p = 0.0057) implied a high significance for the regression model[7]. The goodness of fit of the model was checked by the coefficient of determination (R^2), which should be at least 0.80 for the good fit of a model[26]. The R^2 value of 0.9302 indicated that the sample variation of 93.02% for biodiesel was attributed to the independent variables, and only 6.98% of the total variations are not explained by the model. The value of the adjusted determination coefficient (adjusted R^2 of 0.8005) was also very high, supporting a high significance of the model[27,28]. The lack-of-fit term of 0.0236 was significant relative to the pure

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error. The final equation in terms of coded factors for the central composite response surface quadratic model is expressed in Equation 4.

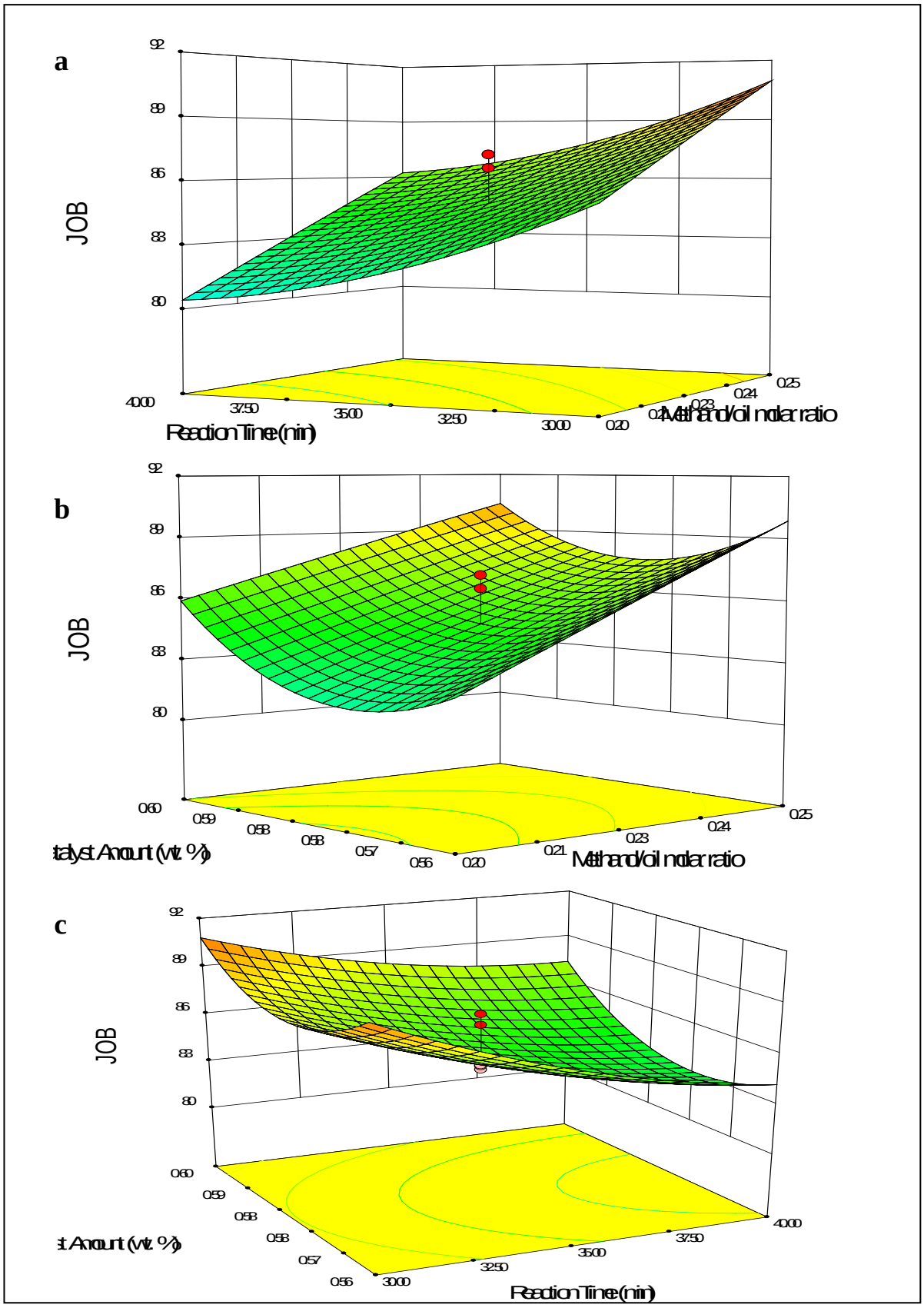
$$Y = 84.84 + 2.91X_1 - 2.37X_2 + 0.93X_3 + 0.00125X_1X_2 - 0.65X_1X_3 + 0.64X_2X_3 - 0.04X_1^2 + 0.89X_2^2 + 2.43X_3^2 \quad (4)$$

All the linear terms except X_2 and the cross-product terms X_1X_2 *and* X_2X_3 had a positive effect on the JOB yield except X_1X_3 while the rest of the surface quadratic model terms had positive effects on the yield except X_1^2. The low values of standard error observed in the intercept and all the model terms demonstrated that the regression model fits the data well, and the prediction was good (Table 7). The variance inflation factor (VIF) obtained in this study showed that the center points are orthogonal to all other factors in the model.

Table 7 ANOVA for response surface quadratic model for intercept

Factor	Coefficient Estimate	Df	Standard Error	95% CI Low	95% CI High	VIF
Intercept	84.84	1.00	0.96	82.74	86.95	-
X_1	2.91	1.00	0.68	1.40	4.42	1.00
X_2	-2.37	1.00	0.68	-3.88	-0.86	1.00
X_3	0.93	1.00	0.68	-0.58	2.43	1.00
X_1X_2	0.00125	1.00	0.89	-1.97	1.97	1.00
X_1X_3	-0.65	1.00	0.89	-2.62	1.32	1.00
X_2X_3	0.64	1.00	0.89	-1.33	2.61	1.00
X_1^2	-0.04	1.00	0.65	-1.48	1.40	1.01
X_2^2	0.89	1.00	0.65	-0.55	2.33	1.01
X_3^2	2.43	1.00	0.65	0.99	3.87	1.01

Generally, the three dimensional response surface plots are graphical representations of the regression equation for the optimization of the reaction variables, and they are presented in Figure 1. Interactions between the reaction time and methanol/oil molar ratio showed JOB yield increases with increasing methanol/oil molar ratio and decreasing time (Figure 1a). Whereas the interaction between catalyst amount and methanol/oil molar ratio showed that JOB yield increases with increasing methanol/oil molar ratio and decreases with increasing catalyst amount to minimum after which the yield increases (Figure 1b). The observation of the interaction between catalyst amount and reaction time was similar to Figure 1b. As the catalyst amount increases with decreasing time, JOB yield decreased to minimum after which the yield increased (Figure 1c). The curvatures' nature of the three dimensional surfaces in Figure 1b and c suggested mutual interactions of catalyst amount with methanol/oil molar ratio and catalyst amount with reaction time. The optimal values of the independent variables selected for the transesterification of JOB were obtained by solving the regression equation (Equation 4) using the Design Expert software.



Response surface plots for optimization of the conditions for JOB production

A statistical model predicted the highest JOB yield of 93.76% at the optimized variables condition: reaction time of 30 min, catalyst amount of 0.56 wt. %, and methanol/oil molar ratio of 0.25. In order to verify the validity of the model, the optimal conditions were applied to three independent replicates, and the average biodiesel yield obtained was 93.03%, which is well within the predicted value for the model equation. Table 8 shows the results of the evaluation of Jatropha oil and fuel properties of the JOB. Most of the fuel properties of JOB from this study compared well with ASTM D6751 and EN 14214 biodiesel specifications.

Table 8 Properties of JOB in comparison to biodiesel specifications standards

Parameters	Unit	Jatropha oil	JOB	ASTM D 6751	EN 14214
Moisture content	-	0.151	0.012	< 0.03	0.02
Kinematic viscosity @ 40°C	mm^2/s	37.655	4.76	1.9-6.0	3.5-5.0
Refractive index	-	1.47	1.45	-	-
Specific gravity @ 25°C	-	0.919	0.844	0.87-0.90	0.85
Density @ 25°C	kg/m^3	919	884	-	860 - 900
%FFA	-	7.05	0.166	-	-
Acid value	mg KOH/g	14.10	0.322	< 0.80	0.50 max
Iodine value	g I_2/100g	29.285	19.035	-	120 max
Peroxide value	meq O_2/kg oil	28.80	7.04	-	-
Cetane number	-	34.365	58.688	48.0 – 65.0	>51
Diesel index	-	33.840	67.622	50.40	-
Higher heating value	mJ/kg	43.480	45.560	-	-
Aniline point	-	150.60	236.60	331.00	-
API	-	22.47	28.58	36.95	-

Conclusions

In the first step of this study, the acid value of the Jatropha oil with very high FFA (7 ± 0.05%) was reduced to 1.02 mgKOH g^{-1} by esterification with methanol-oil ratio of 30 (v/v), H_2SO_4 of 1.05 wt.% (v/v), reaction time of 60 min and reaction temperature of 60°C. The results obtained in the second step of this study using response surface methodology to determine the effects of three reaction variables, namely, methanol/oil molar ratio, catalyst concentration, and reaction time on JOB yield via transesterification, indicate that the biodiesel produced had fuel properties that satisfied both the ASTM D 6751 and EN 1424 standards. A quadratic model predicted the highest JOB yield of 93.76% at the optimized variables condition: reaction time of 30 min, catalyst amount of 0.56 wt. % and methanol/oil molar ratio of 0.25. Using these variables under experimental condition in three independent replicates, an actual JOB yield of 93.03% was achieved. Hence, the work established the usefulness of RSM for optimum conversion of Jatropha seed oil to biodiesel and also suggests that Jatropha oil could be effectively used as feedstock for biodiesel production. The optimized Jatropha biodiesel production using potassium hydroxide as a catalyst could be scaled up to a pilot scale plant.