Optimization of Nigerian Restaurant Waste Cooking Biodiesel Reaction Parameters using Response Surface Methodology

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ABSTRACT

The present work deals with the production of biodiesel from Nigerian restaurant waste cooking oil (NRWCO) and the optimization of the parameters that influences the alkaline transesterification of NRWCO into biodiesel using response surface methodology. The optimization parameters like oil: oil/methanol molar ratio, catalyst amount and reaction time were done using Design Expert 6.06 software. It was found that the maximum yield of biodiesel was obtained in 79.8 min for 1: 5.9, oil: methanol ratio, 1.2 wt. % KOH amount. A total of 20 experiments using Central Composite Design were carried out. The R^2, adjusted R^2 and predicted R^2 values were 0.982, 0.9657 and 0.9088 respectively show that the experimental values are in good agreement with the predicted values. The properties of biodiesel at the optimized parameters, thus, produced confirm to the ASTM, EN and BIS specifications, making it an ideal alternative fuel for diesel engine.

Keywords: *ANOVA, Nigerian Restaurant Waste Cooking Oil, Optimization, Reaction Parameters, Response Surface Methodology*

DOI: 10.4018/ijeoe.2014100102

INTRODUCTION

The depletion of fossil fuels, increasing demands for diesel and uncertainty in their availability, depletion rate of energy source, emission of very dangerous pollutants, rapid industrialization and increasing price that make petroleum no longer economically sustainable have necessitated the search for cheap raw material cost. A sustainable and economical supply of raw material is the key factor for biodiesel to be competitive commercially (Fazal et al., 2011; Zeng et al., 2008; Su et al., 2009; Lei et al., 2010). Waste cooking oil presents a promising choice as an alternative feedstock for biodiesel production and to reduce production cost (Gnanaprakasam et al., 2013). In addition, the utilization of waste frying oils diminishes the problems of contamination, because the reusing of these waste greases can reduce the burden of the government in disposing of the waste, maintaining public sewers, and treating the oil wastewater (Encinar et al., 2007). Arora et al. (2012) reported that significant amount of organic waste from agriculture, industries, and community sources is collected annually; it can be convertible to useful energy forms like biohydrogen, biogas, bioalcohols, etc., through various Waste-To-Energy Routes (WTERs) for sustainable development. Urooj et al. (2013) remarked that usage of bio-energy is becoming more and more prominent due to the peak oil crisis.

The reaction is affected by several parameters such as the concentration of catalyst, oil to methanol ratio, reaction temperature, moisture, presence of free fatty acids (FFA) and agitation intensity (Encinar et al., 2005). This process involves many parameters that affect the reaction and optimizing so many reaction factors require large number of experiments, which is laborious, time consuming, and economically non-viable. Response surface methodology (RSM) is a useful statistical technique for the optimization of complex processes, as it reduces the number of experiments required to achieve ample data for a statistically pertinent result (Jeong & Park, 2009). Abhang and Hameeddullah (2012) hinted that optimization of process parameters is the key step in response surface methods to achieve high quality without cost inflation.

Yang et al. (2012) investigated the optimal production of biodiesel from waste deep frying oil by using different amount of catalyst and different amount of oil to methanol ratio. Seecharan et al. (2009) reported of their investigation on a laboratory scale production from used cooking oil in Trinidad and Tobago. Abdalla and Oshaik (2013) explained the concept of converting recycled oils to clean biodiesel. They further hinted that the use of waste cooking oil can go a long way in improving biodiesel economics. Conversion of waste frying oil into a valuable biodiesel after acid- pre treatments process is necessary in order to lower its FFA below 1% (Syam et al., 2013). Canakci and Van Gerpen (2001) developed a process by employing acid catalyst to pretreat the high FFA feedstocks until their FFA level was below 1%, allowing the subsequent use of alkaline catalysts to convert the triglycerides. Production of biodiesel from three mixtures of vegetable oil and used cooking oil by alkali-catalyzed transesterification was investigated and remarked that supplementary oil feedstock for biodiesel production can be recommended if engine performance tests provide satisfactory results (Nakpong & Wootthikanokkhan, 2009). Owolabi et al. (2011) reported that the properties of waste cooking biodiesel were not only comparable with that of others but also within standard limits. Utlu and Kocak (2008) concluded that frying oil methyl ester as alternative diesel engine fuel can be used successfully to operate a turbocharged direct injection diesel engine without modifications to engine or injection system. Phan and Phan (2008) reported that blend of 20 vol. % waste cooking biodiesel and 80 vol. % diesels (B20) could be applied in engines without major modification. Ketlogestswe and Gandure (2011) reported that performance of the engine when powered by biodiesel and its blends with petroleum diesel is very comparable to its performance when powered by 100% petroleum diesel.

Widayat et al. (2013) employed biodiesel production from bulk frying oil with ultrasound assisted, while Samuel et al. (2013) concluded that feasibility of domestic production of restaurant waste cooking oil biodiesel using blender modified reactor is realistic.

RSM is one of the well-known designs of experiment technique for predicting and optimizing the system parameters with minimum number of experiments. It is used for modeling nonlinear relations between the input factors and the responses (outputs). When it is compared with Taguchi method and factorial design, RSM has the advantage that it can be used for optimizing nonlinear systems, which can be modeled by second order full quadratic models and can give optimal solutions with decimals of factor levels while Taguchi gives the optimal combination of factors for the given factor levels and factorial design is appropriate for systems those can be modeled by first order polynomials. The first advantage for using RSM in the present study is that the RSM provides the mathematical relation that also includes the interactions between the factors, which is difficult to obtain using heuristic optimization techniques. Detection of the interactions between various factors is of critical importance especially for multivariate optimization. The second advantage of RSM is its ability to reduce the number of experiments for optimizing processes. Some other mathematical methods may calculate accurate results as done in the case study presented in this paper (for example, direct search, etc) but all other methods except RSM (and other DOE techniques) require more experimental results for accurate mathematical modeling when compared with RSM (Ileri et al., 2013).

Literature has revealed that limited papers are available on the use of response surface methodology (RSM) for the optimization of process variables to maximize the biodiesel yield. A number of researchers have identified the important variables that affect the transesterification reaction, particularly, the reaction temperature, the type and amount of catalyst, the ratio of alcohol to vegetable oil, the mixing intensity and the reaction time, etc. (Meher et al., 2006; Peterson et al., 1992; Encinar et al., 2005). Boonmee et al. (2010) have investigated the effect of three process variables viz. methanol/oil molar ratio, catalyst concentration and reaction time on the methyl esters yield of Jatropha curcas. Central composite design (CCD) of 20 experiments was employed and 99.87% biodiesel yield was achieved. Similarly, Tiwari et al. (2007) have optimized methanol quantity and reaction time using RSM based on central composite rotatable design (CCRD) of 20 experiments and obtained biodiesel yield of 99%. In view of the above, few works are reported on the optimization of biodiesel production from Nigerian restaurant waste cooking oil using three process variables. The present paper, therefore, reports the results of the optimization of three process variables viz. catalyst (KOH) amount (0.5-1.5% w/w), reaction time (40-80 min) and methanol/oil ratio (M) (4:1–8:1) for the maximization of restaurant waste cooking biodiesel yield. CCD of 20 experiments based on RSM with the help of Design Expert 6.0.6 software has been used. A model to predict the response (WCO yield (%)) has been formulated and validated by Analysis of Variance (ANOVA). In this paper, the statistical analysis of product yield was performed using Design Expert 6.0.6. Central Composite Design (CCD) was applied to optimize the catalyzed transesterification reaction variables. Optimization of parameters for biodiesel production from RWCO oil such as oil: alcohol ratio, catalyst amount and reaction time were carried out. Regression analysis and analysis of variable were also made to test the significance of model.

MATERIALS AND METHODS

Materials

Waste cooking oil (WCO) was donated by Mr. Biggs's (UAC Restaurants Limited), Sango, Ogun State, Nigeria. Methanol (99.8%) used is analytical grade product of Aldrich chemicals, Germany; while the KOH used was also an analytical grade product of Aldrich Chemicals, England and acetic acid were obtained from Uche Scientific Company Limited, Lagos. Other major materials used include thermometer, separating funnels and magnetic stirrer.

Experimental Method

WCO was filtered to remove all the insoluble impurities followed by heating at 100°C for 10 min to remove all the moisture. WCO had low FFA (0.9%), which is in agreement with the limit suitable for base catalyzed transesterification reaction. Preheated Waste cooking oil was subjected to base catalyzed transesterification for biodiesel production at specified reaction conditions according to the Central Composite Response Surface Design (CCRD) using conical flask equipped with a condenser, thermometer, chiller, heating plate and stirrer. The pre-heated oil was trans-esterified with methanol and potassium hydroxide to yield waste cooking biodiesel and glycerol. The resulting product was poured into a separating funnel mounted on a clamp stand and was allowed to settle down overnight. It was observed that the resulting mixture from the reaction had settled into yellow biodiesel on the top with the black glycerol at the bottom of the separating funnel. The ester was washed with water three times. Small amount of acetic acid (2.5ml/l of the oil) was used in the first washing. At the end of the process, the ester was heated to 90°C at vacuum to remove any water left from the oil. The final ester became golden yellow. The procedure was replicated three times and average biodiesel yield.

Design of Experiment

The alkaline transesterification of waste cooking oil and optimization of the methyl ester was studied using Design Expert 6.0.6 (Stat-Ease Inc., USA). Response Surface Methodology (RSM) and Central Composite Design (CCD) were used to find the interactions between the three variables and to predict the optimum condition for biodiesel yield. Selection of these parameters was based on the literature studies reported on the alkaline solutions. These parameters were catalyst (KOH) amount, methanol to oil molar ratio and time. Methyl ester yield was used as the response. The reported data in this work were average of triplicate measurements to ensure accuracy. Table 1 shows the levels of the variables used and the range. Selection of the levels was based on the preliminary study and literature research. The reaction time, methanol to oil molar ratio catalyst (KOH) amount, were varied from 40 to 80 minutes, 4 to 8 mol/ mol and 0.5 to 1.5 wt. %, respectively. The reactant mixture temperature was held constant below the boiling point of methanol (65°C) in order to prevent mass transfer limitation and bubble formation of methanol. A three-level-three-factors CCD (20 experiments) was adopted, consisting of 8 factorial points, 6 axial points and 6 replicates at the center points design. The yield of methyl ester was analyzed and optimized using Equation (1) as follows:

$$y = \beta_o + \sum_{i=1}^{3} \beta_i x_i + \sum_{i=1}^{3} \beta_{ii} Xi^2 + \sum_{j=1}^{3} \sum_{j=1}^{3} \beta_{ij} X_i X_j \quad (1)$$

where β_o, β_i and β_{ii} are regression coefficients for intercept, linear, quadratic and interaction coefficients, respectively are x_i and x_{ij} are coded independent variables.

RESULTS AND DISCUSSION

Development of Regression Model

In this study, the correlation between response (yield) and three parameters, reaction time, methanol to oil molar ratio and amount of catalyst, KOH, were analyzed using alkaline transesterification (See Table 2). The Response Surface Methodology was conducted to find the relationship between the time, methanol to oil molar ratio and catalyst dosage. This investigation was employed to obtain high yield of biodiesel without adding co-solvent. A model for the experimental design was fit-

Table 1. Levels for biodiesel production from waste cooking oil

Variables	Coding	Unit	Lower Level	High Level
Time	A	min	40 (-1)	80 (+1)
Methanol to oil ratio	B	M	4 (-1)	8 (+1)
Catalyst amount	C	wt. %	0.5 (-1)	1.5 (+1)

Table 2. Optimization of base transesterification of restaurant waste cooking using CCD

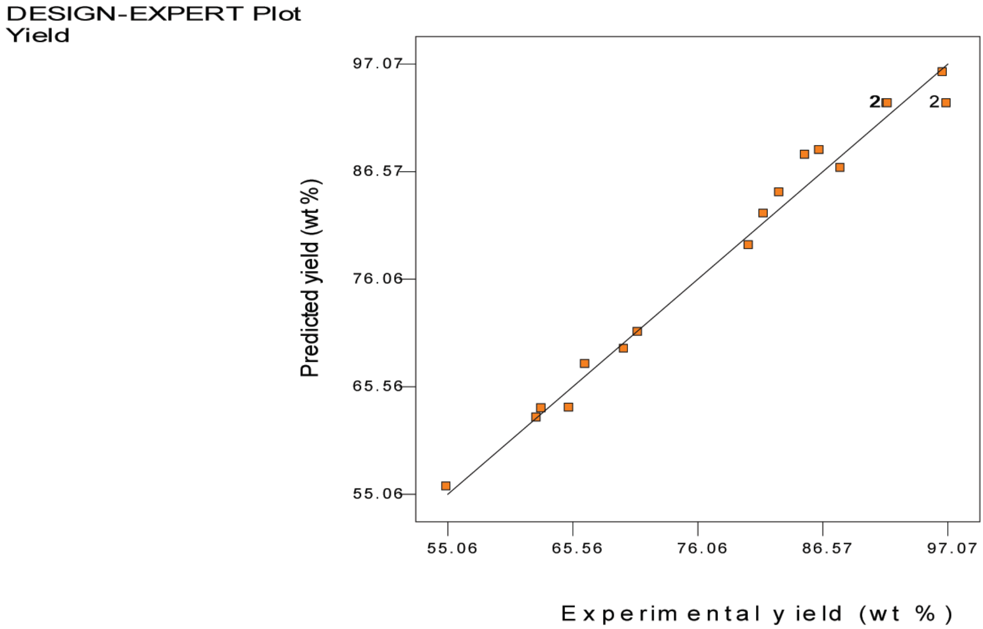
Levels of Variables (Actual)						
Standard	Time, A (Min)	Methanol-Oil Molar Ratio, B (M)	Catalyst Amount, C (wt. %)	Experimental Yield (%)	Predicted Yield (%)	Residual
1	40	4	0.5	55.06	55.76	-0.70
2	80	4	0.5	62.63	62.48	0.15
3	40	8	0.5	65.36	63.46	1.90
4	80	8	0.5	63.04	63.38	-0.34
5	40	4	1.5	69.97	69.21	0.76
6	80	4	1.5	83.01	84.48	-1.47
7	40	8	1.5	71.13	70.85	0.28
8	80	8	1.5	80.45	79.32	1.13
9	40	6	1	86.38	88.62	-2.24
10	80	6	1	96.75	96.21	0.54
11	60	4	1	88.15	86.88	1.27
12	60	8	1	85.18	88.15	-2.97
13	60	6	0.5	66.71	67.71	-1.00
14	60	6	1.5	81.71	82.41	-0.70
15	60	6	1	97.07	93.18	3.89
16	60	6	1	92.04	93.18	-1.12
17	60	6	1	92.04	93.18	-1.14
18	60	6	1	97.07	93.18	3.89
19	60	6	1	92.13	93.18	-1.05
20	60	6	1	92.13	93.18	-1.05

ted using Design Expert 6.0.6 (Stat-Ease Inc., USA). Final equation in terms of (coded factors) experimental data was:

$$Yield = 93.18 + 3.80A + 0.63 B + 7.35C - 0.77A^2 - 5.67B^2 - 18.13 C^2 - 1.70A B + 2.14AC - 1.51BC$$

where A, B and C denote time (min), methanol to oil molar ratio (M) and catalyst (KOH) amount (wt. %) respectively, and Zabeti et al. (2010) reported that positive sign in front of each term denotes synergetic effect and negative sign shows the antagonic effect. Figure 1 shows the predicted value and experimental value using the

Figure 1. A parity plot between actual experimental values and predicted values of waste cooking biodiesel yield



model equations in Equation (1) and the graph shows that the predicted values are quite close to the experimental value and confirmed by the R^2 value, 0.982. Analysis of variance (ANOVA) was employed to investigate the significance and fitness of the model. ANOVA also represents the interaction of variables on the respond and the effect of individual parameters. Table 3 indicates that the model was very significance because the p value < 0.0001. There was no lack of fit since the value was not significant. Lack of fit shows that model was well fitted to the data. Adequate precision measures the signal to noise ratio. This model can be utilized to navigate the design region as ratio greater than 4. Therefore is desirable and in this case, the ratio was 23.24.

Table 3 illustrates the time (A), catalyst amount (C), time- catalyst amount (AC) and quadratic term methanol to oil ratio (B^2) and catalyst amount (C^2) were significant as the p- value < 0.05. There exist two insignificant values which were higher than p- value > 0.005, linear methanol to oil ratio (B) and quadratic term time (A^2). This indicates that the model could be utilized to predict biodiesel yield. Figure 1 again shows that the regression model equation provided a very accurate description of the experimental data, indicating that it was successful in capturing the correlation between the process parameters to the yield of waste cooking biodiesel. This is further supported by the correlation coefficient, R^2 which was found to be very close to unity, 0.982.

Interaction between Parameters

Three-dimensional surface plots of the predicted yield of the biodiesel production are given in Figures 2 – 4. Each surface was generated by keeping one factor constant at the central point of CCD and varying the other two within their experimental ranges. The figures show the yield of biodiesel increases with the increase of time, oil to methanol molar ratio and catalyst loading.

Figure 2 shows the effect of interaction of methanol- to- oil molar ratio and reaction time on conversion of waste cooking oil to biodiesel

Table 3. ANOVA table for response surface quadratic model

Source	Prob. > F	Remark
Model	< 0.0001	Significant
A	0.0006	Significant
B	0.4343	Not significant
C	< 0.0001	Significant
A^2	0.6156	Not significant
B^2	0.0034	Significant
C^2	0.0034	Significant
AB	< 0.0791	Not significant
AC	< 0.00338	Significant
BC	0.1126	Not significant
Lack of fit	0.5796	Not significant
Pure error	6.63	Not significant
R^2	0.982	
Adj R^2	0.9657	
Pred R^2		

Figure 2. Response surface plot of the interaction effect of methanol-to-oil molar ratio and reaction time on WCO biodiesel yield when the amount of catalyst is 1 percent

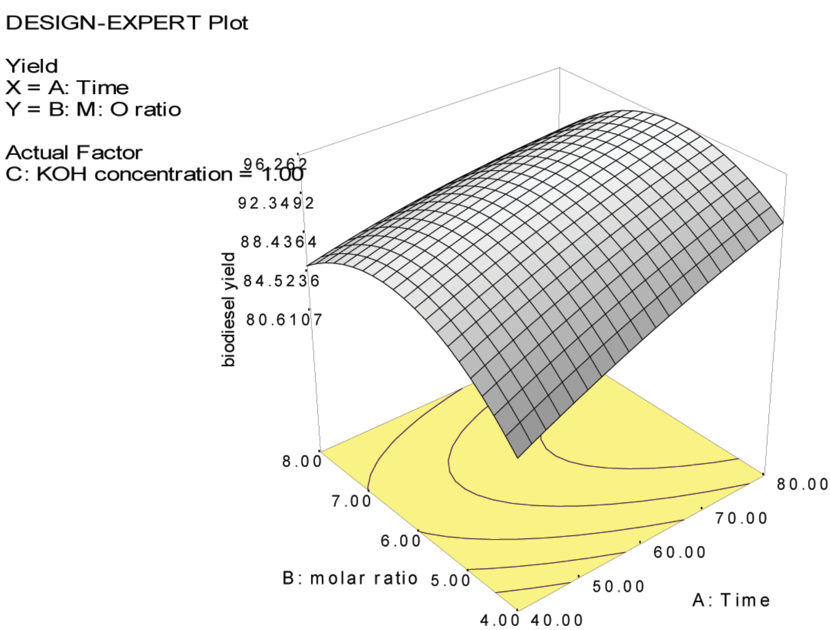


Figure 3. Response surface plot of the interaction effect of catalyst amount and reaction time on WCO biodiesel yield when the molar ratio of methanol to oil is 6:1

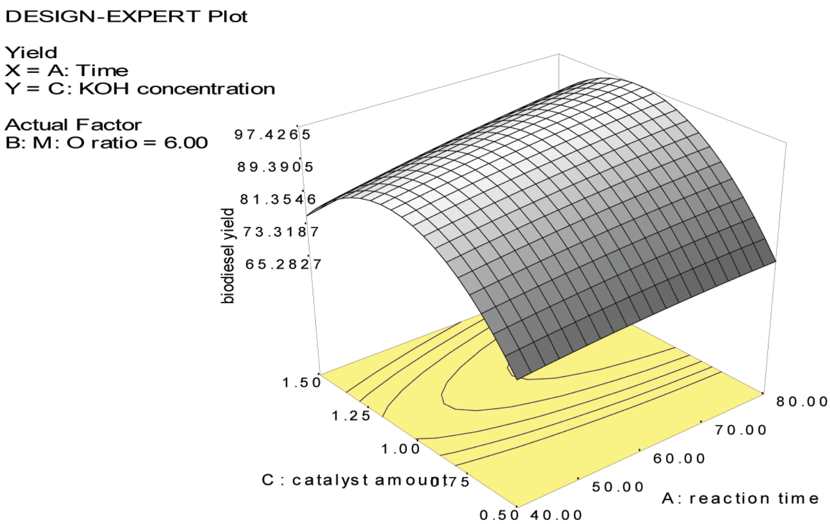
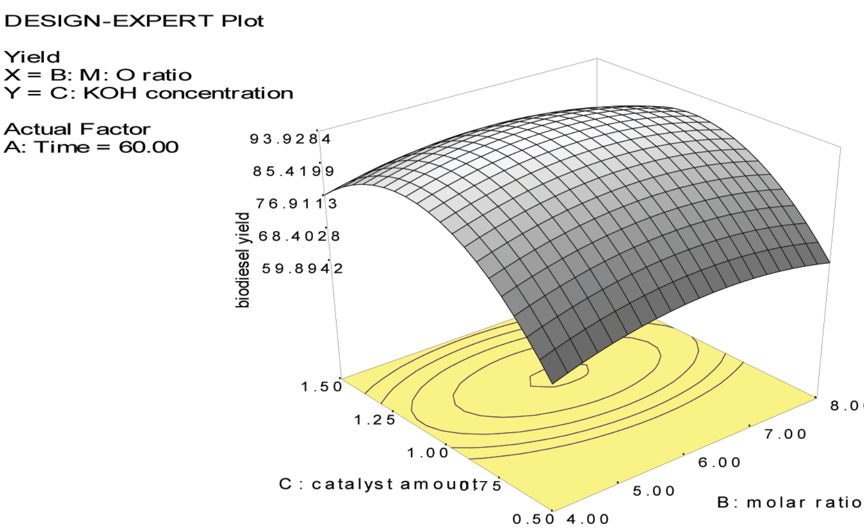


Figure 4. Response surface plot of the interaction effect of methanol-to-oil molar ratio and catalyst amount on WCO biodiesel yield when the reaction time is 60 minutes



at a constant catalyst amount (1.0 wt. %). There was a high significant effect of reaction time on KOH- catalyzed transesterification under such condition, methanol – to – oil molar ratio of about 6:1 gave the best conversion to biodiesel. In addition, there was a negative significant interaction between methanol- to - oil molar ratio and reaction time. This indicates that waste cooking biodiesel increased as methanol/ oil molar ratio increased from 4 mol/mol to 6mol/ mol. Above 6 molar ratio, yield of biodiesel slightly decreased.

As shown in Table 3 among all the interaction terms, only the term AC is significant at

95% confidence level. This implies that only the interaction between reaction time (A) and catalyst amount (C) affect the yield of waste cooking oil biodiesel yield significantly. The developed model was then employed to construct a response surface plot to facilitate a straightforward study on the two dimensional plots for the interaction between time and catalyst amount respectively at a fixed 6:1 methanol to oil molar ratio. It can be seen that when the transesterification of waste cooking oil was carried out at shorter reaction time (40min), an increase of catalyst amount from 0.5 wt. % to 1.5 wt. % cause a significant in the yield of waste cooking oil biodiesel. However, at longer reaction time (80min), an increase of catalyst amount from 0.5 wt. % to 1.5 wt. % only cause slight increase in the yield of waste cooking oil biodiesel. Therefore at longer reaction time, an increase in amount of catalyst does not affect the yield as significant as at lower reaction time (See Figure 3).

The effect of methanol- to oil molar ratio and catalyst amount on biodiesel at a constant reaction time of 60 min is shown in Figure 4. At lower methanol-to- oil molar ratio, the conversion to biodiesel increased with increase in catalyst amount; at higher methanol – to- oil molar ratio, increment of catalyst amount does not necessarily increase waste cooking oil biodiesel yield. However, excess catalyst produce emulsions and the biodiesel that is produced have difficulty in the separation phase.

The optimal condition to achieve a higher yield was at 1.2 wt. % of catalyst, 5.9 of methanol to oil molar ratio and time of 79.8 min. No soap formation was observed when the catalyst amount ranges between 0.5 and 1.5 wt. %. The neutralization of KOH catalyst, where the biodiesel was washed with warm water (45–50^{o}C) to purify and remove impurities from the oil resulted in the highest yield of 97.7%.

Model Evaluation of Base Transesterification

The optimum condition of these three parameters was obtained by optimization of the equation (Equation (2)) using Design Expert 6.0.6. Table 4 shows the summary of optimization conditions with high and low limit to optimize based on the previous model. In this case, reaction time, methanol/ oil molar ratio and catalyst amount were in the range. The optimal condition for this study was 1.2 wt. % of KOH catalyst, 5.9 methanol/ oil molar ratio and time of 79.8 min (Table 5).

Table 4. Summary of optimization conditions

Parameter	Objective of Optimization	Lower Limit	Upper Limit
Reaction time	Is in range	40	80
M: O ratio	Is in range	4	8
KOH amount	Is in range	0.5	1.5
Yield	To maximize	55.06	97.07

Table 5. Results of modeling/optimization of biodiesel yield

Experiment	Time, A (Min)	Methanol/ Oil ratio, B (mol)	Catalyst Amount, C (wt. %)	Yield % (Predicted)	Yield % (Experimental)
1	79.8	5.9	1.2	97.3	97.7

To evaluate the accuracy of the developed model, base catalyzed transesterification was conducted according to the optimum condition as in Table 5. Table 5 shows the results obtained and the yield was 97. 7% if compared to the predicted yield, 97.3%. Both of the values are close with smaller error of 0.41% and this shows that this model was in agreement with the actual value.

Validation of the Final Model

Through the *t*-test, no significant differences are observed between the experimental and predicted values ($P \geq 0.05$). Moreover, the closeness of actual and predicted values in Figure 1 shows that the regression equation is adequate (Mirhosseini et al., 2008).

Biodiesel Properties

Table 6 shows the summary of the properties of the biodiesel of Nigerian restaurant waste cooking oil (NRWCO) The properties of NRWCO biodiesel were within the mentioned biodiesel standards, ASTM D6751, EN 14214 and IS 15607. The result of few standard fuel test such as specific gravity, viscosity, flash point and acid value of the NRWCO biodiesel are within biodiesel specification.

CONCLUSION

Central Composite Design was used for optimization of methanol/oil ratio, catalyst amount, and reaction time on the transesterification of Nigerian restaurant waste cooking oil. Thus, research work gave an optimal value of 97.3% biodiesel, in 79.8 minutes of reaction time with 1.2 wt. % of catalyst loading and methanol/ oil ratio of 1:5.9. Quadratic polynomial models were obtained to predict yield of biodiesel. On the basis of ANOVA; reaction time, catalyst amount, quadratic term of methanol/ oil ratio, quadratic term catalyst amount, reaction time – catalyst amount had a significant effect on NRWCO biodiesel yield. The result showed that the predicted value was in agreement with the experimental value, which was established with additional experiments to confirm the optimized parameters. The prepared NRWCO biodiesel gave promising results as alternative diesel fuel with fuel properties in good agreement within limit set by the international biodiesel standard .Therefore production of biodiesel from Nigerian restaurant waste cooking oil is a feasible process.

ACNOWLEDGMENT

The authors are grateful to Pastor Akinola Omotayo, Biotechnology Central Laboratory, Federal University of Agriculture, Abeokuta, Nigeria for his assistance during the research work.

Table 6. Properties of NRWC biodiesel

Fuel Characteristics	Unit	Waste Cooking Oil Methyl Ester	ASTM Standard D6751	EU Standard EN 14214	IS 15607
Specific gravity@ 15°C	g/cm³	0.879	0.880	0.86-0.90	0.860 – 0.900
Viscosity @ 40	mm²/s	4.72	1.9-6.0	3.5- 5.0	2.5- 6.0
Flash point	° c	139	>120	> 120	-
Acid value	Mg KOH/g	0.459	<0.8	<0.50	-

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