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Optimization of Transesterification of Castor Oil through Response Surface Methodology

By

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ABSTRACT

This study was aimed at optimizing the yield of transesterification of castor oil methyl ester (COME) from castor oil using response surface methodology (RSM). RSM was used to analyse and optimize the operating variables for the transesterification process. Box – Behnken design (BBD) was employed to study the effects of reaction temperature (°C), time (min), catalyst concentration (wt.%) and methanol to oil ratio (v/v) on the yield of COME. A total of 29 experimental runs which were generated by BBD were carried out. The results of RSM analysis indicated that reaction temperature, catalyst concentration and methanol to oil volume ratio were significant variables on the yield of COME. The coefficient of determination obtained ($R^2 = 0.9713$) showed a fitness of a second order model. The interactive effect of catalyst concentration and methanol to oil volume ratio exhibited a positive effect on the COME yield. The optimum conditions for the transesterification reaction based on the second - order quadratic model was found to be; temperature 46.88 °C, reaction time 107.41 min, catalyst concentration 1.03 wt.% and methanol to oil ratio 0.44 v/v. The second order quadratic model developed provides a statistical approach to predicting the optimum yield of COME.

Keywords: COME, Response surface methodology, Box – Behnken design, Optimization, transesterification

1. INTRODUCTION

Most of the energy consumed worldwide is derived from fossil sources such as petroleum, coal and natural gas. Today, 86 % of the world's energy consumption and almost 100 % of the energy needed in the transportation sector is derived from fossil fuels [1]. The continuous use of these fossil fuels has also resulted in global warming, and in increase in green house gases (GHG). The primary exhaust emissions that result when fossil fuels are burnt include oxides of nitrogen, carbon monoxide, hydrocarbons, carbon dioxide and particulate matters. These primary pollutants react in the atmosphere to generate secondary pollutants that cause acid rain, photochemical smog and also results in depletion of the ozone layer. Many of these pollutants have serious implications on human health and the environment. Thus, there is the need to look for alternative sources of energy that will help reduce the harmful effect of these emissions.

Biofuels are used as alternative sources of energy generation. They are made from living organisms or the waste that these organisms produce. They can be derived directly from plants, or indirectly from agricultural, commercial, domestic and/or industrial wastes. Biodiesel is a form of biofuel that can be used as an alternative fuel to petroleum-based diesel fuel. Biodiesel is also known as fatty acid methyl ester (FAME). It comprises of monoalkyl esters of long chain fatty acids derived from

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[1] Dorian, P., Herman, T., Dale, R., Simbeck (2006). Global challenges in energy. International Energy Associates Inc., Chevy Chase, USA. 1-5.

renewable lipid feedstock, such as vegetable oils or animal fats. As an alternative fuel, it can be used in neat form or blended with petroleum-based diesel. Biodiesel, as an alternative fuel, has many advantages over the petroleum-based diesel. Apart from the fact that it is derived from a renewable resource, thereby relieving reliance on petroleum-based fuel, it is also biodegradable and non-toxic. Compared to petroleum-based diesel, biodiesel has a more favorable combustion emission profile, such as low emissions of carbon monoxide, particulate matter and unburned hydrocarbons[2]. Carbon dioxide produced by combustion of biodiesel can be recycled by photosynthesis, thereby minimizing the impact of biodiesel combustion on the greenhouse gas effect[3,4]. Biodiesel has a relatively high flash point, which makes it less volatile and safer to transport or handle compared to petroleum diesel.[5] It provides lubricating properties that can reduce engine wear and extend engine life.[6] It also gives a better engine performance due to its higher cetane number. Thus, these advantages of biodiesel make it a good alternative to petroleum-based diesel fuel and have led to its use in many countries.

Biodiesel can be produced economically across a variety of places and scales; from urban to rural, small to commercial, because it can be refined under normal atmospheric temperature and pressure. More than 95 % of biodiesel is prepared from conventionally grown edible oils, such as soyabean oil, sunflower oil, rapeseed oil, coconut oil, palm oil, e.t.c. This causes competition between the utilization of these oils for energy and food consumption. The land use for production of edible oil as feedstock for biodiesel production competes with the use of land for food production. Also, the high cost of edible plant and vegetable oils as raw materials used for biodiesel production is the major bottleneck which greatly prohibits its widespread application.

In other to tackle these challenges, low quality feedstock such as waste or used vegetable oil, non edible oils have been used as alternative feedstock for biodiesel production.[7, 8, 9, 10] Castor oil is a typical example of non-edible oil used for the production of biodiesel. This vegetable oil has peculiar characteristics such as its fatty acid content with over 85 % of ricinoliec acid.[11, 12-13] This fatty acid

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[11] Madankar, C. S., Pradhan, S. and Naik, S. N. (2013). Parametric study of reactive extraction of castor seed (Ricinus communis L.) for methyl ester production and its potential use as bio lubricant. Industrial Crops and Products. 43, 283 – 290.

[12] Canoira, L., Galean, J. G., Alcantara, R., Lapuerta, M., Garcia – Contreras, R. (2010). Fatty acid methyl esters (FAMEs) from castor oil: Production process assessment and synergistic effect in its properties. *Renewable Energy.* 35, 208 – 217.

[13] Dias, J. M., Araujo, J. M., Costa, J. F., Alvin – Ferraz, M. C. M. and Almeida, M. F. (2013). Biodiesel production from raw castor oil. *Energy.* 53, 58 – 66.

has 18 carbon atoms with one hydroxyl group on carbon 12. It therefore contains more oxygen than other oils and, for that, and it is more soluble in alcohol. The viscosity of castor oil is more than 100 times higher than petroleum based diesel. Other vegetable oils possess viscosities that are 10 to 20 times higher than petroleum based diesel.[14] This high viscosity of castor oil is a major problem during the transesterification reaction. Transesterification can occur at different temperatures depending on the vegetable oil used, taking care not to exceed the boiling point of the alcohol used. Compared to other oils, transesterification reaction of castor oil takes place at significantly low temperatures.[15] The transesterification of castor oil at temperatures above 50 ^oC has been found to produce castor oil methyl ester (COME) that solidifies at room temperature and reduces the yield.[12] One of the most important variables affecting the yield of FAME is the molar ratio of alcohol to oil. A higher molar ratio results in higher yield in shorter time. But also worthy of note is the fact that alcohol has chemical affinity for both glycerine and ester hence the higher the molar ratio, the more difficult it is to separate both phases. The catalyst concentration is also very important, in that it increases the rate of the conversion process, but in excess could yield more of glycerol than the ester. The rate of conversion of vegetable oils to FAME increases with increase in reaction time up until a point is reached after which no more conversion occurs. At this time maximum conversion had been achieved. Taking all these variables into consideration, a means of relating these variables so as to optimize the yield has to be developed. In order to optimize these variables, what is normally done in conventional experiments will be to vary one of the factors while keeping the others constant.[11] This method is time consuming and will require carrying out large number of experiments. To overcome this drawback, statistical design of experiments can be used. Response surface methodology (RSM) is an effective statistical tool for designing such experiments. Models can be built by RSM and it can also be used to investigate the relationship between two variables or factors, while keeping the other variables constant at their central point.

This present study was aimed at developing a mathematical model that describes the relationship between the process variables (alcohol to oil ratio, catalyst concentration, reaction time and temperature) and the COME yield during transesterification of castor oil. The Box – Behnken method was used to evaluate the interactive effect between these variables and also to optimize the process conditions of transesterification of castor oil to produce COME.

2. MATERIALS AND METHODS

2.1 Materials

Castor seed oil was purchased from Mosdelic Scientific global services, Benin City, Nigeria. The physicochemical properties of the castor oil are shown in Table 1. Methanol (99.5 %), ethanol (99.8 %), potassium hydroxide, sodium hydroxide, benzene, chloroform, acetic acid, potassium iodide, sodium thiosulphate, Wijs solution, starch solution and phenolphthalein indicator were also purchased from Mosdelic Scientific global services and were all of analytical grade.

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[14] Demirbas, A. (2003). Biodiesel fuels from vegetable oils via catalytic and non-catalytic supercritical alcohol transesterification and other method. Energy Conversion Manager. 44, 2093-2109.

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Table 1. Physicochemical Properties of Castor Seed Oil

Properties	Value
Density at 27 °C (kg/m^3)	954.8
Viscosity at 27 °C (mm^2/s)	128.20
Acid value (mg KOH/g oil)	1.24
Iodine Value (g I_2/100 g oil)	88.46
FFA (%)	0.52
Peroxide Value (meq. O_2/kg)	1.52
Saponification value (mg KOH/g oil)	179.52

2.2 **Experimental Design of COME Production**

The Box – Behnken design (BBD) was employed to optimize the transesterification of castor seed oil. Three – level – four factors design was applied and it generated 29 experimental runs. The factors selected for the transesterification of the castor seed oil were temperature (X_1), reaction time (X_2), catalyst concentration (X_3) and methanol to oil ratio (X_4). The coded and actual levels of the factors are shown in Table 2. The experiments were carried out in a random order in order to minimize the effects of unexplained variability in the observed response which are usually due to extraneous factors.

Table 2. Factors and their levels for Box – Behnken Design

Variable	Symbol	Coded Levels		
		-1	0	+1
Reaction Temperature (°C)	(X_1)	25	45	60
Reaction Time (min)	(X_2)	45	82.5	120
Catalyst Concentration (wt. %)	(X_3)	0.5	1	1.5
Methanol/Oil Ratio (v/v)	(X_4)	0.14	0.36	0.58

The independent variables used were coded according to Eq. 1

$$X_i = \frac{(x_i - x_o)}{\Delta x} \tag{1}$$

X_i and x_i are the coded value and actual value respectively. x_o is the value of X_i at the centre point and Δx is the step change. To correlate the response to the independent variables, multiple regressions were used to fit the coefficient of the polynomial model of the response.

The COME production data were analysed statistically using RSM to fit the quadratic model that was generated by Design Expert 7.0.0 software (Stat – Ease Inc., Minneapolis). The fitted quadratic response model is shown in Eq. 2

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

where Y is the predicted response, b_0 is the intercept value, b_i (i = 1, 2,......, k) is the first order model coefficient, b_{ij} represents the quadratic coefficient of X_i, and e is the random error.

The quality of the fit of the model was evaluated using analysis of variance (ANOVA). The mathematical model was considered satisfactory when the ANOVA data showed high level of statistical significance. Response surface plots were developed using the fitted quadratic polynomial model obtained from the analysis, holding two of variables constant and varying the other two variables.

2.3 Transesterification Procedure

The free fatty acid content of the castor oil was less than 1 % and as a result of this, the base catalysed transesterification reaction was employed for the production of COME. A known weight of NaOH pellet was dissolved in a known volume of methanol and allowed to dissolve. The resulting mixture is transferred to the oil in a round bottom flask placed on a constant temperature equipment with magnetic stirrer (B. Bran Scientific and Instrument Company, England. Model No. HJ – 3D). At the completion of the reaction, the product is transferred to a separating funnel to allow the biodiesel and glycerol to separate. After separation, the glycerol was drained off, while the COME that was left in the funnel was washed with warm water to remove traces of glycerol, catalyst or methanol that may still be present. The washing process of the COME was allowed to continue until the washed water had a pH of about 7. The washed COME was then heated in a round bottom flask to dry the biodiesel. The COME yield was determined by applying Eq. 3

$$\text{Yield (\%)} = \frac{\text{weight of COME produced}}{\text{weight of castor oil used in transesterification}} \times 100 \qquad (3)$$

2.4 Physicochemical Analysis

Physicochemical analysis of the COME produced was carried out according to the American (ASTM D 6751) standards. The analyses that were carried out include moisture content, viscosity, specific gravity, flash point, acid value. The iodine value and peroxide values were carried out according to the AOCS method.[16] The analysis was carried out in triplicate and the mean value was reported.

3. RESULTS AND DISCUSSION

3.1 Statistical Analysis

The effect of the reaction temperature, reaction time, catalyst concentration and methanol to oil volume ratio was evaluated using the response surface methodology (RSM). The actual levels of variables in the experimental design and the responses (yield of COME) are shown in Table 3.
The responses obtained in Table 3 with the four independent variables were correlated using the second order polynomial equation (Eq. 2). The following equation (Eq. 4) in terms of the actual factor was generated to predict the yield of COME

$$\begin{aligned} Y_{yield} &= 93.99 + 2.89X_1 + 2.35X_2 + 1.32X_3 + 8.89X_4 - 0.78X_1X_2 \\ &+0.58X_1X_3 + 0.80X_1X_4 + 0.060X_2X_3 - 0.46X_2X_4 + 5.33X_2X_4 \\ &-2.54X_1^2 - 4.75X_2^2 - 7.38X_3^2 - 13.55X_4^2 \qquad (4) \end{aligned}$$

Where X_1, X_2, X_3 and X_4 are the reaction temperature (^{0}C), reaction time (min), catalyst concentration (wt. %) and methanol to oil ratio (v/v) respectively. In order to check for the adequacy of the model, analysis of variance (ANOVA) and statistical analysis were performed.

The results of the ANOVA for the second order response surface model are shown Table 4 .
The model F - value of 33.79 with a very low probability value (< 0.0001) is an indication of the

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[16] Firestone, D. (1998). *Official methods and recommended practices of the American Oil Chemists' Society* (5th Ed.). Champaign, Illinois: AOCS.

significance of the fitted model. Each term in the model was also checked for its significance. Value of Prob < 0.05 is an indication that the model term is significant. In this case reaction temperature (X_1), reaction time (X_2), methanol to oil ratio (X_4) were significant model terms. The interaction term of catalyst concentration and methanol to oil ratio was also significant. The quadratic terms of reaction time, catalyst concentration and methanol to oil ratio were significant model terms.

Table 3. Actual levels of variables in the experimental design and yield of COME

Standard No	X_1 (Temperature)	X_2 (Reaction Time)	X_3 (Catalyst)	X_4 (Methanol: Oil)	Yield (%)
1	25	45	1	0.36	79.96
2	60	45	1	0.36	89.86
3	25	120	1	0.36	87.62
4	50	120	1	0.36	94.40
5	42.5	82.5	0.5	0.14	71.26
6	42.5	82.5	1.5	0.14	61.66
7	42.5	82.5	0.5	0.58	76.35
8	42.5	82.5	1.5	0.58	88.05
9	25	82.5	1	0.14	66.75
10	60	82.5	1	0.14	70.34
11	25	82.5	1	0.58	84.41
12	60	82.5	1	0.58	91.20
13	42.5	45	0.5	0.36	80.02
14	42.5	120	0.5	0.36	81.20
15	42.5	45	1.5	0.36	82.96
16	42.5	120	1.5	0.36	84.38
17	25	82.5	0.5	0.36	79.40
18	60	82.5	0.5	0.36	81.84
19	25	82.5	1.5	0.36	82.04
20	60	82.5	1.5	0.36	86.80
21	42.5	45	1	0.14	61.19
22	42.5	120	1	0.14	68.79
23	42.5	45	1	0.58	80.42
24	42.5	120	1	0.58	86.47
25	42.5	82.5	1	0.36	92.98
26	42.5	82.5	1	0.36	95.69
27	42.5	82.5	1	0.36	93.98
28	42.5	82.5	1	0.36	92.96
29	42.5	82.5	1	0.36	93.99

There was an insignificant lack of fit. According to Montgomery,[17] the lack of fit is an indication of the failure of the model representing the experimental data at which some points not included in the regression or variations in the model cannot be accounted for by random errors. This therefore implies, that if there is a significant lack of fit, the model is discarded. From the ANOVA, the "lack of fit F-value" of 5.57 implies that it is not significant relative to pure error. There is a 5.63 % chance that "lack of fit F-value", this large value could be due to noise.

Table 4. Analysis of variance (ANOVA) for response surface quadratic model

Source	Sum of square	degree of freedom	Mean square	F – value	p - value	
Model	2619.61	14	187.11	33.79	<0.0001	Significant
X_1	97.81	1	97.81	17.66	0.0009	
X_2	66.13	1	66.13	11.94	0.0039	
x_3	20.86	1	20.86	3.77	0.0727	
X_4	947.5	1	947.5	171.09	<0.0001	
X_1X_2	2.43	1	2.43	0.44	0.5182	
X_1X_3	1.35	1	1.35	0.24	0.6297	
X_1X_4	2.56	1	2.56	0.46	0.5077	
X_2X_3	0.014	1	0.014	2.60×10^{-3}	0.9601	
X_2X_4	0.84	1	0.84	0.15	0.7033	
X_3X_4	113.42	1	113.42	20.48	0.0005	
X_1^2	41.99	1	41.99	7.58	0.0155	
X_2^2	146.23	1	146.23	26.4	0.0002	
X_3^2	352.97	1	352.97	63.74	<0.0001	
X_4^2	1190.14	1	1190.14	214.91	<0.0001	
Residual	77.53	14	5.54			
Lack of fit	72.33	10	7.23	5.57	0.0563	not significant
Pure error	5.2	4	1.3			
Cor. total	2697.14	28				
CV = 2.35	$R^2 = 0.9713$		Adj. $R^2 = 0.9425$		$\text{Pred.}\,R^2 = 0.8425$	

The coefficient of variation (CV) is 2.86 %. A CV of more than 10 % is an indication that the variation in the mean value is high and does not safisfactorily develop an adequate response model. Generally,

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[17] Montgomery, D. (2001). *Design and analysis of experiment* (5th Ed.). New York: Wiley. 445 – 492.

the CV should not be greater than 10 %.[18] The suitability of the model was also tested using the coefficient of determination (R^2). The coefficient of determination is the proportion of variation in the response that is attributed to the model. For a good fitted model, R^2 should not be less than 0.8. R^2 values that are close to unity signify the suitability of the fitting empirical model to the actual model. From the analysis, the R^2 value was 0.9713. A large value of R^2 does not always imply the adequacy of the model. Thus, an adjusted R^2 value of over 0.9 more appriopriately evaluates the model adequacy. The adjusted R^2 value was 0.9425.

Fig. 1 shows the plot of the predicted versus actual experimental values. Most of the data points on this plot lie close to the straight line with some points above and below the line.

3.2 Effect of variables on the yield of COME

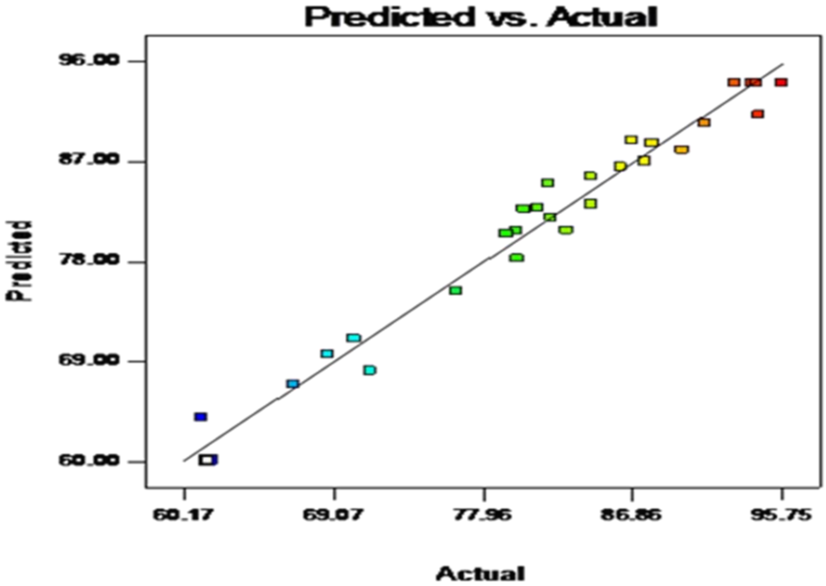

Fig. 1. Plot of predicted versus actual experimental values of COME yield

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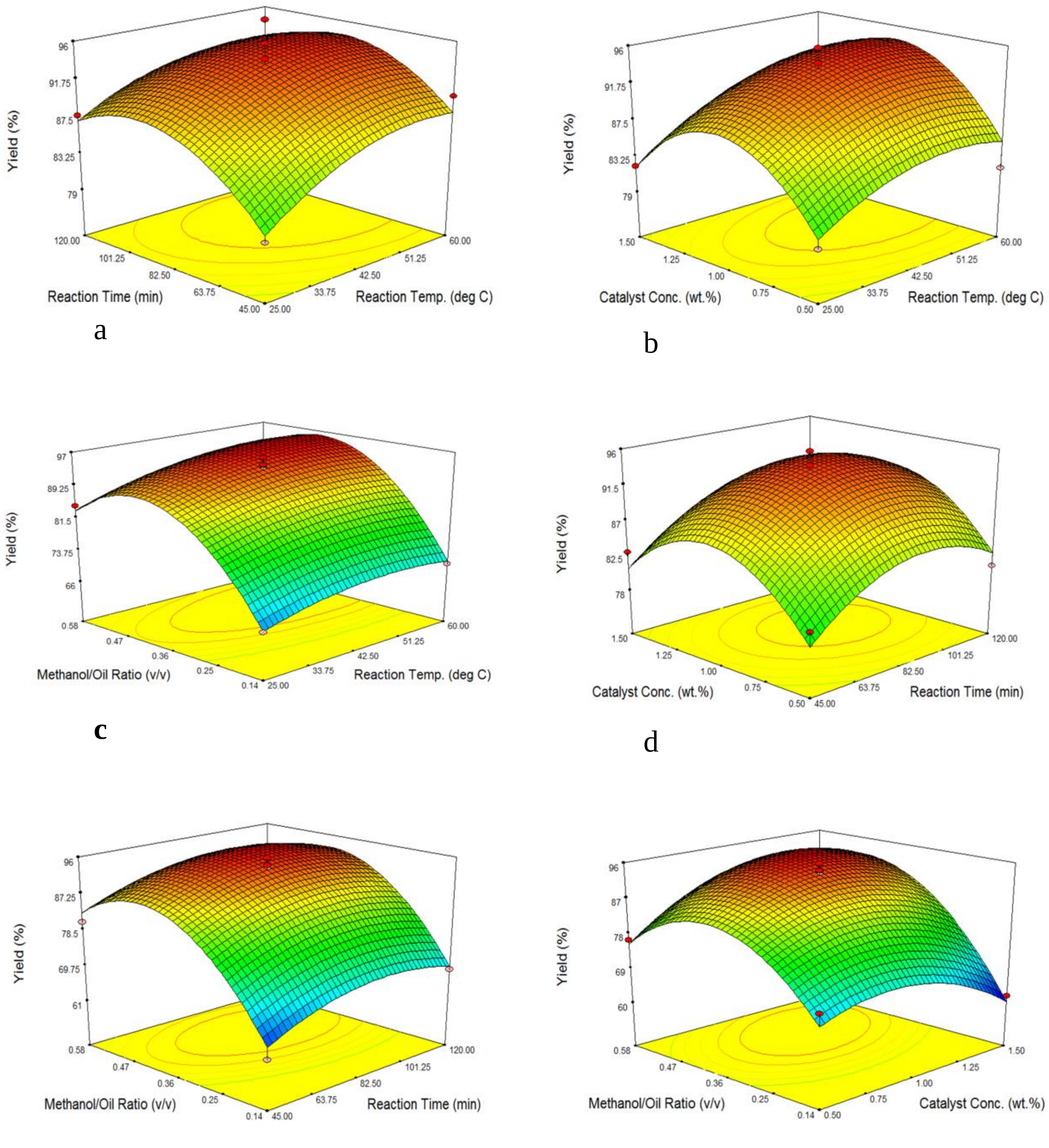
[18] Daniel, W. W. (1991). *Biostatistics: A foundation in the health sciences* (5th Ed.). New York: Wiley. 25 – 31.

Figs. 2 (a – f) show the response surface plots which are graphical representations of the model equation for the optimization of the transesterification reaction variables. These plots describe the combined effect of two variables on the yield of COME while keeping the other two variables constant at their central point.



a

b

c

d

e

f

Fig.2. Combined effect of (a) time and temperature (b) catalyst concentration and temperature (c) methanol oil volume ratio and temperature (d) catalyst concentration and time (e) methanol oil volume ratio and time (f) methanol oil volume ratio and catalyst concentration

The response surface plot for the combined effect of time and temperature is shown in Fig. 2a. The data were generated by keeping catalyst concentration and methanol to oil volume ratio at a constant central level and varying the other two variables within their experimental ranges. From the plot, the COME yield increased to a maximum with increase in time and temperature. Comparing both variables, the effect of increasing temperature on the yield of COME is similar to that of increase in time. It was also observed that when the time was fixed at 45 min and the temperature increased from 25 to 60 ^{0}C, the yield increased from 79 to 89.44 %. On the other hand, increasing the time from 45 to 120 min at a fixed temperature of 25 ^{0}C, the yield increased from 79 to 89.5 % and then decreased to 87.5 %. This reduction may be due to the fact that the catalyst was in excess of the alcoholic solution. At 107.5 min all of the reacting mixture had been used up, so running it any further would cause more of glycerol to form thereby decreasing the yield of the COME.

The COME yield obtained while varying catalyst concentration and temperature at a fixed time of 82.5 min and methanol to oil volume ratio of 0.36 is shown in Fig. 2b. From the surface plot, It was observed that when the catalyst concentration was kept constant and temperature increased from 25 to 60 ^{o}C, COME yield increased, while at a constant temperature of 25 ^{o}C, increasing catalyst concentration from 0.5 to 1.5 wt. %, an initial increase in COME yield was noticed. Further increase in catalyst concentration from 1.06 wt.% resulted in the reduction of the COME yield. The reduction may be due to the fact that at high concentrations of catalyst, the yield decreased due to the enhancement of saponification reaction causing the triglyceride to form soap faster than it formed ester.

Fig. 2c shows the effect of methanol to oil volume ratio and temperature at a fixed time of 82.5 min and catalyst concentration of 1 wt. %. The response surface plot shows that the COME yield increased with increase in methanol to oil ratio as well as temperature. Comparing both variables, the effect of increase in methanol to oil ratio on COME yield is far higher than the increase in temperature. This was further confirmed at constant temperature of 25 ^{o}C and varying methanol to oil volume ratio from 0.14 to 0.58. The yield increased from 66 to 89.22 % and then decreased to 85.3 %. This drop may be due to the fact that the methanol is in excess of the castor oil. Therefore, there is incomplete conversion of the castor oil to COME. At constant methanol to oil volume ratio, it was observed that increase in temperature had minimal effect on the COME yield.

The response surface plot for the combined effect of catalyst concentration and time is shown in Fig. 2d. At a constant temperature of 42.5 ^{o}C and methanol to oil volume ratio of 0.36, varying catalyst concentration and reaction time, the yield of COME increased to a maximum with reaction time as well as catalyst concentration. Also at a fixed time of 45 min and increasing catalyst concentration from 0.15 to 1.5 wt. %, the yield increased from 78 to 84 % and the dropped to 82 %. This may be due to excess catalyst concentration. On the other hand, at a fixed catalyst concentration of 0.5 wt. % by increasing the reaction time from 45 to 120 min, the yield increased from 78 to 84 %.

Fig. 2e shows a variation of COME yield with methanol to oil volume ratio and time at a fixed temperature of 42.5 ^{o}C and catalyst concentration of 1 wt. %. It was observed that as the time is kept constant at 45 min, by increasing methanol to oil volume ratio from 0.14 to 0.58, the COME yield increased from 61 to 87.27 % and then decreased to 85.33 %. This trend may be attributed to separation problems resulting from excessive methanol thereby minimizing the contact of triglyceride molecules on the catalyst's active site, which could decrease the catalyst activity. Moreover, methanol with one polar hydroxyl group can work as an emulsifier that enhances emulsion causing separation of ester layer difficult from the water layer and when glycerol remains in solution it will drive the equilibrium back to the left thereby lowering the yield of the ester.[19]

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[19] Leung, D. Y. and Guo, Y. (2006). Transesterification of neat and used frying oil: Optimization of biodiesel production. Fuel Process Technol. 87, 883 – 900.

The effect of varying methanol to oil ratio and catalyst concentration at constant temperature (42.5 ^{o}C) and time (82.5 min) is shown in Fig. 2f. At a fixed catalyst concentration of 0.5 wt. % and increasing methanol to oil volume ratio of 0.14 to 0.58, it is observed that the COME yield increased from 60 to 80 % and then decreased to 76 %. This was because at methanol to oil ratio of 0.38 v/v, the entire active sites of the catalyst had been occupied, so any further increase in the methanol to oil ratio will lead to plugging, i.e. accumulation of the reacting mixture on the surface of the catalyst thereby hindering its activity and by implication reducing the yield of the COME. On the other hand, at constant methanol to oil volume ratio of 0.14, increasing catalyst concentration from 0.15 to 1.5 wt. %, no significant increase in yield was observed. This is because at 0.5 wt.%, almost all the active sites would have been occupied, hence any further increase in the catalyst concentration will lead to little increase in COME yield.

3.3 Optimization of the transesterification reaction

The optimal values for the independent variables were obtained by solving the second order polynomial equation (Eq. 2) using the Design Expert 7.0.0 software. The optimum conditions for COME yield are: temperature 46.88 ^{0}C, reaction time 107.41 min, catalyst concentration 1.03 wt. %) and Methanol to oil ratio 0.44 v/v. The model predicts that the maximum yield of COME that can be obtained under these optimum conditions of these variables is 95.44 %.

In other to verify the suitability of the model for predicting the optimum response value, transesterification reactions were carried out under these optimal conditions and the results were compared with the predicted values obtained using the model equation. The experimental yield value was calculated to be 95.41 % which is close to the predicted value as shown in Table 5.

Table 5. Model verification under optimum conditions

Reaction Temperature	Reaction Time	Catalyst Concentration	Methanol to oil Ratio	Yield of COME	
(^{o} C)	(min)	(wt. %)	(v/v)	Experimental result	Predicted result
46.88	107.41	1.03	0.44: 1	95.41	95.44

3.4 Physico-Chemical Properties of COME

The physico-chemical properties of the COME produced at optimum conditions were compared with the ASTM Biodiesel standard as shown in Table 6. Apart from the density of the COME (911.6 kg/m^{3}), the values of other properties, viscosity (8.59 mm^{2}/s), acid value (0.16 mg KOH/g), flash point (194 ^{o}C), moisture content (0.02 %), iodine value (80.44 mg I_2/100g) fall within the ASTM D6751 limit for biodiesel.

Table 6. Physicochemical Properties of COME with comparison with ASTM standard

Property	Unit	COME	Test method ASTM	ASTM D6751 Limit
Density (27 ^oC)	(kg/m^3)	911.6	D1298	860 – 900
Acid Value	(mg KOH/g)	0.16	D664	0.8 max
Viscosity (27 ^oC)	(mm^2/s)	8.590	D445	1.9 - 6.0
Flash Point	(^{o} C)	194	D93	130 min
Cetane Number		39.61	D613	47 min
Moisture Content	(%)	0.02		< 0.05
Higher Heating Value	(MJ/kg)	43.42		-
Peroxide Value	(meq.O_2/kg)	2.498	AOCS	-
Iodine Value	(mg I_2/100g)	80.44	AOCS	120 max

4. CONCLUSION

In this study, the transesterification of castor oil was optimized using a constant volume reactor with temperature controlled magnetic stirrer apparatus. The effect of four independent variables (temperature (^0C), reaction time (min), catalyst concentration (wt.%) and methanol to oil (v/v) during transesterification of castor oil were evaluated by RSM. The following conclusions were drawn from this study:

1. Temperature, reaction time and methanol to oil ratio were found to have the most significant effect on the yield of COME. But out of all these variables, the most significant was the methanol to oil volume ratio.
2. The optimum conditions for the transesterification process were found to be temperature (46.88 ^oC), reaction time (107.41 min), catalyst concentration (1.03 wt.%) and methanol to oil volume ratio (0.44 v/v). At these conditions, the experimental yield obtained was found to be 95.41 %, an error of 0.03 % from the predicted value of 95.44 %.
3. The COME produced at the optimum conditions of parameters had fuel properties that satisfied the ASTM D5751 biodiesel standard.

The use of RSM for optimizing the transesterification reaction of castor oil to COME has been established in this study.