One-step production of Biodiesel from Crude Palm Oil planted in Gaanda using Methanol, Adamawa State Nigeria

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Abstract In the production of methyl esters (biodiesel), application of low cost high free fatty acids (FFA) feedstock may reduce its overall cost of production. In this research work, one-step transesterification process was performed to produce biodiesel using crude palm oil, and the process provided slightly low biodiesel yield compared to EN 14214 (96.5 %). The resultant product was subsequently transesterified. All the experimental runs carried out were designed using central composite design (CCD) coupled with surface response methodology (RSM) software (Design-Expert 9 software (Stat-Ease Inc., USA). Thus, the yied obtained via one-step transesterification process was EN 14214 (96.5 %) which is the required standard. Besides, the physical properties determined were also within the limits of the ASTM D6751 and EN 14214 standard specifications.

Keywords Biodiesel, Crude Palm Oil

1. Introduction

The high energy demand in the industrialised world, as much in the domestic sector, as in transport and industry, its increase, and the derived problems of the widespread use of fossil fuels, make increasingly necessary the development of renewable energy sources of limitless duration and smaller environmental impact than the traditional ones. The major energy demand is fulfilled from the conventional energy resources like coal, petroleum and natural gas (NG). Petroleum-based fuels are limited reserves concentrated in certain regions of the world. These sources are in the verge of getting extinct. The scarcity of known petroleum reserves will make renewable energy resources more attractive. Biodiesel obtained from energy crops produces favourable effects on the environment, such as a decrease in acid rain and in the greenhouse effect caused by combustion. Due to these factors and to its biodegradability, the production of biodiesel is considered an advantage to that of fossil fuels. In addition to this, it also shows a decrease in the emission of CO_2, SO_2 and unburned hydrocarbons during the combustion process.

In Nigeria, biodiesel (liquid biofuel from vegetable oils or animal fats) seems to be of the promising alternative to fossil fuels. This is because the crops required for biodiesel production are cultivated in large quantity, under favourable climatic conditions. The global biodiesel production has grown from approximately 5 million gallons in 2001 to 250 million gallons in 2006 [1-2]. Although high oil prices have lately tended to reduce production of biodiesel, several forces may contribute to long-term expansion in the biodiesel industry [2]: High petroleum prices are raising diesel prices and the likely increasing costs of future oil production; Depletable resources follow Hotelling's prices in the long run and tend to increase over time, as petroleum is depleted; Government mandates, such as the provisions of the Energy Independence and Security Act of 2007 that includes mandates of up to 36 million gallons of biofuels; the public and government's concern over global warming may provide a value for biodiesels carbon-dioxide (CO_2) recycling characteristics [3].

Depletion of world's petroleum reserve and the increasing environmental concern are the key issues leading to the search for alternative fuels source. This study explored and exploited esterification and transesterification techniques used for biodiesel production using crude palm oil.

2. Biodiesel Fuel

Biodiesel is a biodegradable, sustainable and clean energy source that can be derived from a variety of biomass feedstocks oils such as waste vegetable oil, yellow grease, animal fats, and virgin vegetable oils [4]. It has proven itself as a technically sufficient alternative diesel fuel in the fuel market since the beginning of the 1990s. Biodiesel is defined as a fuel made up of mono-alkyl esters of long chain fatty acids derived from vegetable oils or animal fats, designated B100, and meeting the requirements of national fuel specification, American Society for Testing and Materials (ASTM D6751), European norm (EN-14214), or its equivalent [5-7].

Feedstocks for the Production of Biodiesel

Feedstocks contribute a major share in the cost of the production of biodiesel fuel. The selection of feedstocks is depended chiefly on its cost and availability. Countries such as United States of America and those belonging to European community are self-dependent in the production of edible oils and even have surplus amount to export [8]. Hence, edible oils such as rapeseed and soybean are used in European Nations and United States of America, respectively. Similarly, countries with coastal area such as Malaysia and Indonesia have surplus palm oil and are utilized for the production of biodiesel. In general, feedstocks for the production of biodiesel can be classified into three groups: animal fats, vegetable oils (edible or non-edible oils), and used waste cooking oil. If we consider the top ten countries in terms of absolute biodiesel synthesis, the average feedstock dependence is 20% for animal fats, 11% for coconut oil, 22% for palm oil, 28% for soybean oil, and 5% each for rapeseed, sunflower and olive oils [9].

Vegetable Oils as Feedstocks for the Production of Biodiesel

This is a flammable or non-flammable, viscous, greasy, liquid substance at room temperature and insoluble in water but soluble in organic compounds which is derived from plants, animals or minerals deposits or manufactured artificially and can be used for food, as fuel, and lubricant [7, 10-11]. There are quite a lot of seeds and nuts which are rich in terms of oil contents, thus Soya beans, groundnut and palm kernel, pipe or mustard seed sesames, all are important sources of edible oils [9, 12].

Edible Vegetable Oils

Globally, edible vegetable oils are the main sources for the production of biodiesel fuel; more than 95% of the global biodiesel production is made from edible vegetable oils. Use of edible oils worldwide increased to 6.6 million tons between 2004 and 2007, 34% of this increase is attributed to biodiesel [13]. The most used edible vegetable oils are consecutively rapeseed oil with 59% of total global biodiesel feedstock sources, followed by soybean (25%), palm oil (10%), sunflower oil (5%), and others (1%). Different investigations are being conducted to replace edible vegetable oils with non edible ones owing to the increase in edible vegetable oils demands for both bio-fuels and food [9, 11].

Advantages of biodiesel as diesel fuel

The advantages of biodiesel as diesel fuel are liquid nature portability, ready availability, renewability, higher combustion efficiency, lower sulfur and aromatic content [14], higher cetane number and higher biodegradability. Main advantages of biodiesel given in the literature include domestic origin, reducing the dependency on imported petroleum, biodegradability, high flash point and inherent lubricity in the neat form.

3. Material and Methods

Methanol, sulphuric acid, potassium hydroxide, crude palm-oil, sodium sulphate, transesterification reactor, water bath, beakers, separating funnel, evaporation apparatus, thermometer and conical flasks.

One-Step Alkali-catalyzed Transesterification Reaction

The alkali-catalyzed transesterification was performed in a transesterification reactor in accordance with the conditions provided in Table **1**. After the transesterification reaction was completed, the crude biodiesel mixture was allowed to settle overnight and was then separated into two layers. The bottom layer having a brownish red

color crude glycerol which contained other impurities (excess catalyst, and excess methanol etc) was drained using a separating funnel. The methyl esters along with the remaining impurities such excess catalyst and excess methanol etc in the upper layer were then transferred into the evaporation unit for the removal of excess methanol. The resultant biodiesel was then washed severally with acidified warm water to remove the remaining catalyst and the dissolved glycerol in the biodiesel phase. It is important to note that twenty (20) experimental runs were performed.

Table 1: Process Variables for Biodiesel Production

S/No.	Run.	Temp.^{o}C	Time. Min.	Conc. %
1	8	55.00	54.00	0.50
2	11	63.00	45.00	0.50
3	20	55.00	90.00	0.50
4	5	63.00	90.00	0.50
5	6	55.00	45.00	1.50
6	9	63.00	45.00	1.50
7	7	55.00	90.00	1.50
8	2	63.00	90.00	1.50
9	14	55.00	67.50	1.00
10	9	63.00	67.50	1.00
11	12	59.00	45.00	1.00
12	18	59.00	90.00	1.00
13	4	59.00	67.50	0.50
14	17	59.00	67.50	1.50
15	15	59.00	67.50	1.00
16	13	59.00	67.50	1.00
17	10	59.00	67.50	1.00
18	3	59.00	67.50	1.00
19	1	59.00	67.00	1.00
20	16	59.00	67.50	1.00

Statistical Analysis

Experimental data (Table 1) were investigated via central composite design coupled with response surface methodology, in order to fit the following second-order polynomial equation generated by Design-Expert 9 software (Stat-Ease Inc., USA). Second-order coefficients were generated by means of regression. The response was initially fitted to the factors using multiple regressions. The quality of the fit of the model was assessed by means of the coefficients of determination and analysis of variance. The quadratic response surface model was fitted to the following equation:

$$X = \beta_0 + \beta_1 Y_1 + \beta_2 Y_2 + \beta_3 Y_3 + \beta_{12} Y_1 Y_2 + \beta_{13} Y_1 Y_3 + \beta_{23} Y_2 Y_3 + \beta_{11} Y_1^2 + \beta_{22} Y_2^2 + \beta_{33} Y_3^2 \text{ ----Eq 1}$$

Where: X is the dependent variable; Y_1, Y_2 and Y_3 are the independent variables; β_0 is the intercept, β_1, β_2, β_3, are linear coefficients and β_{12}, β_{13} and β_{13} are interaction coefficients, β_{11}, β_{22} β_{33} quadratic coefficients, respectively.

Separation and Evaporation Processes

When the transesterification reaction was completed, two major products, methyl esters and glycerol were obtained. The glycerol phase was much denser compared to the biodiesel phase and it therefore settled at the bottom of the separating fuel. In this research work, simple decantation technique was then used to separate biodiesel phase from the glycerol phase. The reaction mixture was allowed to settle overnight in the separating funnel in order to allow complete separation of biodiesel and glycerol. As earlier mentioned, both the biodiesel and glycerol are contaminated with an unreacted catalyst, methanol, and oil during the transesterification reaction. Besides, soap that was generated during the process also contaminates the biodiesel and glycerol

phases. Although the glycerol phase tends to contain a higher percentage of contaminants than the biodiesel, a significant amount of contaminants is also present in the biodiesel. Unreacted methanol was removed by evaporation with evaporation equipment before the washing step to prevent excess methanol from entering the waste-water effluent. The primary purpose of this step is to remove the methanol in the biodiesel. This was done by heating the crude biodiesel to 70^{0}C for average of 30min [15].

Acidified Water Washing

Since both glycerol and methanol are highly soluble in water, water washing was very effective for removing both contaminants. It can also remove all residual potassium salts and soaps. The primary material for water washing is distilled warm water or softened water (slightly acidic). Warm water prevents the precipitation of saturated esters and retards the formation of emulsions with the use of a gentle washing action 2010). The acid neutralizes the residues of the basic catalyst and separates any soap residue that can be produced during the reaction. The soap reacts with the acid forming hydrophilic salts. The salt was removed during washing, while the free fatty acids remain in the biodiesel. The washing water was applied to remove any remaining catalyst, soap, salt, methanol and free glycerol from biodiesel. Neutralization before washing reduces the amount of water needed and minimizes the tendency to produce emulsions. The neutralization was done with 10% acid and 90% water. After the process of washing was completed, the biodiesel phase was dried using anhydrous sodium sulphate Na_2SO_4.

Analysis of Biodiesel

The yields of biodiesel samples were analyzed by a HP 6890 Gas Chromatogram (GC) equipped with a Flame Ionization Detector (FID) and capillary column DB23 (60-m×0.25-m×0.15-μm) according to a methodology proposed by Agilent. Normal hexane solutions of the biodiesel samples with a concentration of 100 mg/ml were injected by an auto injector at an oven temperature of 50 °C, which was then 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 (S. Baroutian, et al., 2011). Besides, other physical properties such as density, viscosity, cetane number, pour Point, moisture water content, cloud Point and flash point were determined using ASTM D6751 and EN 14214.

4. Results and Discussions

One-Step Alkali Transesterification Process

One-step transesterification was first performed using crude palm oil and methanol in the presence of potassium hydroxide as catalyst. Globally, the standard methyl ester yield is 96.5%, as specified by EN 14214. In this study, one-step transesterification reaction; the methyl ester yields for all the experimental runs conducted were below 96.5% (EN 14214), which signified that less refined palm oil was utilized. Table 2 present the methyl ester yields for the twenty (20) runs conducted.

Separation and Purification of Biodiesel

The biodiesel produced was first subjected to gravitational settling overnight and the two distinct layers (biodiesel and glycerol) were separated via decantation. As earlier mentioned the distinct densities difference between biodiesel and glycerol and also the differences in their polarities where biodiesel is hydrophobic and glycerol being hydrophilic made their separation faster. The residual methanol was separated using evaporation equipment (temperature: 70°C and time: 30min). However biodiesel still contain traces of impurities that need to be removed for the biodiesel produced to attain ASTMD6751 and EN14214. Therefore to achieve biodiesel standard specifications, water washing was carefully performed.

Biodiesel Acidified Water Washing

In the refining of crude biodiesel, acids including sulfuric acid, phosphoric acid, and hydrochloric acid are the mostly used agents. After acid use, distilled water is employed to wholly eliminate any water soluble impurity in biodiesel. Biodiesel is usually dried so as to be safely utilized on diesel engines, and be stored for long period of time [16]. Faccini et al. [17] used 10v/v% acidified water at a temperature of 55 ºC to thoroughly wash biodiesel samples. The process of washing the biodiesel sample was performed in the same transesterification reactor at a

temperature of 55ºC by means of constant stirring for 5 min. Afterward, the waste-water and biodiesel samples were estranged by means of a separatory funnel. The upper layer, consisting of refined biodiesel was then dried [17]. Furthermore, ethyl ester prepared from palm oil was refined using acidified water (5% of phosphoric acid). For biodiesel to meet specifications designed by ASTM D6751 standard, the water content of fuel ought to be minimized to a maximum value of 0.050% volume by volume.

In this research work, the biodiesel produced was separated from glycerol by allowing the samples to settle overnight. The resultant biodiesel was then purified using warm acidified water (10% H_3PO_4). In order to completely remove all the contaminants such as glycerol, residual alcohol, soap and catalyst etc, the biodiesel samples were thoroughly washed with distilled water. After which the biodiesel samples were transferred into a separatory funnel and set aside for 0.5 hr. The cleaned samples of biodiesel consisting of lighter colour was dried using 10% heated Na_2SO_4. Compared to EN 14214 standard specification (96.6%), most of the biodiesel samples after refining met the international standard specification for the yields of ester as shown in Table 2.

Additionally, it was noted that vacuum flash technique can also be used to dry refined biodiesel samples [18] [18]. Moreover, the process of biodiesel water washing can also be performed using rotary evaporation at a temperature of 70 ^{o}C [14].

Table 2: Yields of Biodiesel (One-step Transesterification)

Runs	Factor 1 A:Temperature (ºC)	Factor 2 B:Time (Min)	Factor 3 C:Cat.Conc (%)	Response 1 R1(Yield) (%)
1	63.00	45.00	1.50	84.36
2	59.00	67.50	0.50	88.36
3	59.00	67.50	1.00	91.78
4	59.00	90.00	1.00	93.04
5	59.00	67.50	1.00	93.48
6	59.00	67.00	1.00	92.53
7	55.00	45.00	0.50	87.75
8	55.00	54.00	0.50	95.17
9	55.00	90.00	1.00	90.16
10	59.00	67.00	1.00	90.16
11	63.00	90.00	1.50	92.53
12	63.00	90.00	0.00	85.81
13	59.00	45.00	1.00	92.06
14	55.00	67.50	1.00	91.87
15	59.00	67.50	1.00	93.19
16	55.00	90.00	1.50	91.41
17	59.00	67.50	1.00	93.19
18	63.00	67.50	1.00	87.76
19	63.00	45.00	0.50	86.01
20	59.00	67.50	1.50	88.39

Data Analysis

The statistical analysis of variance (ANOVA) was carried out using the software (Design-Expert 9 software (Stat-Ease Inc., USA) so as to evaluate the precision of the model, the fitness and the significance of the model, the effects of the individual parameters and interaction effects on the response. In accordance with the results obtained using the ANOVA (Table 2), the model was significant with a p-value less than 0.0001. Additionally, the Model F-value of 9.01 implies that the model is significant. Values of "Prob > F" less than 0.0500 indicate model terms are significant. In addition, reaction temperature, A, time, B, catalyst concentration, C, reaction temperature–catalyst concentration interaction, AC, and the interaction term of reaction time–catalyst concentration, BC, were significant model terms with p-values less than 0.05 (both the model and term p-value<0.05 indicated that the model is significant for 95% confidence intervals). Further, temperature-time interaction, AB, quadratic term of temperature, A^2, quadratic term of time, B^2, and quadratic term of catalyst

concentration were insignificant terms (term p-value>0.100 indicated that the model terms are insignificant). In Addition, the insignificant terms in this optimization study, were kept to improve the model precision [19-20]. The lack of fit is regarded as the weighted sum of squared deviations between the mean response at each factor level and the corresponding fitted value [19]. In this research work the lack of fit is not significant for the response with a P-value of 0.0610 (lack of fit p-value>0.05); this indicates that the model is fitted to all data (Not-significant, lack of fit is good). Adequate precision is a measure of signal to noise ratio; it compares the range of the predicted values at the design points to the average prediction error and as prerequisite of the model, a ratio of greater than 4 is desirable. In this model, the ratio of 13.058 indicates sufficient model discrimination.

Table 3: ANOVA for response surface quadratic model analysis of variance

Source	Sum of Squares	Df	Mean Square	F. Value	p-value Prob-F	
Model	2223.81	9	24.87	9.01	0.0010	significant
A-Temp.	21.21	1	21.21	7.68	0.0197	
B-Time	14.18	1	1418	5.14	0.0469	
C-Cat. Conc.	21.47	1	21.47	7.78	0.0192	
AB	7.80	1	7.80	2.82	0.1238	
AC	14.26	1	14.26	5.16	0.0464	
BC	16.54	1	16.54	5.99	0.0344	
A^2	10.27	1	10.27	3.72	0.0826	
B^2	0.89	1	0.89	0.32	0.05825	
C^2	4.04	1	4.04	1.46	0.2543	
Residual	27.61	10	2.76			
Lack of Fit	24.61	6	4.10	5.47	0.0034	Not Signif.
Pure Error	3.00	4	0.75			
Cor Total	251.42	19				

Development of Regression Model Equation

The complete design matrixes together with both the experimental as well as predicted values obtained for yield response at the design points are shown in Table 3. The runs at the center points were used to determine the experimental error. Biodiesel yield obtained ranged from 83.54 to 93.4%. A polynomial regression equation was developed by using CCD coupled with RSM to analyze the factor interactions by identifying the significant factors contributing to the regression model. Design-Expert software (version 9) fitted four models to the response: linear, two factor interaction (2FI), quadratic and cubic polynomials. According to the sequential model sum of squares, the best model was selected based on the highest order polynomial where the additional terms were significant and the model was not aliased. The quadratic model was selected as suggested by the software for biodiesel yield as shown in Table 3. The final empirical model in terms of coded factor (R1) is shown in Eq.1. Where the values of A, B and C are in terms of coded factor represent reaction temperature, reaction time and catalyst concentration, respectively.

Table 4: Sequential Model Sum of Squares [Type I]

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Mean vs Total	1.891E+005	1	1.891E+005			
Linear vs Mean	130.89	3	43.63	5.79	0.0071	
2FI vs Linear	43.41	3	14.47	2.44	0.1110	
Quadratic vs 2FI	49.51	3	16.50	5.98	0.0133	Suggested
Cubic vs Quadratic	24.61	6	4.10	5.47	0.0610	Aliased
Residual	3.00	4	0.75			
Total	1.894E+005	20	9469.16			

Final Equation in Terms of Coded Factors

$R1$ =98.44+1.31* B+1.56* C+0.62* AB+0.59* AC-0.51* BC-1.46* A^2-0.26* B^2-0.30

$\quad$ * C^2 --Eq.1

The equation in terms of coded factors can be used to make predictions about the response for given levels of each factor. By default, the high levels of the factors are coded as +1 and the low levels of the factors are coded as -1. The coded equation is useful for identifying the relative impact of the factors by comparing the factor coefficients.

Final Equation in Terms of Actual Factors

The ANOVA for the response surface quadratic model is provided in Table 4. The coefficients of the response surface model as provided by Eq.2 were also evaluated. A p-value showed that all of the linear coefficients were more highly significant than their quadratic and cross-product terms. However, in order to minimize error, all of the coefficients were considered in the design. According to the ANOVA analysis of factors, a reasonable lack of fit was noted. This indicated that the model represented the actual relationships of reaction parameters, which are well within the selected ranges (Table 4). The final estimative response model equation (based on the actual value) by which the production of biodiesel from crude palm oil was estimated was as follows:

R_1 = +12.07253* Temperature – 0.32560 * Time – 16.08280 * Cat. Conc. +0.011726 * Time + 0.67910 * Temperature * cat. Conc – 0.13664 * Time * cat. Conc – 0.11900 * $Temperature^2$ 1.13865E003 * $Time^2$ 4.77600 cat. $Conc^2$-- Eq.2

each factor. It is worthy to mention that the negative sign indicates antagonistic effect while positive sign in front of the terms indicates synergistic effect. The quality of the model developed was evaluated based on the correlation coefficient value [19]. The R^2 value was 0.9902. This signified that 99.02% of the total variation in the biodiesel yield was attributed to the experimental parameters studied. The closer the R^2 value to unity, the better the model will be as it will give predicted values which are closer to the actual values for the response. The R^2 of 0.9902 was considered relatively high, indicating that there was a good agreement between the experimental and the predicted yield of biodiesel from this model.

Influence of the Parameters on the Methyl Ester Yields

Based on the developed models, it was observed that all the three parameters (A, B, and C) played a vital role during the production of biodiesel via transesterification. Surface plots of the predicted methyl ester yields, which can be generated by either Equation (Q1) or Equation. (Q2), are illustrated in Figures 1(a) and (b), Figures 2 (a) and (b).

From the figures, it can be shown that, at low reaction temperatures, the amount of methyl ester yields increased with reaction time. However, low biodiesel yields were recorded at higher temperatures, which could be attributed to the volatility of methanol as it approaches its boiling point. It is worthy to mention that high biodiesel yields were achieved at a reaction of 78 min. Furthermore, to complete transesterification reactions, sufficient contact time must be provided so that conversion of triglycerides to biodiesel increased with an increase in reaction time [21].
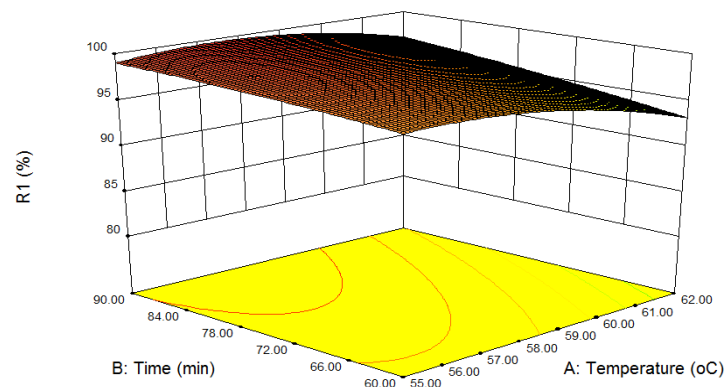
Figures 1(a): Plot of (3D) for surface response methodoly presenting the effects of time (min) and temperature (^{o}C) on the yield of biodiesel
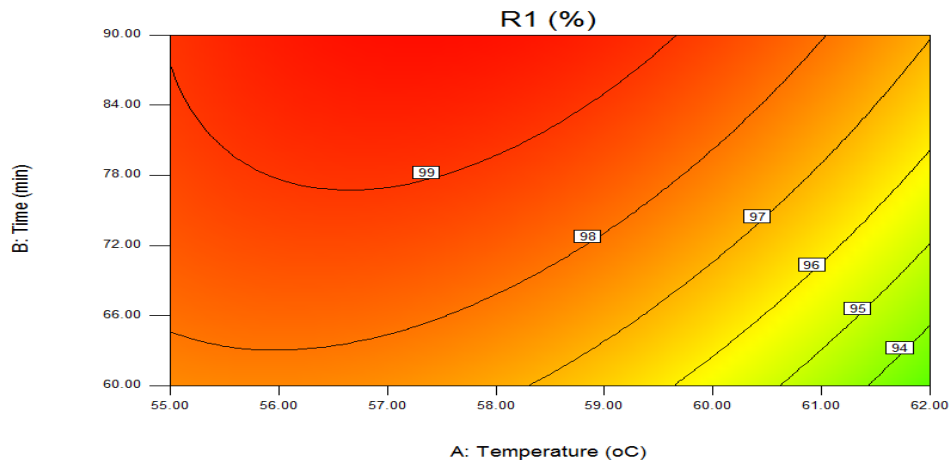
Figures 1(b): Plot of contour (2D) presenting the effects of time (min) and temperature (^{o}C) on the yield of biodiesel.

For Figures 1 (a) and (b), an increased in biodiesel yields can be observed due to the fact that the quadratic terms of the two factors are more significant with a positive effect (Eq. (Q1)). At low temperatures, there is a considerable increase in the amount of the biodiesel yields with the catalyst concentration as a result of a positive significant effect of AC on the response. Conversely, as a result of increasing catalyst concentration at higher temperatures, the amount of biodiesel yields was considerably reduced (Figures 1 (a) and (b). Additionally, temperature higher will burn the alcohol and will result in much lesser yield [21].
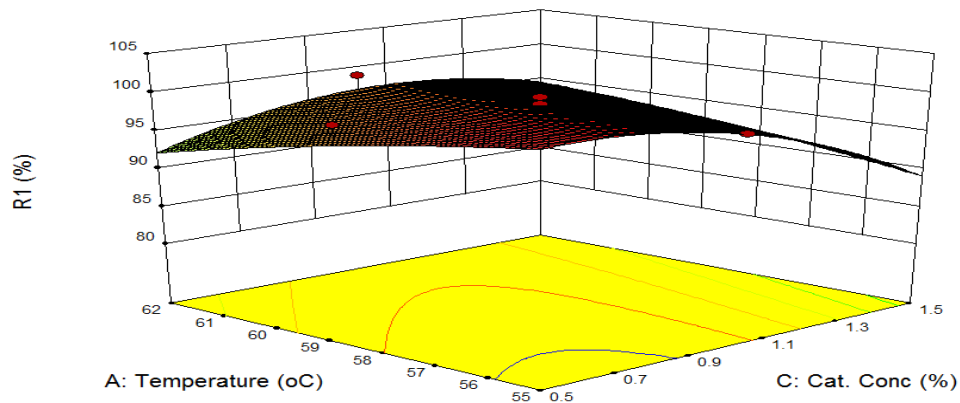

Figure 2(a): Plots (3D) for Response surface presenting the effects of temperature (^{o}C) and cat. Conc (%) on the yields of biodiesel.
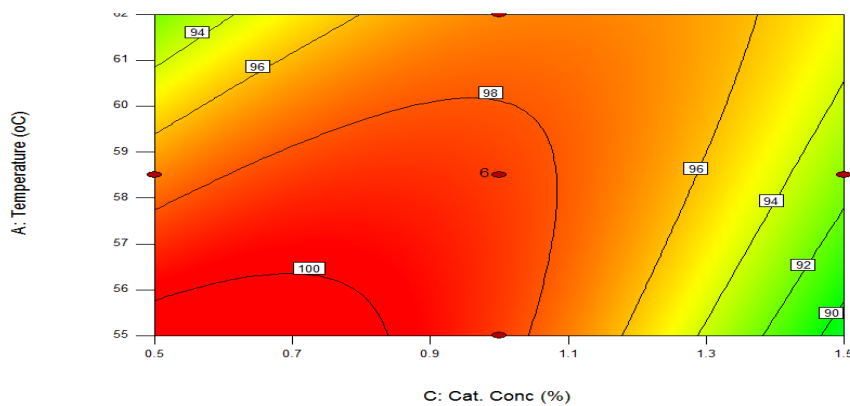

Figure 2(b): Plots (2D) for contour presenting the effects of temperature (^{o}C) and cat. Conc (%) on the yields of biodiesel.

For Figures 2 (a) and (b), it can be seen that the effect of differing catalyst concentration and time on the synthesis of biodiesel is considerable. At high catalyst concentration, synthesis of biodiesel was slightly low; this could be attributed to some catalyst be consumed via process of saponification.

Process of Optimization

There are abundant technologies obtainable for the optimization of chemical processes. Based on the predicted models, numerical hill-climbing algorithms were employed to seek for the most desirable outcome [4]. Numerical optimization provides up-to-date and a complete description of the most effective methods in continuous optimization. It responds to the growing interest in optimization in business, science, and engineering by focusing on the technologies that are most suited to practical problems [22, 23]. Thus the optimization process was performed based on the limits of parameters and response generated for the transesterification. At these optimum points, the value of biodiesel yield is 93.48, almost met the condition specified by EN 14214 (96.5%). Good agreement between the results achieved for the experimental and predicted values established the validity of the models and the existence of the optimum conditions [22]. Further the accuracy of the models can be further justified by p-values presented in the ANOVA Table 4. In addition, the results obtained demonstrated that RSM with appropriate design of experiment can be successfully applied for the optimization of the parameters in a production process. Therefore this study is centered on the application of RSM to optimize biodiesel yield via two-step alkali-catalyzed transesterification reaction. The optimization process may present valuable information pertaining to the development of efficient and economic processes for the production of biodiesel using transesterification process [23].

Table 5: Overall optimization results and model evaluation

Temp (^{o}C)	Time (Min)	Cat. Conc (%)	%R (Yield)	
			Predicted	Experimental
59	67.50	1	96.50%	93.48%

Physical Properties of Biodiesel Produced

The properties of the biodiesel produced from crude palm oil were determined at optimum conditions. The fuel properties of biodiesel are given in Table 6. Furthermore the properties of the biodiesel are discussed as follows:

Table 6: Comparison of physical properties of biodiesel produced

Properties	Test method	Unit	ASTM Standard	Biodiesel
Flash point	ASTM D93-07	°C	130 min	189
Viscosity	ASTM D445-06	mm^2/s	1.9–6.0	4.08
Density	ASTM D4052-96	kg/m^3	-	871
Cloud point	ASTM D2500	°C	Report	10.5
Pour point	ASTM D97-93	°C	-	5
Moisture content	KARL FISHER	%	<0.10	0.12
Cetane Number	ASTM D 613		47 min	68

Viscosity

Viscosity is a measure of the internal fluid friction or resistance of oil to flow, which tends to oppose any dynamic change in the fluid motion. As the temperature of oil increases, its viscosity decreases, and it is therefore able to flow more readily. It is also important for the flow of oil through pipelines, injector nozzles, and orifices. In this research work, a viscosity of 4.08 mm^2/s was recorded, this was found to be within ASTM D445-06 standard specification. The lower the viscosity of the oil, the easier it is to pump and atomize and achieve finer droplets. Viscosity is the most important property of biofuel since it affects the operation of fuel injection equipment, particularly at low temperatures when the increase in viscosity affects the fluidity of the fuel. Biodiesel has a viscosity close to that of diesel fuels. High viscosity leads to poorer atomization of the fuel spray and less accurate operation of the fuel injectors [24].

Density

Density is another important property of biofuel. Density is the mass per unit volume of any liquid at a given temperature. Specific gravity is the ratio of the density of a liquid to the density of water. Density has

importance in diesel-engine performance since fuel injection operates on a volume metering system. Also, the density of the liquid product is required for the estimation of the Cetane index [25, 26]. The density of the density was determined using a density meter at 298 K according to ASTM D4052-96 and the value obtained 871 kg/m^3.

Cetane Number

The cetane number (CN) is a measure of ignition quality or ignition delay and is related to the time required for a liquid fuel to ignite after injection into a compression ignition engine. CN is based on two compounds, namely, hexadecane, with a cetane of 100, and heptamethylnonane, with a cetane of 15. The CN scale also showed that straight-chain, saturated hydrocarbons have higher CNs than branched-chain or aromatic compounds of similar molecular weight and number of carbon atoms. The CN relates to the ignition delay time of a fuel upon injection into the combustion chamber. It is a measure of ignition quality of diesel fuels; a high CN implies short ignition delay. The longer the fatty acid carbon chains and the more saturated the molecules, the higher the CN [4, 24]. Thus the cetane number obtained in this work was 68 min (See Table 6).

Cloud Points

The CP is the temperature at which a cloud of crystals first appears in a liquid when cooled under different conditions. Thus, a CP of 10°C was recorded using the procedure provided by ASTM D2500.

Pour Point

This is the temperature at which the amount of wax from solution is sufficient to gel the fuel; thus it is the lowest temperature at which the fuel can flow [27]. The PP is the lowest temperature at which an oil specimen can still be moved. It is determined according to ASTM D97-93 and the pour point recorded was 5°C. These two properties are used to specify the cold-temperature usability of a fuel. Triglycerides have higher CP and PP compared to conventional diesel fuel [21].

Moisture Content

The water content of a fuel is required to accurately measure the net volume of actual fuel in sales, taxation, exchanges, and custody transfer. Various methods are used for the determination of water content in oil samples such as evaporation methods, distillation methods, the xylene method, Karl-Fischer titration method, *etc*. Evaporation methods rely on measuring the mass of water in a known mass of sample [28, 29]. The moisture content is determined by measuring the mass of an oil sample before and after the water is removed by evaporation. Distillation methods are based on direct measurement of the amount of water removed from an oil sample by evaporation. The Karl-Fischer titration method was used for determining the moisture content of the biodiesel produced [2]4, which was found to be 0.12%. The lower the moisture content the better fuel, and the lesser the difficulty in usage in combustion engines.

Flash Point

The flash point specification in both ASTMD6751 and EN14241 standards restricts the alcohol level. Thus, by stating a flashpoint specification of 130 ℃, the ASTM standard restricts the amount of alcohol to a very low level (<0.1%). Experimental results showed that as little as 1% methanol in the biodiesel products can significantly reduce the flashpoint from 170°C to <40 ℃. Thus careful removal of methanol via rotary evaporation could reduce residual alcohol in biodiesel to level that is too low to have negative impact on the fuel performance [21, 30]. As can be seen in **Table 9**, achievement of biodiesel with flash points above 180°C indicated that the biodiesel produced met both standards specifications. Further, biodiesel is much easier to use and store due to its relatively higher flash point compared to petro-diesel.

5. Conclusion and Recommendations

The objective of this study was to produce biodiesel from crude palm oil with high free fatty acids. The FFA level of the palm oil was reduced from 3.451% to 0.185% during the employment alkali-catalyzed transesterification reaction. Thus, the concentration of acid catalyst used had effect on the reduction of the FFA level of the crude palm oil in the pretreatment reaction. Sulfuric acid with concentration of 3% is is effective for reducing FFA level of the feedstock. More research be made on the potential of Nigerian palm oil for the production of biodiesel using different catalysts. Different feedstocks should be explored and exploited.

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