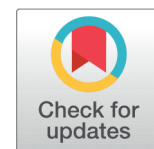


# APPLICATION OF RESPONSE SURFACE METHODOLOGY FOR THE OPTIMIZATION OF BIODIESEL PRODUCTION VIA TRANSESTERIFICATION OF CASTOR OIL WITH CALCIUM OXIDE



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## ABSTRACT

Castor seeds were processed and the oil was extracted from the paste by cold pressing and the yield obtained was 42.80%. Moisture content of 4.38% was found. The crude oil was obtained as clear, viscous, pale-yellow liquid. Transesterification of crude castor oil with methanol to produce biodiesel in the presence of calcium oxide (CaO) as a catalyst was carried out. This study analysed the fuel properties of biodiesel; the FTIR spectrum for the biodiesel revealed the functional groups with characteristics bands, C=O, - (CH<sub>2</sub>) n-, C-O, C=C, OH and C-H in the spectrum. From the result of the FTIR and GC-MS, the methyl ester groups present in the biodiesel produced was determined and 9-Octadecenoic acid-12-hydroxy- methyl ester was identified as the most abundant compound in the biodiesel which have all the functional groups as revealed by the FTIR. The experimental design as well as statistical analysis were done and analysed using Response Surface Methodology (RSM) design expert 12.0 version software which was also used for the optimization of the three process parameters. The optimum conditions for biodiesel production were a reaction temperature of 62<sup>0</sup>C, methanol to oil ratio of 11:1 and a catalyst of 3.5 wt% of oil at 1hour. The biodiesel content under these optimum conditions was 54%. R<sup>2</sup> value of 0.9996 was obtained showing good fitting.

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**Keywords:** Coconut Oil, Sodium Hydroxide, Transesterification, Biodiesel, Purification

## 1. INTRODUCTION

Energy is the prime mover for economic development. Depletion of fossil reserves increases interest to search for alternative fuel, which have same properties as diesel, to replace such fossil fuel (Andrijanto (2012) ). Among the renewable energy sources, biodiesel is ranked the best alternative to diesel fuel (Zavarize et al. (2021) ). Biodiesel has been defined as a mono-alkyl ester of vegetable oils or animal



fats. Biodiesel is best substitute for petro-diesel and also most advantageous over petro-diesel for its environmental friendliness (Andrijanto (2012) ). With the aim of developing a process of biodiesel production that is environmentally benign much interest has been focused on the use of solid base catalysts, such as calcium oxide, for the transesterification of vegetable oils with methanol (Arzamendi et al. (2007) ). It has very similar physical properties to diesel fuel and even higher cetane number which allows it to be used directly as substitute fuel for diesel engines without any modifications or as a blending agent for diesel fuel. Since it is produced from renewable and domestically grown feed-stocks, it can reduce the demand for petroleum based fuels and possibly lower the overall cost of diesel fuel (Nahadi and IM (2018) ).

The chemical process used to obtain biodiesel is based on the solid in the system catalyzing the reaction to reduce the time and the cost of the process through the reuse of the catalyst, decreasing the level of impurities in the reaction products (Nahadi and IM (2018) ). Biodiesel due to its agricultural source essentially contains no or very little sulfur and thus offers promise to reduce particulate and toxic emissions, which is one of the primary objections to diesel engine (Musa (2016) ). One of such oils is castor oil that is obtained from castor plant *Ricinus Communis* which belongs to the family of Euphorbiaceae. The beans contain 35 – 55 % oil by weight for high yield breed type; it has higher viscosities among vegetable oils and a molecular weight of 298. It is hard non-drying oil that neither becomes stiff with cold nor unduly thin with heat hence it is used as a lubricant for jet and racing cars engines. It is the only source of an 18-carbon hydroxylated fatty acid with one double bond in each of the fatty acid chain and ricinoleic acid make up over 87% of the fatty acid composition (Atapour and Kariminia (2011) ). The presence of a hydroxyl group at C12 of the ricinoleic acid, the ester linkages, double bonds and hydroxyl groups provide reaction sites that makes it unusually polar which provides sites for the production of a wide range of natural and synthetic resins, waxes, polymers and elastomers. It also has excellent emollient and lubricating properties, a marked ability to wet and disperse dyes, pigments and fillers and as well as several medicinal values (Balat and Balat (2008) ). Castor oil has excellent solubility in alcohols, for this reason it is theoretically ideal oil for transesterification to biodiesel, requiring a minimum amount of catalyst and heating which can reduce costs of production (Chew and Bhatia (2008) ). The homogeneous catalyzed transesterification of vegetable oil for biodiesel production have some drawbacks such as saponification of the oil, unable to recover catalyst, and limitation in establishing a continuous process and therefore reducing biodiesel yield (Chukwueyem et al. (2015) ). In contrast, the solid catalysts could easily be separated from the reaction mixture by filtration and reused. Among the heterogeneous catalysts, calcium oxide has derived researcher's attention because it is a cheap and abundantly available in the nature. Calcium oxide can be reused up to 3 times in transesterification reaction which made it an economic catalyst (Nahadi and IM (2018) ). As the calcium oxide is derived from the natural source, it is envi-

ronmentally friendly and causes no harm to the ecosystem ([Chew and Bhatia \(2008\)](#)).

Environmental concerns have also led to the search for solid catalysts which are eco-friendly and effective ([Atapour and Kariminia \(2011\)](#)). The transesterification reaction proceeds with catalyst or without any catalyst by using primary or secondary monohydric aliphatic alcohols having 1–8 carbon atoms. Among the alcohols that can be used in the transesterification reaction are methanol, ethanol, propanol, butanol and amyl alcohol. Methanol and ethanol are used most frequently. A successful transesterification reaction produces two liquid phases: ester and crude glycerol ([Chung et al. \(2008\)](#)).

Application of heterogeneous catalysts in the field of renewable energy to produce renewable fuel has become a significant area of research due to environmental concerns resulting in continual dependence on finite fossil fuel sources, current industrial energy demands and upswing in the world population. To this end, current research is focused on fuel derivable from renewable sources using heterogeneous catalyst and its utilization in the transportation industries and other industrial processes.

Catalytic reactions are known to go faster to completion whether homogeneous or heterogeneous. However, usage of homogeneous catalyst is characterized by cumbersome process and high cost of product vis-a-vis environmental pollution ([Demirbas \(2008\)](#)). Therefore, in this research work castor oil was chosen for the production because it has excellent solubility in alcohols.

## **2. MATERIALS AND METHODS**

### **2.1 MATERIALS**

Crude Castor oil was extracted from castor seed, methanol, 99.8% purity (analytical grade), and Calcium oxide (CaO) were purchased.

### **2.2 EXPERIMENTAL DESIGN**

The experimental design was carried out using Response Surface Methodology (RSM) design expert 12.0 version software as presented in [Table 1](#)

### **2.3 TRANSESTERIFICATION PROCESS FOR THE PRODUCTION OF BIODIESEL**

The transesterification process was carried out using a batch reactor. A stirrer was used to stir the mixture in the screw-capped vessel. The vessel was kept in a water bath to maintain the temperature. The mixture was stirred at a speed of 300 rpm for all test runs. After the transesterification reaction, the product mixture was poured into a beaker and then allowed to settle into two phases. Since methanol has boiling point of 64.7°C higher reaction temperature of 62°C was used ([Farahmandjou and](#)

Golabiyan (2016) ). As per stoichiometry reaction carried out at 6:1 to 16:1 methanol to oil molar ratio and a catalyst concentration of 1w/w% to 6w/w%. Further by keeping required time constant, the mixture of methanol, and calcium oxide solution was added into the batch reactor. Further the reaction time was 60 min for all experimental runs.

**Table 1** Process Parameters for the Production of Biodiesel

Run	Factor 1: A: Cat. Con. w/w%	Factor 2 B: Molar ratio	Factor3C: Temperature °C
1	6	16	50
2	3.5	6	56
3	6	6	62
4	3.5	11	56
5	3.5	11	50
6	3.5	16	56
7	6	16	62
8	3.5	11	56
9	3.5	11	56
10	1	11	56
11	1	6	50
12	1	16	50
13	3.5	11	56
14	3.5	11	56
15	3.5	11	62
16	6	11	56
17	1	6	62
18	6	6	50
19	1	16	62
20	3.5	11	56

## 2.4 PURIFICATION OF CRUDE BIODIESEL PRODUCED

After the transesterification is completed, in the end, the excess methanol was distilled off under vacuum. The product was centrifuged, and three phases were formed, the upper layer was biodiesel, the middle layer was glycerol, and the lower layer was a mixture of solid CaO and a small amount of glycerol. The biodiesel was collected for chromatographic analysis (Freedman et al. (1984) ). The phase separation between biodiesel and catalyst was characteristically clean and absolutely complete (Fukuda et al. (2001) ). The upper layer, containing the refined biodiesel product, was then stored and prepared for analysis.

## 2.5 ANALYSIS OF THE BIODIESEL PHASE

The ethyl ester compounds in biodiesel samples was analyzed by a HP 6890 Gas Chromatogram (GC) equipped with a Flame Ionization Detector (FID) and capillary col-

umn DB23 (60-m $\times$ 0.25-m $\times$ 0.15- $\mu$ m) according to a methodology proposed by Agilent. Normal hexane solutions of the biodiesel samples with a concentration of 100 mg/ml was injected by an auto injector at an oven temperature of 50 °C, which was heated up to 230 °C. The injector temperature and the detector temperature were 250 °C and 280 °C, respectively; helium was used as the carrier gas (Demirbas (2008)).

## 2.6 GAS CHROMATOGRAPHY- MASS SPECTROSCOPY (GC-MS)

The biodiesel product was analyzed by Fourier Transform Infra-Red spectrometer (Nicolet 5700) and Gas Chromatography-Mass Spectrometry. The fatty acid methyl esters (FAMES) in the produced biodiesel were then characterized and identified using gas chromatography-mass spectrometry fitted with 5MS column (30 m, 0.25mmID, 0.25 $\mu$ m). GC-MS analysis mainly identifies the quality and quantity of the produced biodiesel resembled in the methyl esters present in the crude castor oil sample. This technique also gives the distribution area for each component in the produced sample. The total yield of the biodiesel was finally calculated.

## 2.7 FOURIER TRANSFORM INFRARED SPECTROSCOPY (FT-IR)

Fourier transform infrared spectroscopy was used to determine the main functional group presence in the produced biodiesel and its castor oil. The IR spectrum was scanned through a wavelength range of 4000–400  $\text{cm}^{-1}$ . The FT-IR technique was used for quantitative and qualitative analysis of the produced biodiesel and to confirm GC-MS results.

# 3. RESULTS AND DISCUSSIONS

## 3.1 BIODIESEL YIELDS

The yields of biodiesel obtained using twenty experimental runs were shown in Table 2. It is worthy to mention that highest biodiesel of 54% was obtained in the run 4 and 13 during the transesterification reaction, and the lowest yield of 23% was recorded in the run 3, as shown in Table 2. The process parameters considered in this study include amongst others, the molar ratio of oil to methanol, catalyst concentration, and reaction temperature. From Table 2, the molar ratio of methanol to oil has a significant impact on the biodiesel yield. Thus, 54% yield was reported at 11:1 molar ratio of oil to methanol. The excess methanol was necessary because it increased the rate of methanolysis. The higher amount of methanol promoted the formation of methoxy species on the CaO surface, leading to a shift in the equilibrium towards forward direction, thus increasing the rate of biodiesel conversion (Gomes et al. (2010)). However, the yield was slightly reduced when the ratio of oil to methanol was higher than 11:1, this is due to excessive methanol above the optimal point.

Catalyst concentration played an important role during the transesterification reaction. From Table 2, it was seen that biodiesel yield increase with increased catalyst concentration ranging from 1%w/w to 6%w/w. Thus catalyst concentration of 3.5%w/w of CaO provided highest biodiesel yield of 54%. Furthermore, Table 2, showed the significance of reaction temperature during castor oil transesterification to biodiesel fuel. The biodiesel yields obtained were determined at different reaction temperature ranging from 50 to 62 °C. The biodiesel yield increased with the increased in reaction temperature until an optimal point of 56 °C, with biodiesel yield of 54%. Above this optimum temperature, the yield was decreased. High temperature are not preferred; as the temperature increases and approaches boiling point of methanol, the methanol quickly vaporizes and form a large number of bubbles and thus decreased the yield of biodiesel (Gui et al. (2008) ).

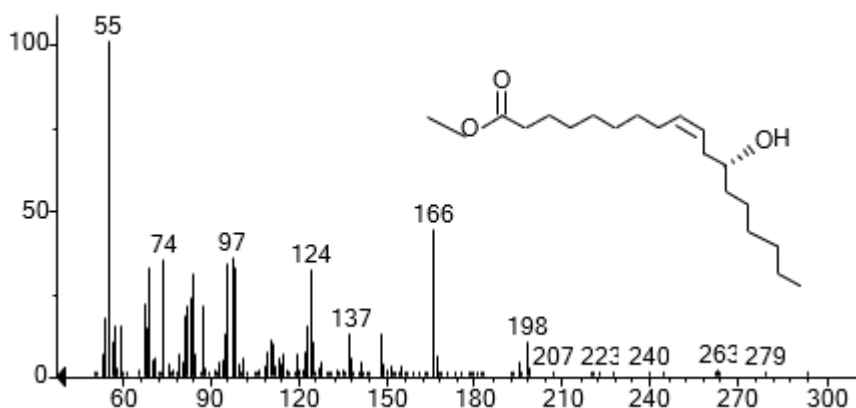
**Table 2** Biodiesel Yields

Std	Run	Factor 1 A: Cat. Con w/w%	Factor 2 B: Molar ratio	Factor 3 C: Temp °C	Biodiesel Yield %
4	1	6	16	50	40.50
11	2	3.5	6	56	42.80
6	3	6	6	62	23.00
19	4	3.5	11	56	54.00
13	5	3.5	11	50	52.60
12	6	3.5	16	56	53.00
8	7	6	16	62	42.00
16	8	3.5	11	56	52.40
20	9	3.5	11	56	53.30
9	10	1	11	56	33.00
1	11	1	6	50	31.50
3	12	1	16	50	27.60
17	13	3.5	11	56	54.00
18	14	3.5	11	56	52.70
14	15	3.5	11	62	52.80
10	16	6	11	56	37.00
5	17	1	6	62	31.50
2	18	6	6	50	23.10
7	19	1	16	62	29.00
15	20	3.5	11	56	53.20

### 3.2 CHARACTERISTIC BAND POSITIONS FOR BIODIESEL

From Table 3 it is worth noting that the characteristics peaks found in the region  $1169.82\text{ cm}^{-1}$  indicated stretching vibrations of C-O and C-O-C. They can also indicate the bending vibration of O-CH<sub>3</sub> in the spectrum (12). The band region of  $1461.82\text{ cm}^{-1}$  can be ascribed to the bending vibration of C-H methyl groups in the fuel. The characteristics peak at wave number  $1739.96\text{ cm}^{-1}$  which is the strongest in the spectrum is attributed to C=O groups with the stretching mode of vibration.

These groups indicated the presence of carbonyl functional groups in the biodiesel. The groups indicated the conversion of triglycerides in the oil to methyl esters (biodiesel) (13). The peaks at  $2862.15\text{cm}^{-1}$  and  $2932.39\text{cm}^{-1}$  indicated symmetric and asymmetric stretching vibrations of C-H alkane groups respectively. They could be methyl ( $\text{CH}_3$ ) or methylene groups in the esters chains of the biodiesel and they require high energy to cause stretching vibrations within their bond when compared to the ordinary C-H bending vibrations of alkene groups detected at low energy and frequency region (14). The peak at  $3014.44\text{cm}^{-1}$  is attributed to the stretching vibration of =C-H alkene groups. They are detected above wave number  $3000\text{cm}^{-1}$  in the spectrum compared to corresponding alkane C-H stretching groups detected below  $3000\text{cm}^{-1}$ . However, biodiesel showed oxygen functional group of ester C-O bond at  $1104.67\text{cm}^{-1}$  to  $1175.96\text{cm}^{-1}$  and ester C=O bond at  $1739.96\text{cm}^{-1}$ . The presence of oxygen in biodiesel promotes cleaner and complete combustion. On the other hand, the conventional diesel with less oxygen component produced more black smoke and incomplete combustion during burning (12). From the result of GC-MS, it was possible for one to determine the methyl ester groups present in the biodiesel produced. 9-Octadecenoic acid-12-hydroxy- methyl ester was identified from Table 2 . It was found to be the most abundant compound.



**Figure 1** Structure of the 9-Octadecenoic acid, 12-hydroxy-, methyl ester from GC-MS. Other names: Methyl ricinoleate; Ricinoleic acid methyl ester; Flexricin P-1; Methyl ricinolate; 12-Hydroxy-9-octadecenoic acid methyl ester.

### 3.3 OPTIMIZATION OF BIODIESEL PRODUCTION USING RESPONSE SURFACE METHODOLOGY

The selected independent reaction parameters (reaction temperature, methanol to oil molar ratio and catalyst concentration) were optimized using design of experiment software (version 12.0). Response surface method (RSM) provides elaborate analysis of reaction parameters on the production process. In order to optimize the process parameters, a twenty designed experiments were conducted and analyzed



**Table 3** FT-IR Characteristic Band Positions for Castor oil and Castor oil Biodiesel

Castor oil		Biodiesel	
Characterization absorption vibration	Functional group	Characterization absorption vibration	Functional group
2930.28	C-H Aliphatic stretching	3014.44	C-H Stretching of vinyl
2861.40	C-H Aliphatic stretching	2932.39	C-H Aliphatic Stretching
1745.39	C=O stretching	2862.15	C-H Aldehyde stretching
1451.63	C-H Bending	1739.96	C=O
1363.03	O-H Bending	1461.82	O-H Bending
1169.82	C-O Stretching	1175.96	C-O Stretching
		1104.67	C-O Stretching

with multiple regressions using Design-Expert 12.0 software. Regression analysis yield three linear coefficients (A, B, C), three quadratic coefficients ( $A^2$ ,  $B^2$ ,  $C^2$ ) and three cross product coefficients (AB, AC, BC) for the full model (Table 4). Furthermore, Table 4.3 describes the ANOVA for the response surface quadratic model. The Statistical analysis on factors affecting biodiesel yield, the Design Expert 12.0 program was used in the regression analysis and analysis of variance (ANOVA).

### 3.3.1 ACTUAL AND PREDICTED VALUES

Response Surface Methodology was utilized to evaluate the relations between the response (% biodiesel yields) and the three reaction variables. Twenty experiments were performed in a randomized order. The results for each point base on the experimental design are shown in Table 4.3. The percentage of biodiesel yield obtained at the design points of different reaction conditions as shown in Table 4, where the actual or experimental as well as predicted values of percentage yield of biodiesel at the design points are shown in Table 4. The actual yields of biodiesel produced via different process parameters were calculated and obtained, thus ranged from 23% to 54%. The yield of the transesterification processes were calculated as the sum of weight of biodiesel produced to weight of oil used multiplied by 100. It was also observed from Table 4 that the actual values of the biodiesel yield are in agreement with the predicted values as their differences are minimal.

### 3.3.2 THE QUADRATIC MODEL

The multiple regression coefficients were obtained by employing a least square technique to predict quadratic polynomial model for the biodiesel content. Hence, the best fitting model was determined. The model was selected based on the highest order polynomial where the additional terms were significant and the model was not aliased as suggested by the software (see Table 5 and Table 6). The coefficients of the



**Table 4** Relationship between Actual and Predicted Biodiesel Yield

Run Order	Actual Value	Predicted Value	Residual	Leverage	Internally Studentized Residuals	External Studentized Residuals	Cook's Distance	Influence on Fitted Value DFFITS
1	40.50	40.91	-0.4132	0.793	-1.011	-1.012	0.392	-1.983
2	42.80	44.31	-1.51	0.491	-2.351	-3.335	0.533	-3.275 <sup>1</sup>
3	23.00	22.77	0.2268	0.793	0.555	0.535	0.118	1.048
4	54.00	52.98	1.02	0.118	1.207	1.239	0.020	0.453
5	52.60	52.83	-0.2273	0.491	-0.355	-0.338	0.012	-0.332
6	53.00	52.35	0.6527	0.491	1.018	1.020	0.100	1.002
7	42.00	42.26	-0.2632	0.793	-0.644	-0.624	0.159	-1.222
8	52.40	52.98	-0.5818	0.118	-0.690	-0.670	0.006	-0.245
9	53.30	52.98	0.3182	0.118	0.377	0.360	0.002	0.132
10	33.00	34.13	-1.13	0.491	-1.758	-2.007	0.298	-1.971
11	31.50	31.02	0.4768	0.793	1.167	1.191	0.522	2.333 <sup>1</sup>
12	27.60	27.61	-0.0132	0.793	-0.032	-0.031	0.000	-0.060
13	54.00	52.98	1.02	0.118	1.207	1.239	0.020	0.453
14	52.70	52.98	-0.2818	0.118	-0.334	-0.319	0.001	-0.117
15	52.80	53.43	-0.6273	0.491	-0.978	-0.976	0.092	-0.959
16	37.00	36.73	0.2727	0.491	0.425	0.407	0.017	0.400
17	31.50	30.87	0.6268	0.793	1.534	1.664	0.903	3.259 <sup>1</sup>
18	23.10	22.92	0.1768	0.793	0.433	0.414	0.072	0.812
19	29.00	28.96	0.0368	0.793	0.090	0.086	0.003	0.167
20	53.20	52.98	0.2182	0.118	0.259	0.246	0.001	0.090

response surface model as provided by the above quadratic model equation was also evaluated. From the ANOVA of response surface quadratic model for biodiesel conversion, the Model F-value of 348.17 and Prob> F of <0.0001 implied that the model was significant.

For the model terms, values of P-values less than 0.0500 indicated that model terms were significant. In this case A, B, AB, A<sup>2</sup>, B<sup>2</sup> were significant model terms. Values greater than 0.1000 indicated the model terms were not significant. If there are many insignificant model terms (all have Prob> F less than 0.050). This tells us that the catalyst concentration, methanol to oil ratio, and their quadratic terms affect the yield much significantly. However, the interaction terms were found to be insignificant except AB since the values were greater than 0.0500 indicating that the model terms were insignificant (Table 5). As we observed from p-values of the model coefficients in Table 4.8, the value of the catalyst concentration and methanol to oil molar ratio in both linear and quadratic model are much less than 0.0001. This indicated that they are the most significant in determining the model than the rest. However, in order to minimize error, the coefficients were considered in the design. The lack of fit from the ANOVA analysis indicated that the model does indeed represent the actual

relationships of reaction parameters, which were well within the selected ranges. The Lack of Fit F-value of 2.75 implied that the Lack of Fit is not significant relative to the pure error. There is a 14.57% chance that a Lack of Fit F-value this large value could occur due to noise. Non-significant lack of fit is good because we want the model to fit.

**Table 5** Analysis of Variance (ANOVA) for Response Surface Quadratic Model of Transesterification Process

Source	Sum of Squares	df	Mean Square	F-value	p-value	
<b>Model</b>	2529.62	9	281.07	348.17	< 0.0001	significant
A-Cat.con	16.90	1	16.90	20.93	0.0010	significant
B-Molar ratio	161.60	1	161.60	200.18	< 0.0001	significant
C-Temp	0.9000	1	0.9000	1.11	0.3159	not significant
AB	228.98	1	228.98	283.64	< 0.0001	significant
AC	0.0000	1	0.0000	0.0000	1.0000	not significant
BC	1.13	1	1.13	1.39	0.2651	not significant
A <sup>2</sup>	847.45	1	847.45	1049.75	< 0.0001	significant
B <sup>2</sup>	59.58	1	59.58	73.80	< 0.0001	significant
C <sup>2</sup>	0.0582	1	0.0582	0.0721	0.7938	not significant
Residual	8.07	10	0.8073			
Lack of Fit	5.92	5	1.18	2.75	0.1457	not significant
Pure Error	2.15	5	0.4307			
<b>Cor Total</b>	2537.69	19				

### 3.3.3 FINAL EQUATION IN TERMS OF CODED FACTORS

$$\text{Yield} = +52.98 + 1.30 \cdot A + 4.02 \cdot B + 0.3000 \cdot C + 5.35 \cdot A \cdot B + 0.0000 \cdot A \cdot C + 0.3750 \cdot B \cdot C - 17.55 \cdot A^2 - 4.65 \cdot B^2 + 0.1455 \cdot C^2 \quad \text{Equation (3.1)}$$

Where, A = Reaction temperature, B= Molar ratio of methanol to oil, C= Catalyst concentration

### 3.3.4 COEFFICIENTS IN TERMS OF CODED FACTORS

The coefficient estimate represents the expected change in response per unit change in factor value when all remaining factors are held constant. The intercept in an orthogonal design is the overall average response of all the runs. The coefficients are adjustments around that average based on the factor settings. When the factors are orthogonal the Variance Inflation Factor (VIFs) are 1; Variance Inflation Factor (VIFs) greater than 1 indicate multi-collinearity, the higher the Variance Inflation Factor, the more severe the correlation of factors. As a rough rule, Variance Inflation Factors less than 10 are tolerable.

### 3.3.5 FINAL EQUATION IN TERMS OF ACTUAL FACTORS

$$\text{Yield} = 19.43162 + 15.47309 \cdot C + 2.70200 \cdot M - 0.540025 \cdot T + 0.428000 \cdot C \cdot M - 1.35388 \cdot 10^{-15} \cdot C \cdot T - 2.80873 \cdot C^2 - 0.186182 \cdot M^2 + 0.004040 \cdot T^2 \quad \text{Equation (3.2)}$$

Where, T = Reaction temperature, M= Molar ratio of methanol to oil, C= Catalyst concentration

### 3.3.6 MODEL ADEQUACY OF QUADRATIC MODEL

The quality of the model developed was evaluated based on the correlation coefficient value, R square ( $R^2$ ). The  $R^2$  value for Equation above was 0.9996. This indicated that 99.96 % of the total variation in the biodiesel yield was attributed to the experimental variables studied. The closer the  $R^2$  value to unity, the better the model will be (5), as it will give predicted values, which are closer to the actual values for the response.

**Table 6** Regression Coefficients and Significance of Response Surface Quadratic Model

Factor	Coefficient Estimate	df	Standard Error	95% CI Low	95% CI High	VIF
Intercept	52.98	1	0.3089	52.29	53.67	
A-Cat.con	1.30	1	0.2841	0.6669	1.93	1.0000
B-Molar ratio	4.02	1	0.2841	3.39	4.65	1.0000
C-Temp	0.3000	1	0.2841	-0.3331	0.9331	1.0000
AB	5.35	1	0.3177	4.64	6.06	1.0000
AC	0.0000	1	0.3177	-0.7078	0.7078	1.0000
BC	0.3750	1	0.3177	-0.3328	1.08	1.0000
A <sup>2</sup>	-17.55	1	0.5418	-18.76	-16.35	1.82
B <sup>2</sup>	-4.65	1	0.5418	-5.86	-3.45	1.82
C <sup>2</sup>	0.1455	1	0.5418	-1.06	1.35	1.82

**Table 7** Model Adequacy of Quadratic Model

Std. Dev.	Mean	C.V. %	R <sup>2</sup>	Adjusted R <sup>2</sup>	Predicted R <sup>2</sup>	Adeq Precision
0.8985	41.95	2.14	0.9968	0.9940	0.9831	48.2493

The Predicted  $R^2$  of 0.9831 is in reasonable agreement with the Adjusted  $R^2$  of 0.9940 (Table 4.6); i.e. the difference is less than 0.2. Adeq Precision measures the signal to noise ratio. A ratio greater than 4 is desirable. The ratio of 48.249 indicated an adequate signal. This model can be used to navigate the design space. The value of the adjusted coefficient of determination (Adj  $R^2$  = 0.9940) is also high, thus indicating the significance of the model as well as the value of coefficient of variation (CV) is low (2.14%), thereby indicating the reliability of the results of the fitted model.

From the ANOVA and regression analysis on Table 4.4 and Table 4.5 respectively, it can be seen that the linear terms (A, B), the quadratic term (A<sup>2</sup>, B<sup>2</sup>) and the cross

product AB, were significant (because Prob> F less than 0.05), but the interactions (cross products) AC, BC, and C, C<sup>2</sup> were insignificant.

### **3.3.7 EFFECT OF INTERACTIVE OPERATING CONDITIONS ON BIODIESEL YIELD**

From Equation (3.2), it was clearly shown that, all the linear terms had positive coefficients except C which is reaction temperature, whereas the quadratic terms and the interaction terms had negative coefficients except AB and C<sup>2</sup>. Therefore, an increase in CaO catalyst concentration, methanol to oil molar ratio and reaction temperature, to a certain extent could result in a higher percentage of biodiesel. However, a reduction in the percentage of biodiesel could be noticed when using too high CaO catalyst concentration, and methanol to oil molar ratio. Similar pattern was followed when increasing methanol to oil molar ratio. This is due the positive coefficient for the linear parameters (A B, and C) played the main role when the CaO catalyst concentration and methanol/oil molar ratio were at lower level. While at higher level, the interaction as well as the quadratic terms shows negative significant effect that leads to decrease the yield since the methanol and triglyceride in the oil are immiscible. Addition of catalyst can facilitate the transesterification reaction and rapidly increase the yield (12). However, when the catalyst concentration was too high, the yield of the biodiesel was reduced. Similarly, the increase of the methanol amount, on one side, it will drive the reaction to the right since the reaction is an equilibrium process; on the other hand, excess methanol will help to increase the solubility of glycerol, which favours the backward reaction to the left. Therefore, the yield of biodiesel is decreasing.

## **4. CONCLUSIONS**

In this research work, the experiments were conducted using surface response methodology couple with central composite design to examine the optimal reaction condition for the production of biodiesel using crude castor oil. The biodiesel produced contained an ester functional group and showed that castor oil methyl ester can be used as a possible alternative fuel for diesel engines. Based on the forgoing discussion, the conclusions of this study are summarized as follows: Castor oil can be used as a biodiesel raw material with its high oil content and its non-edible characteristics. Of all the variables studied, the interaction between methanol to oil ratio and amount of catalyst had more influence on the yield of fatty acid methyl ester. The ester yield obtained from the transesterification process of crude castor oil ranged from 23 to 54%. The optimum biodiesel yield of 54% was obtained at a catalyst concentration of 3.5wt%, methanol to oil molar ratio of 11:1 and Reaction temperature of 62°C at a reaction time of 1hr and 300rpm. From the result of the FTIR and GC-MS the methyl ester groups present in the biodiesel produced was determined. 9-Octadecenoic acid-12-hydroxy- methyl ester was identified as the

most abundant compound in the biodiesel which has all the functional group reveal by the FTIR.

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