
Optimization of Biodiesel Production from Crude Cotton Seed Oil Using Central Composite Design

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To cite this article:

Mustapha Mujeli, Haruna Mavakumba Kefas, Awwal Shitu, Ibrahim Ayuba. Optimization of Biodiesel Production from Crude Cotton Seed Oil Using Central Composite Design. *Advances in Biochemistry*. Vol. 4, No. 6, 2016, pp. 94-100. doi: 10.11648/j.ab.20160406.14

Received: August 24, 2016; **Accepted:** September 10, 2016; **Published:** December 16, 2016

Abstract: Cheap raw materials and optimum process conditions of a transesterification reaction continued to be the most essential factors in determining the production of the biodiesel in commercial quantity to meet up the current global demand. In this study the crude cottonseed oil was used as an economical feedstock for biodiesel production since its demand as a cooking oil has reduced due to health issues related to its consumption. The process variables affecting the transesterification reaction such as methanol/oil ratio (4:1-9:1 mol/mol), catalyst weight (0.5-2%), temperature (40-65°C), reaction time (50-120 min) were optimized using rotatable central composite design of the response surface methodology in order to enhance the percentage yield of the biodiesel production. The maximum biodiesel yield (93.34%) was achieved under 8.08:1 mol/mol methanol/oil ratio, 1.87% catalyst weight, 40°C reaction temperature and 120 min reaction time. The properties of the biodiesel produced which include kinematic viscosity, density, cloud point, pour point and flash point were determined and compared with the European fatty acid methyl ester standard.

Keywords: Biodiesel, Cottonseed Oil, Central Composite Design (CCD), Biodiesel Properties

1. Introduction

The environmental impacts and the global instability of conventional fuel supply have forced the policy makers and scientists to look for alternative sources of energy. Biodiesel is a promising substitute for petroleum based fuels due to its unique properties of being renewable, biodegradable, non-toxic and better exhaust gas emission [1], [2]. The biodiesel utilization as a source of energy will reduce the environmental pollution problems caused by the petroleum based fuels and the over dependence on a few regions for world energy source.

Biodiesel is a mono alkyl ester of long chain fatty acid (methyl, ethyl, or propyl) mainly derived from alcoholysis of tri-alkyl glycerides (TAG) of vegetable oils (e.g. peanut oil, cottonseed oil, soybean oil, palm oil) or animal fats [3], [4]. It is produced mainly through transesterification reaction comprising feedstock and alcohol in the presence of a catalyst (methanol, ethanol or enzyme), on the other hand, it

can also be produced through direct blending, micro emulsification and pyrolysis [5]. The choice of catalyst for alcoholysis reaction depends on the free fatty acid (FFA) content of the feedstock. Usually, base catalysts (such as NaOH, KOH, NaOCH₃, CH₃OK) are more effective than acid catalysts, but their tendency of generating soaps when the FFA of the feed oil is higher than 3, which lead to difficulties in the downstream purification of the product [6]. Otherwise, two steps are carried out; esterification followed by transesterification using an acid catalyst when the FFA of the raw material is high. Alternatively, enzymatic catalyst is used when the feedstock FFA is high, but the high cost of enzymes and longer reaction period makes them less attractive.

To produce biodiesel in commercial quantity in order to meet up the present world's demand depends chiefly on the accessibility and sustainability of the feedstock. Then, cheaper raw materials exploration remained the most essential factor that determines the final cost of the products when compared to the conventional diesel. The most

commonly used vegetable oils in commercial production of biodiesel include canola, rapeseed, karanja, cottonseed, olive, palm, peanut, soybean, sesame, linseed, corn, jatropha, mahu, castor, neem and sunflower oils [7], [8]. Lately, Cottonseed oil demand in the food industry is diminishing because of the health problems linked to its consumption. The imbalance between the percentage of Omega-6 fatty and Omega-3 fatty acid content of the cottonseed oil is regarded as harmful, unless supplemented elsewhere in the diet [9]. For that reason, it has a great potential of becoming inexpensive raw materials for biodiesel production compared to several edible oils.

Previous studies indicate that biodiesel yield of 77-98% is feasible from crude cottonseed oil when significant variables of the transesterification reaction such as methanol oil ratio, catalyst weight, reaction time, temperature and stirring speed are optimized using response surface methodology (RSM) technique. Therefore, Nabi *et al.* [10] used traditional optimization method and obtained maximum biodiesel yield of 77% from crude cottonseed oil under optimum variable conditions; methanol oil ratio (20:1), catalyst weight (0.5%), temperature (55°C) and reaction time (8 hours). Similarly, Qian *et al.* [11] attained optimum biodiesel yield of 98% from *in situ* transesterification of crude cottonseed oil at 135:1 methanol oil ratio, 0.1 mol/L catalyst concentration, 40°C temperature and 3 hours reaction time. However, traditional method of process optimization has shortcomings in assessing the effects due to interactions between the process parameters which lead to local optimization solutions. Thus, global optimum solution could be achievable when statistical optimization techniques such as Response surface methodology (e.g. central composite design, Box-Behnken, D-optimal) Factorial Design (e.g. 2-level factorial, Plackett Burman, Taguchi) and mixed design (e.g. Simplex lattice, Simplex Centroid, Distance Based) are utilized for process optimization. Recently, Onukwuli *et al.* [12] used 2-level factorial design and optimize process parameters of refined cottonseed oil transesterification reaction. Their optimum biodiesel yield (96%) was achieved under 6:1 methanol oil ration, 0.6% catalyst weight, 55°C temperature and 60 min reaction time. Similarly, Fan *et al.* [1] used RSM technique and obtained optimum biodiesel yield (97%) at 7.9:1 methanol oil ratio, 1.0% catalyst weight, 53°C temperature, 268 rpm mixing speed and 45 min reaction time. Moreover, the biodiesel yield, temperature and reaction time of the biodiesel production were further reduced when Joshi *et al.* [4] utilized central composite design (CCD) method to enhance the production of biodiesel using high gossypol content cottonseed oil. They achieved a maximum biodiesel yield of 98% at 20:1 methanol oil ratio, 1.07% catalyst weight, 25°C temperature and 30 min reaction time.

However, it seems that no enough studies in the literature specifically on the application of central composite design for biodiesel production optimization using crude cottonseed oil. Moreover, it is very difficult to compare and evaluate results for pilot scale studies. Therefore, enough process data, such as alcohol/oil molar ratio, amount of catalyst, reaction

temperature and reaction time and their interaction effects on the biodiesel yield and quality are required in order to transform the process to commercial level. Response Surface methodology (RSM) developed by Box and Wilson [1] has the ability to build a second order quadratic model for estimation of dependent variable (response) using only two-level factorial design. The objective of this present study is to employ rotatable central composite design (CCD) of the RSM technique and optimize the process factors of crude cottonseed oil transesterification reaction.

2. Materials and Methods

2.1. Materials

The crude cottonseed oil derived from expeller (i.e., screw pressed cottonseed) was obtained from the Yola oil mill, Adamawa, Nigeria. The chemicals used (methanol, sodium hydroxide, Isopropanol, phenolphthalein solution, and n-hexane) were of analytical grade. All the glass wares and containers were washed with liquid detergent and rinsed with deionized water. The containers were rinsed with appropriate reagents before utilization.

2.2. Methods

2.2.1. Determination of Free Fatty Acid (FFA)

The free fatty acid of the oil was determined using Official Methods and Recommended Practices of the AOCS [13]. A precise quantity of crude cottonseed oil was weighed into an Erlenmeyer flask and 75 ml of Isopropanol and 15 ml of n-hexane were added to the flask. Three drops of phenolphthalein solution were added to the mixture and titrated against 0.1N sodium hydroxide solution to a steady pink end point. The procedure was repeated twice and the average titre value of the sodium hydroxide used was recorded when the precision of each run does not exceed 0.1%. Thus, the free fatty acid (FFA) of the sample was calculated using equation 1:

$$\text{FFA (\%)} = \frac{(28.2 \times \text{Volume of titre value} \times 0.1 \text{ N NaOH})}{\text{Weight of oil used (g)}} \times 100 \quad (1)$$

where 28.2 is 10% molecular weight of oleic acid.

2.2.2. Transesterification Process

The CCD design generated 21 experiments and the corresponding responses (% yield of biodiesel) were determined experimentally using a batch scale transesterification process using crude cottonseed oil.

A known quantity of crude cottonseed oil was weighed and transferred into a 250 ml conical flask and preheated at 60°C in a water bath for 30 min. Some quantity of sodium hydroxide pellets was weighed and dissolved into the desired amount of methanol and allowed to dissolve completely. The quantities of oil, methanol and sodium hydroxide were weighed according to the values provided in the experimental design. The preheated oil and the dissolved methanol-sodium hydroxide mixture (sodium methoxide) were transferred into

a rotary evaporator spin flask and placed in the hot water bath. Similarly, the reaction temperature and time were regulated based on the experimental design objective. The reaction was stopped at the end of each specific time and the product was transferred into the separating funnel and allowed to stand overnight for proper sedimentation. The glycerol was then drained the following day from the separating funnel since it is heavier than the biodiesel. Subsequently, equal volume of warm water (50°C) was added to the biodiesel and gently mixed (to avoid emulsification) and the mixture was allowed to stand overnight. Lastly, the biodiesel was separated from the water using separating funnel and dried at 110°C for 45 minutes. The procedure was repeated 20 times corresponding to the total number of experiments generated by the CCD design. The percentage yield of biodiesel for each experimental run was evaluated by relating the weight of biodiesel obtained and the weight of crude cottonseed oil consumed by the reaction using equation 2.

$$\% \text{ yield} = \frac{\text{weight of biodiesel produced (g)}}{\text{weight of oil used (g)}} * 100 \quad (2)$$

2.2.3. Design of Experiment and Statistical Analysis

The design of experiment (DOE) and statistical analysis were performed using the Design-Expert Software 10.0.1. The central composite design (CCD) is the most widely used second order design for fitting the response surface regression models. In this study the design was used to design and optimize four factors that might have an influence on the transesterification process of crude cottonseed oil, i.e. methanol-to-oil ratio, catalyst weight, temperature and reaction time. The parameters low and high values with their corresponding alpha levels for the rotational CCD are shown in Table 1.

Table 1. The experimental factors and their corresponding levels.

Name	Low	High	-alpha	+alpha
Methanol/oil (mol/mol)	4	9	2.2955	10.7045
Catalyst wt. (%)	0.5	2	-0.0113	2.5113
Temperature (°C)	40	65	31.4776	73.5224
Time (min)	50	120	26.1373	143.8627

In order to minimize the experimental runs, the small CCD design was chosen and no replicate of points due to factorial levels are added. Therefore, the four factors were varied over 5 levels; + and - 4 factorials, + and - 4 axial points ($\alpha=1.68179$ for rotatable design) and 5 center points which provided 8, 8, and 5; 21 total number of experiments, respectively. The alpha value of rotatable design was computed using equation 3:

$$\alpha = F^{1/4} \quad (3)$$

where F is the total number of points due to factorial design (i.e. 8 experiments in this case).

The 21 experimental runs were performed and their corresponding responses (% yield of biodiesel) obtained for each run was recorded in the response column of the design

expert table. The design was implemented using second order response surface model and the performance of the model was evaluated using Analysis of variance (ANOVA).

2.2.4. Optimization Process

The numerical optimization tool of the Expert-Design was used to optimize the percentage yield of the biodiesel, according to the response surface model earlier developed. The design range of the four factors (dependent variables) were selected based on the design boundary conditions. However, 97% biodiesel yield was chosen as the optimum target in order to achieve the European standard of biodiesel yield of 96.5 wt.% [14].

2.2.5. Biodiesel Analysis

The produced biodiesel using the optimum process conditions was purified and analyzed according to the European Committee for Standardization requirement [15]. The biodiesel density, Kinematic viscosity, flash point, cloud point and pour point properties were determined at the Nigerian National Petroleum Corporation (NNPC) Depot Laboratory, Yola, Nigeria. The kinematic viscosity and the density of the biodiesel were determined using ISO 3104 [16] and ISO 3675 [17] standards, respectively. While, ISO 1523 [18], ISO 3015 [19] and ISO 3016 [20], were used to determine the flash point, cloud point, and pour point of the biodiesel, respectively.

3. Results and Discussion

3.1. Free Fatty Acid of the Oil

Free fatty acid content of the raw materials for biodiesel production using alkaline catalyst has great influence on the product quality and yield. High tendency of soap generation happens when the FFA of the feedstock is higher than 3% [6]. Consequently, the soap increases the viscosity of the transesterification products and make the separation of the biodiesel from the by-products more difficult [21]. Thus, the FFA value obtained in this study was 2.44%, which is within the recommended range for alkaline catalyst transesterification. However, the level of impurities in the feedstock could be responsible for this high FFA content obtained in this study, since [22] obtained a lower value of 1.69 using the same methodology.

3.2. Design of Experiment & Statistical Analysis

The effects of each independent variable (methanol/oil ratio, catalyst wt., temperature and time) and their respective interactions on the biodiesel yield were investigated using the CCD technique. A second order quadratic model was acquired to describe the precise correlation between the response and the independent variables. The experimental and predicted biodiesel yields (%) are presented in Table 2. The empirical correlation between the biodiesel yield (%) in terms of coded independent factors is described by Equation 4:

$$\text{Yield} = +53.76 - 2.74 * A + 5.86 * B - 17.74 * C + 2.38 * D +$$

$$8.25*AB+4.65*AC+10.58*AD+4.00*BC+1.39*BD-13.90*CD+1.98*A^2-3.34*B^2-3.88*C^2+1.91*D^2 \quad (4)$$

where A, B, C and D are the methanol/oil ratio, catalyst wt., temperature and time respectively. The optimum biodiesel yield (%) can be estimated by equation 4 using the best values of the model variables and their interactive effects within the initial boundary conditions of the model.

The statistical analysis of the model was evaluated using analysis of variance (ANOVA). The ANOVA results (Table 3) indicates that the model terms could be used to explain the response (biodiesel yield) effectively. The model has a high correlation coefficient ($R^2=0.9995$) between the predicted and experimental biodiesel yield (%), the result revealed that about 99.95% variations of the response could be explained

by the model and only 0.05% deviation could be possible due to errors. Although, if the model consists of several independent variables adjusted R^2 is more appropriate to be used for model evaluation because it decreases the number of insignificant terms in the model when the model factors increases. Hence, the adjusted R^2 (0.9983) and predicted R^2 (0.9548) of the model are in good agreement and only less than 0.2 difference, this shows a good fit between the actual and predicted biodiesel yield (%). In addition, the signal to noise ratio (adequate precision) of the model ($98.892 > 4$) has a strong signal and can be used for the biodiesel yield optimization as far as the model factors are within the design space.

Table 2. Actual and predicted biodiesel yield (%).

Run	Space Type	Methanol/oil ratio (mol/mol)	B:Catalyst wt. (%)	Temperature (°C)	Time (min)	Actual Yield (%)	Predicted Yield (%)
1	Axial	6.50	1.25	31.48	85.00	72.13	72.63
2	Axial	2.30	1.25	52.50	85.00	63.50	63.97
3	Axial	6.50	1.25	73.52	85.00	12.53	12.97
4	Center	6.50	1.25	52.50	85.00	54.00	53.76
5	Axial	6.50	-0.01	52.50	85.00	34.00	34.47
6	Center	6.50	1.25	52.50	85.00	54.60	53.76
7	Factorial	4.00	2.00	40.00	120.00	76.60	76.26
8	Factorial	4.00	0.50	65.00	50.00	53.00	52.68
9	Center	6.50	1.25	52.50	85.00	53.30	53.76
10	Axial	10.70	1.25	52.50	85.00	54.30	54.77
11	Axial	6.50	1.25	52.50	143.86	62.70	63.17
12	Axial	6.50	2.51	52.50	85.00	53.70	54.17
13	Axial	6.50	1.25	52.50	26.14	54.70	55.17
14	Factorial	9.00	2.00	40.00	50.00	43.00	42.66
15	Center	6.50	1.25	52.50	85.00	54.00	53.76
16	Factorial	9.00	2.00	65.00	50.00	52.60	52.28
17	Factorial	4.00	2.00	65.00	120.00	12.00	11.68
18	Factorial	9.00	0.50	40.00	120.00	76.50	76.16
19	Factorial	9.00	0.50	65.00	120.00	14.50	14.18
20	Factorial	4.00	0.50	40.00	50.00	78.00	77.66
21	Center	6.50	1.25	52.50	85.00	54.00	53.76

In addition, the model fitness was further assessed using the F-value test for comparing the sources mean square of the residual and P-value for testing the null hypothesis of the observed F value of possible effects of the variables. Therefore, the larger F-value (863.51) and smaller P-value (0.0001) of the model indicated the model was significant, because only 0.01% probability that F-value this large at 95% confidence level could be due to an error.

Table 3. Analysis of Variance (ANOVA) of the model.

Source	Sum of Squares	DF	F-value	P-value
Model	7532.73	14	863.51	< 0.0001
Residual	3.74	6		
R^2	0.9995			
Adjusted R^2	0.9983			
Predicted R^2	0.9548			

3.3. Effects of Variables Interactions

The effects between two variable interactions on the biodiesel yield (%) are best displayed by the response surface

3D contour plots (Fig. 1a-d). The 3D plot (Fig. 1a) illustrates the response surface area of the biodiesel yield (%) as a function of methanol/oil ratio and catalyst weight, when temperature and time remained constant at 52.5°C and 85 min, respectively. The plot indicates no simple optimal point, however, two separate extreme locations could be viable for maximum biodiesel yield (%); at the maximum levels of both the two factors or their minimum design values. It is obviously shown that an increased in methanol/oil ratio equally demand an increased in catalyst wt. for the best result. The maximum biodiesel yield could be possible at any point between the minimum and the maximum methanol/oil ratio values, provided that other variables are maintained at optimal levels. Therefore, the complete conversion of the triglyceride to methyl esters is possible when the methanol-to-oil ratio is in excess of the stoichiometric quantity [23]. Similarly, favorable conditions of the backward reaction at high catalyst wt. enhances the conversion of the reactants to biodiesel [4].

The interactive influence of the methanol/oil ratio and the

reaction temperature on the biodiesel yield (%) are highlighted in Fig. 1b. The contour plot revealed that an increased in the reaction temperature, decreases the biodiesel yield both at high and low methanol/oil ratio. This obviously indicates that temperature has more control on the methyl ester conversion when the methanol/oil ratio amount is within the range that would keep the reaction at equilibrium. Also, the severe decreased in biodiesel yield as the temperature approaches the methanol boiling point (from 49 to 65°C) might be because of methanol reduction due to evaporation, particularly when the feedstock oil has a low viscosity [24].

Similarly, Fig. 1c show negative influence of rapid increased in reaction temperature (i.e. from 49°C upward) on the biodiesel yield when catalyst wt. virtually remained constant. The saturation of the catalyst wt. amount on the design surface (0.5 to 2) could be the reason why it has less effect on the yield, because both the two factors have similar goals of enhancing the rate of reaction of product formation. Moreover, the increased in temperature beyond 49°C has a high tendency of promoting the saponification process by the base catalyst, especially when the feedstock oil has high FFA content [25]. On the other hand, the biodiesel yield (%) rises as the reaction time increases at low temperature (40°C) and the situation reverses when the temperature reaches 64°C (Fig. 1d). Generally, the best options for obtaining a maximum biodiesel yield lies within the range of (41-50°C)

and (80-120 min), respectively.

3.4. Biodiesel Yield Optimization

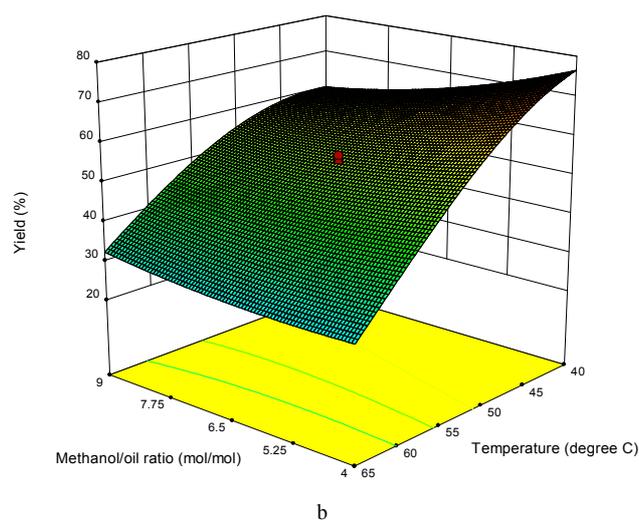
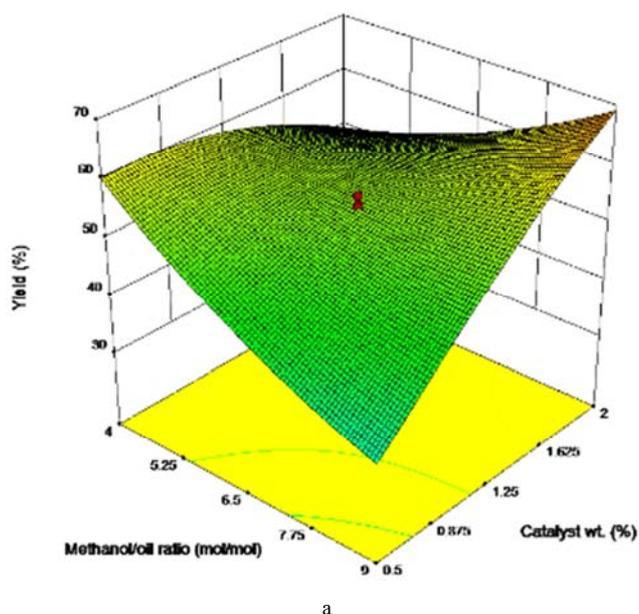
The numerical optimization tool of the design expert 10.0.1 software was used to obtain the optimum values of the independent variables using the model regression equation (eq. 4). The target of the biodiesel yield (%) was set based on the European standard (97%) proposal while the variables were set according to their initial design range (Table 1). The optimum predicted yield generated by the model was 93.34% at 8.08 mol/mol (methanol/oil ratio), 1.869%, (catalyst wt.) 40°C (temperature) and 120 min (time). The experimental biodiesel yield obtained at the optimum conditions was 86.04%, with a variation of 7.3% due an effect that might not be included in the design. For instance, the stirring speed of the process was kept at 200 rpm throughout the study. The mixing intensity is very significant in starting the esterification reaction, because it increases the surface area between methanol and oil [23].

3.5. Biodiesel Analysis

The maximum biodiesel yield achieved under the optimum conditions was characterized according to the European Committee for Standardization requirement [15]. The properties of the biodiesel obtained in this study are presented in Table 4.

Table 4. Properties of optimum biodiesel produced.

PROPERTY	EN:14214 (2008)	RESULT	Rashid et al. (2009)
Density @ 15°C (kgm ⁻³)	860-900	896	875
Kinematic viscosity@40°C (mm ² /s)	3.5-5.0	3.7	4.07
Flash point (°C)	>101	117	150
cloud point(°C)	-	6	7
pour point(°C)	-	2	6



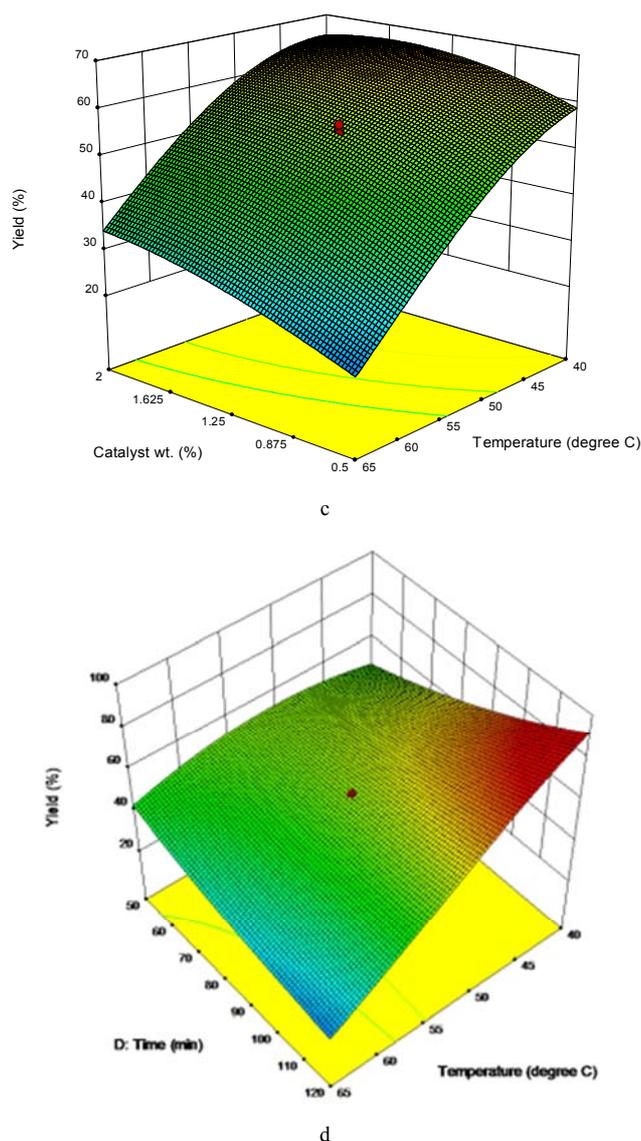


Figure 1. The 3D plots of independent variables interactions effect on the biodiesel yield.

The fuel characteristics such as viscosity, kinematic viscosity and flash point control the thermal efficiency of the biodiesel and its blends. The density is a key property in measuring the heating value of the fuel when it burnt completely. The density of the biodiesel obtained in this study (896 kg/m^3) is within the required range. The viscosity and flash point of the biodiesel are also in the range recommended by the European Committee for Standardization requirement [15].

4. Conclusions

Central composite design was used to design and optimize the second order response surface biodiesel yield (%) model using independent variables; methanol/oil ratio, catalyst wt., temperature and time. The analysis of variance (ANOVA) of the developed model indicates that the model was significant enough to predict the biodiesel yield (%) provided that the

model factors are within the design range. The maximum biodiesel yield (%) acquired using the numerical optimization procedure was 93.34% at 8.08 mol/mol, 1.869%, 40°C and 120 min for methanol/oil ratio, catalyst weight, temperature and time respectively. The result was validated experimentally and the average biodiesel yield obtained was 86.04%. However, the 7.3% difference obtained between the predicted and experimental values could be to an effect that is not present in the model, for example, stirring speed (in this study the stirring speed was kept constant at 200 rpm).

Acknowledgements

The authors would like to thank the laboratory technicians of the Chevron Biotechnology center (Modibbo Adama University of Technology, Yola) for their support in providing laboratory facilities for the research. We also express our gratitude to the laboratory staff of NNPC Yola Depot for their help in analyzing the biodiesel properties.

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