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Effect of Varying Temperatures, Mixing Speeds and Time of Reaction With Catalyst on Biodiesel Production From Waste Palm Oil

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Abstract

Waste palm oil (WPO) has proved to be a source of feedstock for producing biodiesel using the transesterification method. In this work, WPO collected from restaurants were converted to biodiesel using transesterification method. The effects of temperatures varying from 45 to 65^{O}C, mixing speeds varying from 600 to 1000rpm and time of reaction varying from 45 to 120 minutes with catalyst concentration of 1.6% weight of the WPO sample and methanol to oil ratio of 6:1 were studied to determine waste palm oil biodiesel (WPB100) yield. The experiment was designed using Central Composite Design of Design Expect software (6.0.6 version) and analysed using response surface methodology (RSM) showed optimal biodiesel yield of 94% at the combinations of 59.8^{0}C reaction temperature, 103.15minutes time of reaction and 874.91rpm mixing speed. The quadratic model developed with the software indicated R-square value of 0.9690 which implies strong correlation between the experimental and predicted yields. Also, amongst the varied variables, temperature of operation had the highest significant impact on yield whereas the interaction between temperature of reaction and mixing speed had least impact. The cetane number (CN), calorific value (CV), density, kinematic viscosity (KV), and moisture content (M.C) of the biodiesel produced were 57, 40.56MJ/kg, 0.88g/mL, 5.2mm^{2}/sec and 0.03% respectively which are within American Standard for Testing Material (ASTM) for biodiesel. The work concluded that reaction temperature and time of operation are more impactful on biodiesel yield than mixing speed. The quadratic model fitted by Design Expect statistical software successfully predicted the expected yield.

Keywords: Waste palm oil, biodiesel production, transesterification, reaction time, response surface method.

INTRODUCTION

Biodiesel is a mono alkyl ester of long chain fatty acids derived from vegetable oil (Samuel et al. 2013). It is produced through chemical reaction known as transesterification. Transesterification occurs when triglyceride which is the main composition of vegetable oil reacts with alcohol in the presence of a base catalyst and heat to produce two phase liquids biodiesel fuel and glycerol (Gnanaprakasam et al. 2013). Aside the ratios and concentrations of reagents used for transesterification reaction, other variables that determine biodiesel production yield are temperature of reaction, mixing speed and time or duration of reaction. Each of these non-reagents variables play significant roles in influencing rate of reaction, yield of products and complete reaction. Stirring (mixing) provides the agitation that increases the collision between the particles and diffusion of one reactant into another. It facilitates reaction, increases yield as well as shortens the reaction time (Canakci and Van Gerpen, 2003; Adeyemi et al. 2011; Gnanaprakasam et al. 2013). More so, early formation of the two-phase liquids of biodiesel and glycerol that does not allow for complete reaction are prevented by mixing (Saroj and Singh 2011). However, Gnanaprakasam et al. (2013) reported that beyond certain speed of stirrer, there would not be significant rise in the yield is influenced by the reaction temperature (Jing, 2011; Saroj and Singh 2011). However, Gnanaprakasam et al. (2013) opined that the heating operation should not exceed the boiling point of alcohol to avoid vaporization of alcohol which could reduce the quantity of methanol involved in the production process. Consequently, most transesterification reactions are conducted close to the boiling point of methanol (60 – 70^{0}C) at atmospheric pressure. Meanwhile, Kapilakarn and Peugtong, (2007) submitted that if the reaction temperature is maintained below 50^{0}C, the viscosity of biodiesel produced increases and separation of biodiesel from glycerol will be difficult with negative consequences on the quality of biodiesel. Temperature above 70^{0}C might cause loss of methanol as well as incomplete reaction. Also, the time or duration of transesterification reaction influences production yield (Akpan et al. 2006, Refaat et al. 2008, Said et al. 2015). One hour duration is mostly reported for transesterification reaction (Kinast, 2003; Akpan et al. 2006; Kapilakarn and Peugtong, 2007; István and Ioan-Adrian, 2011; Samuel et al. 2013 and Alemayehu and Abile, 2014). Saroj and Singh (2011) and Kinast (2003) argued that although longer reaction time produces more yield, at very high temperatures the yield is not favoured.

Fossil fuel in Nigeria is projected to go into extinction in about thirty years' time. This raises great concern because sales made from crude oil export is the mainstay of her economy. However, crude oil exploration in the country goes unabated despite the adverse effects of gas flaring to the ecosystem as well as extensive soil degradation due to oil spillage arising from pipe line vandalism which causes the nation loss of about ₦ 5.2 trillion annually (Chukwuezie et al. 2016). Moreso, the country's oil industries is bedevilled by corruption such as money embezzlement and misappropriation of oil revenue which has left over 62% of the country's 180 million populations impoverished. The situation is even more worrisome by the continuous fluctuation of the price of crude oil in international market such that the nation economic projection is unpredicted. It is on record that her gross domestic product (GDP) ng nosedived from about -0.36% in first quarter (Q1) of 2016 to -2.92% by Q1 of 2017 as a result of fall in oil price in international market since 2013. As a result, she has been borrowing to fund her budget since 2016 till date. Although the economy is out of recession according to Nigeria Economic Statistics of 2017; the programmes of economic recovery put in place by the government has not ameliorated the hardship on the populace. There is therefore no doubt that use of renewable energy like biodiesel is one of the panacea to the imminent energy and economics crises in Nigeria. Use of waste vegetable oil remains the best raw material for biodiesel production in the country because of its ease of generation and the problems its indiscriminate disposal cause to the ecosystem. For example in this study a total of twelve litres of waste vegetable oil was collated from three major restaurants in the state municipal council. It implied that an average of four litres was generation per restaurant in a week. By implication, Imo state municipal council which houses more than a hundred restaurants and hostel outlets can generate about 400litres of waste palm oil per week and about 1200litres in a month which is a great prospect for biodiesel production. The use of such a large amount of waste oil for biodiesel production drastically reduce environmental pollution as well as overdependence on fossil fuel. This study is aimed at biodiesel production from waste palm oil to reduce the extent of waste palm oil disposed indiscriminately and reliance on fossil fuel. The specific objectives are to establish the optimal process conditions of reaction temperature, mixing speed and reaction time for maximal biodiesel production from waste palm oil. Also to generate a prediction model using the process parameters for biodiesel production from waste palm oil.

MATERIALS AND METHODS

Collection of Waste Palm Oil

Waste palm oil (WPO) was collected and collated at the close of work from three restaurants; Levi, Rennys and Sunnis all situated at Owerri, capital of Imo State, Nigeria. A total of twelve (12) litres were collated all together in a week.

Pre-treatment of WPO Sample

The 12 litres of WPO was preheated at 65^0C on hot plate for 30 minutes and filtered with 25 micron and 10 micron filters respectively. The process of filtration removed large debris from the WPO. The filtrate was dried in oven (DHG 9023A) for two hours at temperature of 110^0C to 0.05% moisture content.

Also, waste vegetable oil contains high FFA (Bello et al. 2016). The method used by Ayodele et al. (2017) was adopted to remove FFA from the WPO. To every 100g of waste palm oil, 10 mL of 0.125 M NaOH solution was added and stirred continuously at a temperature of 40°C for 15 minutes to allow the FFA in oil to react with NaOH. Using a separating funnel after a period of thirty minutes two distinct layers was formed: A top layer of less viscous waste palm oil lean of FFA and a bottom layer of soap emulsion. The WPO was separated from the soap emulsion.

Characterization of Waste Palm Oil

The density was determined using density bottle, moisture content (mc.%) was obtained by the oven dry method, the kinematic viscosity (kV) was determined using a viscometer, Iodine value (IV), the acid value (AV), saponification value (SV), were obtained by titrimetry. Free fatty acid (FFA) was obtained using the relationship between acid value and FFA as shown in Equation 1. Cetane number (CN) was obtained using the expression in Equation 2, while the calorific values were obtained using the expression in Equation 3.

$$\text{Acid number } AV \times 0.5 = FFA \quad \text{(Ibeto et al., 2011) (1)}$$

$$CN = 46.3 + \frac{5458}{SV} = 0.225 \quad IV \qquad (2)$$

$$HHV = 49.43 - 0.041(SV) - 0.015(IV) \qquad (3)$$

Where :

SV = Saponification value (mgKOH/g)
IV = Iodine value (gI/100g)
HHV = high heating value (JM/kg)
CN = Cetane number
FFA = free fatty acid (%)
Av = Acid number (mgNaOH/mg)

Experimental Design

Transesterification Reaction for Biodiesel Production

For transesterification, 100g of WPO samples were used. The WPO was fed into magnetic stirrer and heated for 10 minutes until uniform temperatures used in the optimization as stated in Table 1 were maintained. Catalyst concentration of 1.6% weight of WPO sample and methanol to oil ratio of 6:1 was used for the test as shown in Table 1. The selection of the catalyst concentration and methanol to oil ratio was based on results of earlier experiments and reported works by Lotero et al. (2005), Wang et al. (2007) Hossain et al. (2010), Hossain et al. (2010), and Chen et al. (2011) Gan et al. (2012), Gnanaprakasam et al. (2013), Samuel et al. (2013 on biodiesel production using waste cooking oil as feedstock. NaOH was used because it was readily available. After each reaction, reactor content was poured in a separating funnel and left for 24hour to allow the biodiesel to separate from glycerol. The denser glycerol settles at the bottom while the biodiesel at the top. The glycerol is discarded and the biodiesel collected. Design Expect 6.0.6 version (Stat-Ease Inc., USA) software was used to design the experiment while optimization of biodiesel yield was studied using Central Composite Design (CCD) in Response Surface Methodology (RSM).

Table 1: Transesterification Process parameters

Conditions of test	Values used for each experiment
Waste palm oil	100g
Methanol ratio	6:1
NaOH	1.6 % wt of WPO
Temperatures of reaction	varied per test from 45^0C to 65^0C at 5^0C step intervals
Mixing speed	varied per test from 600rpm to 1000rpm at 100rpm step intervals
Time of reaction	varied per test from 45minutes to 125 minutes at 20minutes step intervals

The three factors namely time (minutes) of reaction, temperatures (^{0}C) of operation, and mixing speed (rpm) (Table 1) are the independent variables while biodiesel yield is the dependent variable or the response. The three-factors-five-levels factorial experiment designed in CCD consists of 20 experimental runs (six centre and axial points, and eight factorial) which provided sufficient information to allow a second-order polynomial model. The design summary for the optimisation experiment is as shown in Table 2.

Table 2: Design summary of CCD using Design Expert 6.0.6

Study Type:	**Response Surface**			**Experiments: 20**				
Design:	**Central Composite**			Blocks: No Blocks				
Design Model:	**Quadratic**							
				Codes and factor levels				
Factors	Names	Units	Type	-2	1-	0	-1	-2
A	Temp.	^{0}C	Numeric	45	50	55	60	65
B	Time	mins	Numeric	45	65	85	105	125
C	Mixing speed	Rpm	Numeric	600	700	800	900	1000

In Table 2 Factors A, B and C represent temperature of reaction, time of reaction and mixing speed, whereas the five levels of each factor from the lowest to the highest are coded as -2, -1, 0, +1 and +2 respectively. These codes were used in software in the arrangements of the treatment combinations

Washing of the Biodiesel

The biodiesel produced from each test was washed five times with warm water of 50^{0}C in the ratio of 1:2 litres of biodiesel to water until the water drained from biodiesel using the separating funnel was clean. Unwashed biodiesel does not meet ASTM standards (2011).

Drying of Biodiesel

The washed biodiesel was separated in a separating funnel and subsequently dried at 65^{0}C for 2 hours to remove traces of water. The yield of biodiesel was estimated using the equation shown in Equation 4.

$$\text{Biodiesel yield (\%)} = \left(\frac{\text{Amount of biodieselproduced}}{\text{Amount of oil used}}\right) \times 100 \quad \text{(Sulaiman, et al. 2013)} \qquad (4)$$

RESULTS AND DISCUSSION

Table 3 presents the results obtained during waste palm oil and waste palm oil biodiesel characterisation.

Table 3: The properties of the waste palm oil and the biodiesel

	WPO	WPB100	ASTM
MC(% vol)	0.25	0.03	0.05 maximum
AV (mgKOH/g)	2.98	0.28	0.8 maximum
FFA	1.49	0.14	-
KV @40 0C (mm2/sec)	42	5.2	1.6 to 6.0
FP (0C)	204	118	93 minimum
Density (g/mL)	0.92	0.88	0.86 minimum
IV (gl/100g)	142	85	-
SV (mgKOH/g)	188.97	182	-
Calorific value (MJ/kg)	38	40.56	37 minimum
Cetane number	43	57	44 to 60

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The properties of WPB100 in Table 3 are within the ASTM (2010) stipulated standard of biodiesel. Therefore the fuel can run smoothly in internal combustion engine without modification on the engine components (Chukwuezie, et al. 2014). The moisture content of 0.25% from WPO (Table 3) is lower than moisture content of 0.63% and 0.67% from waste groundnut oil and waste cotton seed oil reported in Ayodele et al. (2017). The physiochemical properties of WPO and WPB100 plotted in a bar chart (Figure 1) show that properties such as the KV, IV, SV, FP, MC, and the AV of WPO are higher than those of WPB100, whereas the CV and CN of the WPB100 are higher than those of WPO respectively.

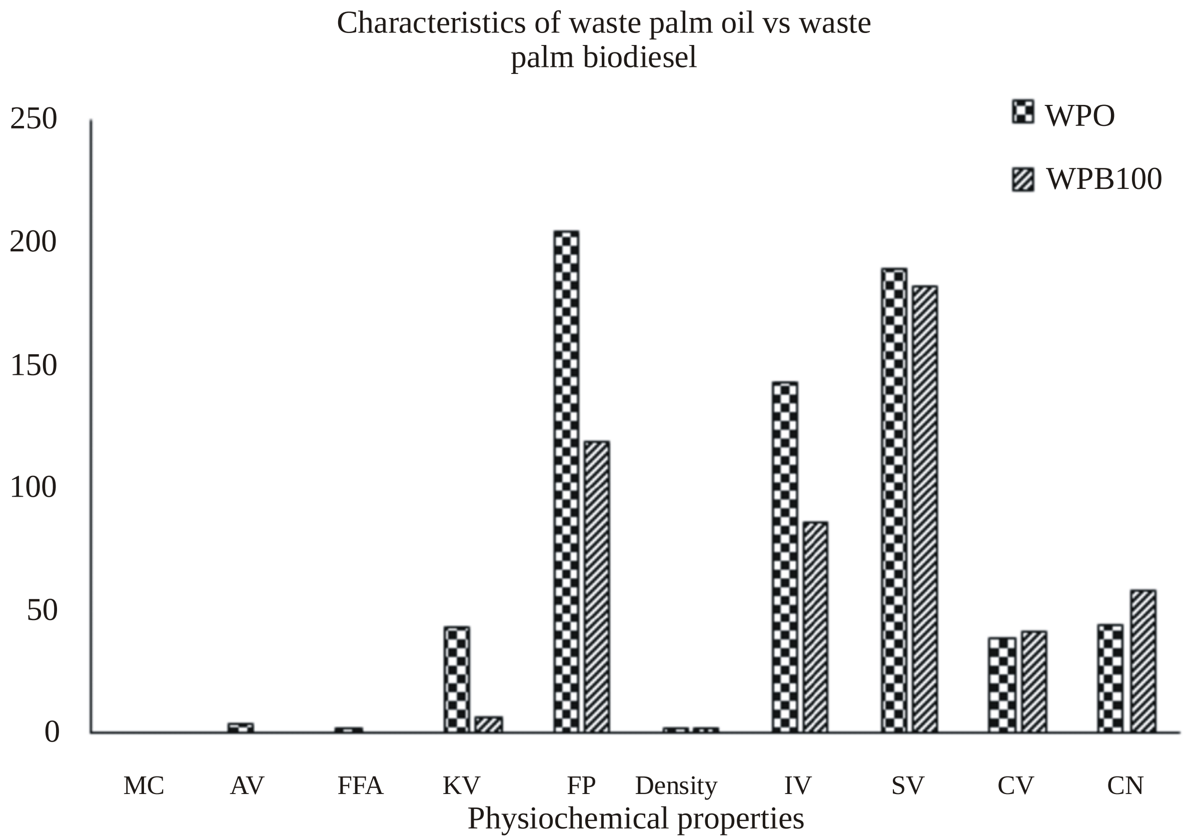

Figure 1: Physiochemical properties of WPO compared with WPB100

The reason is that through transesterification reaction large molecules of tri-glyceride which are the major components of oil are chemically modified by the bonding of free fatty acid with methanol in the presence of NaOH catalyst to form biodiesel or Fatty acid methlyester (FAME) as shown in Figure 2. The molecules of FAME (biodiesel) are smaller and are chemically modified such that it could burns smoothly in diesel engine (Kinast, 2003). The balance of transesterification reaction is the by-product known as glycerine (Figure 2). By implication the process of transesterification is the substitution of glycerine with alcohol in a chemical reaction using a catalyst (Hossain et al. 2010b).

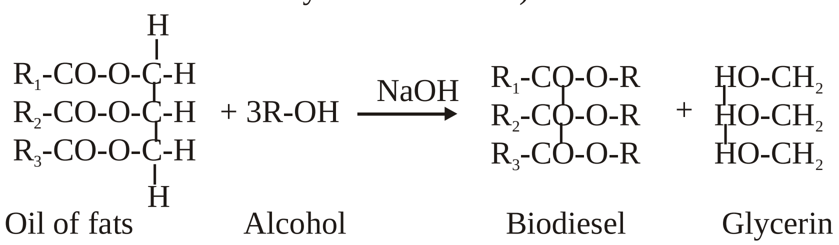

Figure 2: Transesterification reaction (Source: Hossain et al. 2010)

The results of the biodiesel production by transesterification experiment designed using Central Composite Design (CCD) are presented Table 4. Table 4 contains the results of the actual yield as well as the predicted yields obtained at different levels of treatment combinations of the variables as generated by software. The actual yields in Table 4 are average of experimental results obtained during tests in

Table 4: Results of Yield and Predicted yield at different temperature, mixing speed and time of operation combinations.

S/N	Codes for Temp (A)	Codes for Time (B)	Codes for Mixing (C)	Actual temp. (^{0}C)	Actual time (mins)	Actual mixing speed (rpm)	Actual Yield (%)	Predicted yield (%)
1	-1	-1	-1	50	65	700	34	35.08
2	1	-1	-1	60	65	700	68	70.45
3	-1	1	-1	50	105	700	56	53.38
4	1	1	-1	60	105	700	75	79.75
5	-1	-1	1	50	65	900	42	41.42
6	1	-1	1	60	65	900	53	59.79
7	-1	1	1	50	105	900	74	75.72
8	1	1	1	60	105	900	82	85
9	¯2	0	0	45	85	800	46	48.24
10	+2	0	0	65	85	800	94	85.86
11	0	¯2	0	55	45	800	43	39.22
12	0	+2	0	55	125	800	78	75
13	0	0	¯2	55	85	600	64	62.03
14	0	0	+2	55	85	1000	77	72.46
15	0	0	0	55	85	800	88	88.02
16	0	0	0	55	85	800	88	88.02
17	0	0	0	55	85	800	88	88.02
18	0	0	0	55	85	800	88	88.02
19	0	0	0	55	85	800	88	88.02
20	0	0	0	55	85	800	88	88.02

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These average experimental values were fitted in Design Expert 6.0.6 software to generate the predicted yields using quadratic model. The quadratic regression models are given in Equations 5 and 6 in terms of coded factors and actual factors.

Final equation in terms of coded factors:

$$\text{Yield} = 88.17 + 11.18A + 10.90B + 2.92C - 7.47A^2 - 10.83B^2 - 7.29C^2 - 2.25AB - 4.25AC + 4.00BC \qquad R^2 = 0.9690 \qquad (5)$$

The corresponding final equation in terms of actual factors is given as Equation (6):

$$\text{Yield} = -485.37 + 11.51\text{temp} + 1.33\text{time} + 0.38\text{mix} - 0.07\text{temp}^2 - 6.77X10^{-3}\text{time}^2 - 1.82251X10^{-4}\text{mix}^2 - 5.62500X10^{-3}\text{temp.time} - 2X10^{-3}\text{temp. mix} + 5.\ X10^{-4}\text{time. mix} \qquad (6)$$

Meanwhile, among five models namely mean, linear, 2FI, quadratic and cubic models tested for fitness by the software, quadratic model was selected as the best fitted model in prediction of biodiesel yield. Quadratic model was selected because the Prob>F value obtained from the software in the fitness test was 0.0001 which is the most significant at 0.05 probability level among those of the five models as shown in the sequential model sun of squares in Table 5. Those of 2FI and Cubic were not significant whereas that of Linear was significant but less than that of quadratic model.

Table 5: **Sequential Model Sum of Squares**

Source	Sum of Square	DF	Mean Square	F value	Prob>f	Remarks
Mean	99969.80	1	99969.80			
Linear	3446.95	3	1148.98	5.62	0.0079	
2FI	313.00	3	104.33	0.46	0.7159	
Quadratic	2749.97	3	916.66	4.01	<0.0001	Suggested
Cubic	121.05	4	30.26	2.08	0.2015	Aliased
Residual	87.23	6	14.54			
Total	1.067E+005	20	5334.40			

Another reason for the selection of quadratic model was that it had the highest Adjusted R-Square value of 0.7609 among the five models as well as the least PRESS value of 1606.0 and standard deviation of 4.56 (Table 6). Models with highest Adjusted R^2 and lowest Press values are preferred to those because they are indicators of strong correlation between predicted and actual values. Although Cubic model has the least standard deviation (3.81) among the five models as shown in Table 6, however the Adjusted R- square value is negative. It is also Aliased and the PRESS value is very high therefore cannot be selected.

Table 6: **Model Summary Statistics**

Source	Std. Dev	R-Squared	Predicted R-Squared	Adjusted R-Squared	PRESS	Remarks
Linear	14.30	0.5131	0.4218	0.2946	4739.13	
2FI	15.09	0.5597	0.3564	-0.4216	9550.86	
Quadratic	4.56	0.9690	0.9411	0.7609	1606.23	Suggested
Cubic	3.81	0.9870	0.9589	-1.8621	19228.25	Aliased

Meanwhile the relationship between the predicted and actual yields shown in Figure 3 indicated that the quadratic model successfully captured the relationship between the actual and the predicted yield going by the high R-square value of 0.9926 in the regression shown in Equation 7.

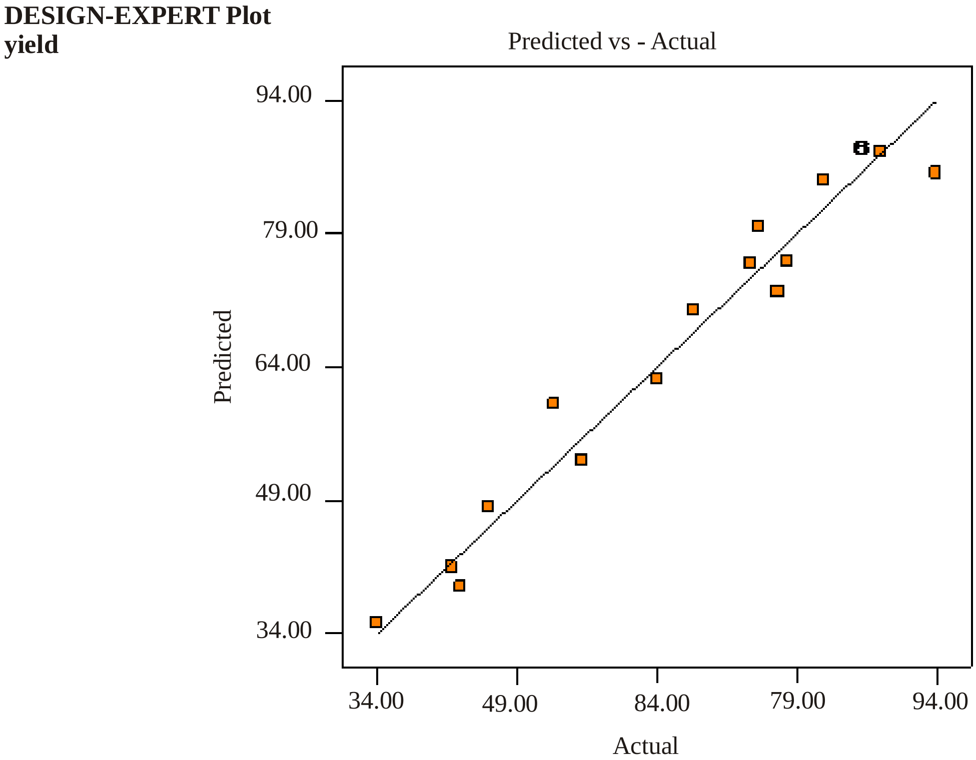

Figure 3: Relationship between predicted and actual yield for WPB100

$Y_{Pred.} = 0.9794\, Y_{ACT} + 1.53 \quad R^2 = 0.9926$ (7)

Where $Y_{Pred.}$ = the predicted yield (%) and Y_{ACT} = Actual yield (%)

According to Yang et al. (2015) large values of R^2 reveal that the model developed adequately captured the points in the workspace. It therefore implies that the developed model is reliable in representation of the relationship between the responses and the independent variables. The fact that high correlation coefficient existed between the predicted yield and the actual yield; it therefore gives credence to the model generated.

Result of the analysis of variance (ANOVA) performed to study the significance and fitness of the model is shown in Table 7.

From Table 7 it was observed that the model F-value of 34.73 implies that the model is significant. There is only a 0.01% chance that a model F-Value this large could occur due to noise. Values of Prob > F less than 0.0500 in Table 7 indicate the model terms that are significant.

Table 7: Results of the ANOVA of Response Surface Quadratic model and coefficient of model variables

Source	Coefficient of model factors	P -Values	Prob > F	Remarks
Model	88.17	34.73	< 0.0001	Significant
A(temp)	11.18	82.00	< 0.0001	Significant
B(time)	10.90	77.91	< 0.0001	Significant
C(mix)	2.92	5.59	0.0397	Significant
A^2	-7.47	38.58	< 0.0001	Significant
B^2	-10.83	81.09	< 0.0001	Significant
C^2	-7.29	36.77	< 0.0001	Significant
AB	-2.25	1.94	0.1934	Non-significant
AC	-4.25	6.94	0.0250	Significant
BC	4.0	6.15	0.0326	Significant

R-Squared	0.9690	Std. Dev.	4.56
Adjusted R-Squared	0.9411	Mean	70.70
Predicted R-Squared	0.7609	C.V.	6.46
Adequate Precision	16.450	PRESS	1606.23

In this experiment A, B, C, A^2, B^2, C^2, AC, BC are the significant model terms (Table 7). The significant terms have been explained as terms that have more positive influence on production yield while the non-significant terms are those that their changes could not meaningful change the amount of yield during experiment (Sulaiman et al. 2013). The high value of the Adjusted R^2 (0.9411) implied strong correlation between the predicted data and the experimental data. In this test the predicted R-Squared of 0.7609 is in reasonable agreement with the Adjusted R-Squared of 0.9411 (Table 7). The closer the regression coefficient R^2 is to 1, the better the models are fitted to the experimental data (Yang et al. 2015). According to the software, Adequate Precision value which is a measure of noise ratio greater than 4 is desirable. In this test, Adequate Precision ratio of 16.450 (Table 7) indicates an adequate model signal.

Also, the lack of fit of the models are not significant. Non-significant lack of fit indicates that the models were well fitted to the data, therefore transformation of the model was not required.

Again, in Table 7 it can be seen that some Model variables have positive and negative coefficients as well as significant and non-significant. According to (Sulaiman et al. 2013) a positive coefficient of model terms reveals synergistic effect while a negative term implies the antagonistic effect on transesterification process. In this test when a variable has both positive coefficient and also significant effect, it implies that such variable was every active in the enhancement of biodiesel production. The reserved is the case of a variable with negative coefficient and significant effect. Among the variables with positive coefficients and significant effect are temperature, time and mixing speed. Temperature has value of 11.18, time of operation has coefficient of 10.90 and mixing speed has 2.92 as shown in Table 7. It can be deduced that reaction temperature and time of operation had more impact on biodiesel yield than mixing speed. The test of optimization of waste palm kernel oil biodiesel yield done by Ayodele et al. (2017) indicated temperature of reaction as the most active variable among methanol to oil ratio, catalyst concentration, and reaction time. But reaction temperature was the least effective factor in the optimization of sunflower oil biodiesel yield among methanol to oil ratio and catalyst concentration (Abdullah et al. 2012). Also, observed in Table 7 is that the interaction between time and mixing speed with positive coefficient of 4.00 is significant; therefore it affected yield than the interaction between temperature and time of operation with negative coefficient of -2.25 and that between mixing speed and temperature of operation with negative coefficient (-4.25) that have non-significant effects.

The results of the 3D surface plots of the interaction effects of temperature-time, temperature-mixing speed, and time-mixing speed on yield are shown in Figure 4, 5 and 6 respectively. The Figures show that yields were influenced by three variable: temperature of reaction, mixing speed, and time of reaction. Figure 4 shows the 3D surface plot of the effect of time of reaction from 45minutes to 125 minutes and temperature from 45^{0}C to 65^{0}C on the biodiesel yield. The red lines under are the contour lines.

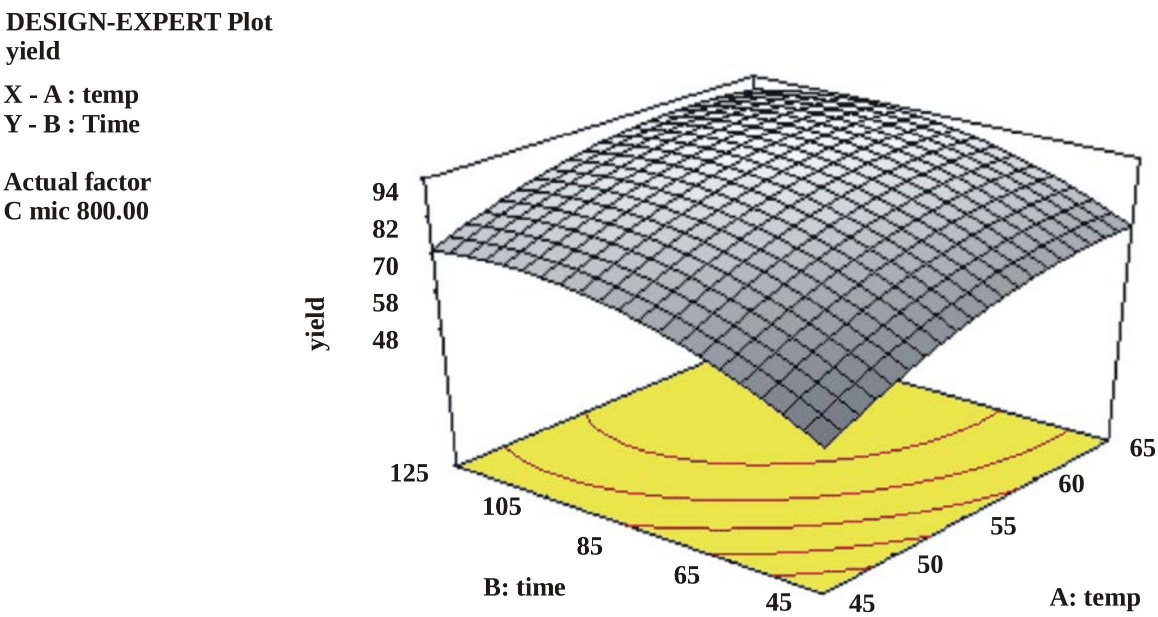

Figure 4. 3D surface plot of temperature and time interaction on WPB100 yield

From Figure 4, it can be seen that biodiesel yield increased from 45% at 45 ^{0}C and 45mins treatment combination to 72% at 65^{0}C and 45mins interactions. It was lowest at 45^{0}C and highest at 65^{0}C at 45mins of operation time. Also yield increased from 45% at 45minutes and 45^{0}C treatment combination to 93.6% at 101minutes reaction time thereafter dropped to 89.7% at 125 minutes. From the study increasing the reaction time above 125minutes was counterproductive in term of biodiesel yield. Also it can be seen from the curves that increase in temperature caused more yield than increase in time of operation. The observation is in agreement with the work reported by Eevera et al. (2009) and Ayodele et al. (2017). Gan et al. (2012), Wang et al. (2007) also reported increase in biodiesel production at temperature increase. Figure 5 shows the 3D surface plot of the effect of temperature and mixing speed on yield.

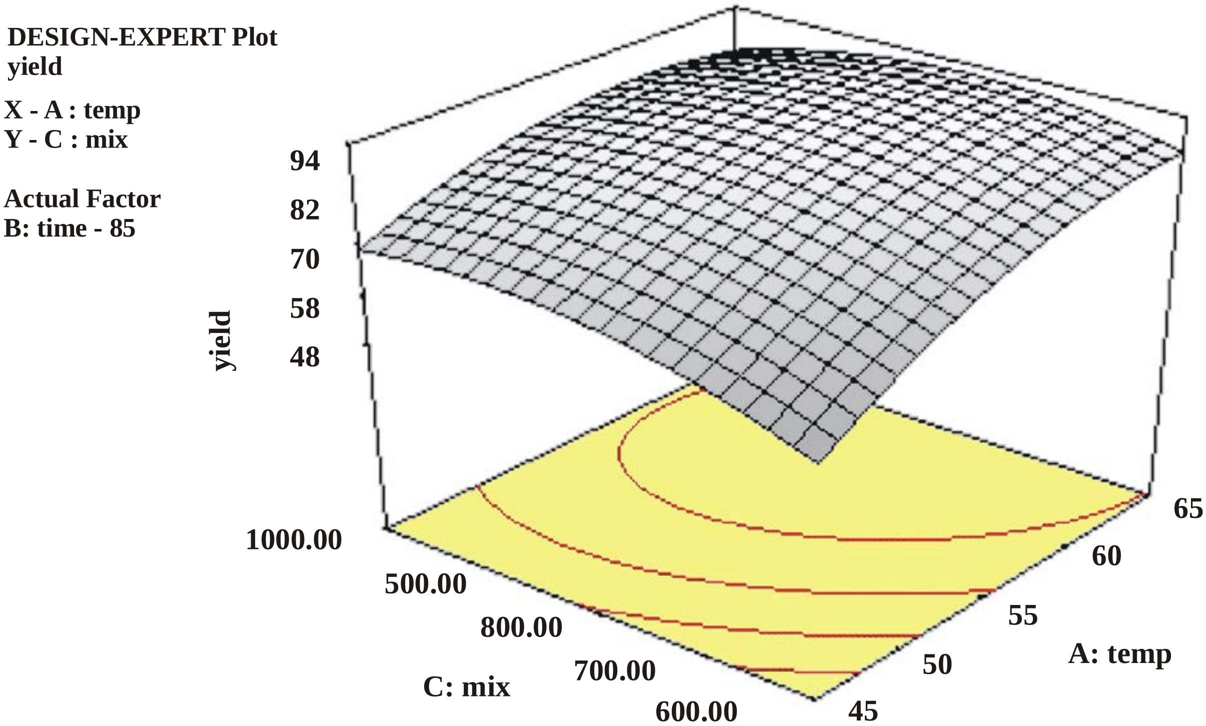

Figure 5: 3D response surface plot of the effect of temperature and mixing speed on WPB100 yield.

Figure 5 shows that biodiesel yield increased from 55% at 600rpm and 65^{0}C interaction to 86% at 65^{0}C. But as mixing speed increases from 600rpm to 1000rpm, yield increased to 92% at 819rpm and then dropped to 83% at 1000rpm. It can be deduced from Figure 5 that beyond the mixing speed rate production yield was reversed because the reagents had less contact as a result of high speed. Figure 6 shows the 3D response surface plot of the effect of time and mixing speed on yield.

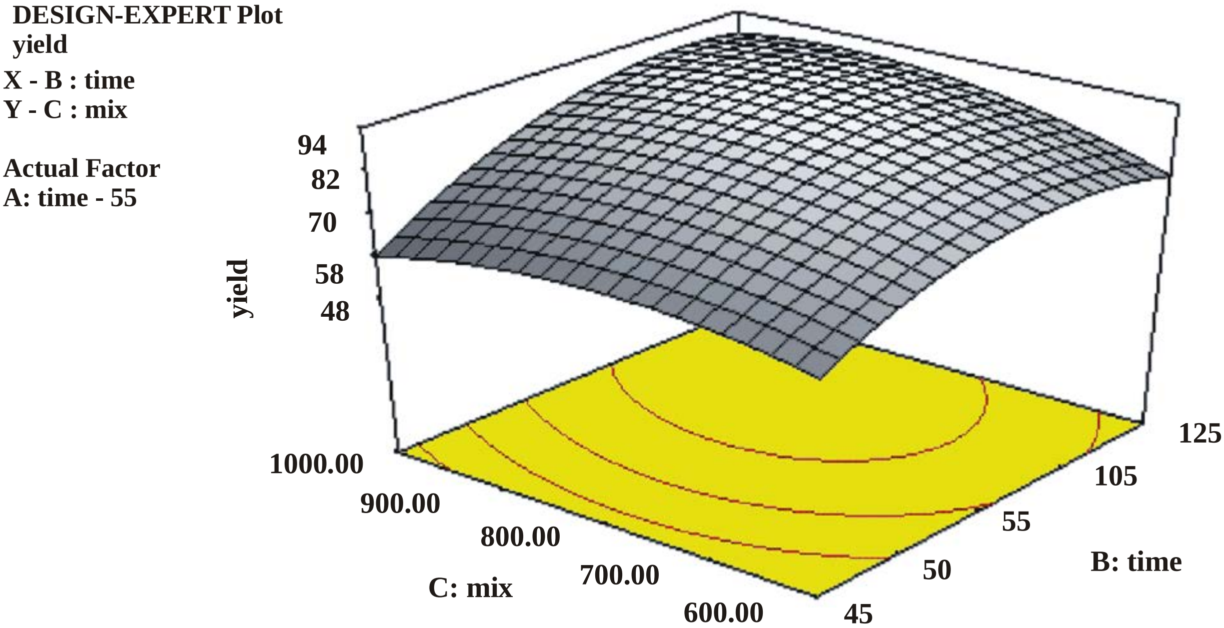

Figure 6: 3D surface plot of the effects of temperature and mixing speed interaction WPB100

From Figure 6 it can be seen that at mixing speed of 600rpm and time of 45mins treatment combination, biodiesel yield was 60%. Yield increased to 79% at 101mins and 600rpm treatment combination but dropped to 74% at 1000rpm and 45mins treatment combination. Also observed was that increase in time of operation at mixing speed of 1000rpm indicated yield increased from 58% to 89 % (at 112mins) thereafter dropped to 88% (at 125mins). It implies that the highest yield was observed at mixing speed of 816rpm. Therefore beyond 819rpm rate of production yield was reversed because the reagents had less contact as a result of high speed. Bello et al. (2016) also reported yield increase with speed but not up to 816rpm.

Also, the model standard error shown in Figure 7 indicates that error in model prediction increases towards the two extreme temperatures of 65^{0}C and 45^{0}C but least at 55^{0}C. Error also increased at the two extreme time of 45mins and 125mins respectively but least at 85mins. Therefore prediction of biodiesel yield with the developed model was most appropriated close to 85minutes of operation time and 55^{0}C operation temperature. At these two conditions, errors in prediction with the developed model was minimal as shown in Figure 7. Therefore the model is dependable in its prediction.

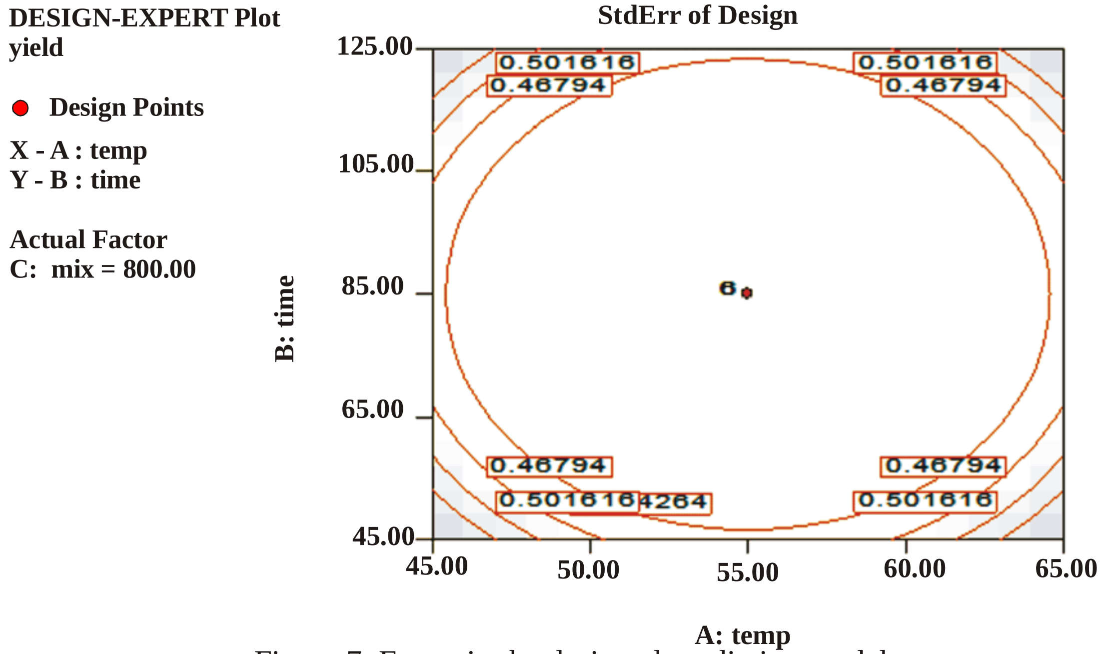

Figure 7: Errors in the designed prediction model

Optimisation of yield was undertaken using the conditions listed in Table 8. The constraints for reaction temperature, time of operation, mixing speed were 'in range' as shown in Table 8, whereas the goal for biodiesel production was maximal yield.

Table 8: Constraints of optimisation of yield

Name	Lower goal	Upper limit	Lower limit
Temp	is in range	45	65
Time	is in range	45	125
Mix	is in range	600	1000
Yield	Maximize	34	94

The results of ten selected treatments combinations that gave optimal yield are presented in Table 9. These ten selected treatment combinations were suggestion by the software.

Table 9: Results of percentage yield of the ten selected treatment combinations

Serial number of experiment	Temperature	Time	Mixing speed	Yield (%)	Desirability
1	61.50	100.03	866.53	94	1.00
2	62.77	101.07	836.51	94	1.00
3	59.80	103.15	874.91	94	1.00
4	62.24	98.08	796.65	94	1.00
5	59.98	107.05	821.76	94	1.00
6	60.25	105.15	828.10	94	1.00
7	62.37	108.09	821.79	94	1.00
8	63.34	102.43	805.18	94	1.00
9	62.63	102.03	849.77	94	1.00
10	61.97	104.33	850.55	94	1.00

From Table 9, the ten selected experiments indicate that optimal yield was 94% at the different treatment combinations stated. Also, the desirability is 1 indicating that for all treatment combinations the model satisfactorily predicted the yields and that the model can be used to optimize yield of waste palm oil biodiesel. Treatment combinations of 59.80^{0}C, 103.15mins and 874.91rpm in Table 9 was used for biodiesel production and after three replications yielded average of 94%. Meanwhile biodiesel yield optimisation conducted by Yang et al. (2015) using camelina as raw material indicated 97% yield which is above the yield of this work. Their yield was at the optimal reaction conditions of 38.7°C reaction temperature, 40 min reaction time, 7:1 molar ratio of methanol/oil, and 1.5% weight of oil for catalyst concentration. The maximum yield for the production of methyl esters from sunflower oil was predicted to be 98.181% under the condition of temperature of 48°C, the molar ratio of methanol to oil of 6.825:1, catalyst concentration of 0.679 % weight of oil, stirring speed of 290rpm and a reaction time of 2hours in the experiment done by *Mansourpoor and Shariati (2012)*. The study conducted by Ayodele et al. (2017) on biodiesel yield from waste groundnut oil (WGO), waste soybean Oil (WSO) and waste palm kernel oil (WPKO) production catalyzed with potassium hydroxide (KOH) reported 98.5% yield for WGO and WSO and 97.7% yield for WPKO respectively. The optimal conditions of the experiment indicated that methanol per oil mole ratios and the catalyst concentrations were higher than the values used in this sudy whereas the time of operation in this work were higher than theirs. But the range of temperature of operation of this experiment and theirs are same.

CONCLUSION

Waste palm oil is mostly discarded by restaurants in Owerri, Imo state because they claim that it has lost its initial nutritional quality therefore is no longer fit to prepare palatable meal that appeals to their customers. In this study, waste palm oil was collected from three restaurants and converted to biodiesel by transesterification with methanol and NaOH alkaline catalyst. The optimal yield of 94% was recorded at 59.8^{0}C temperature, 103.15minutes reaction time and 874.91rpm mixing speed. It was also observed that effects of the linear variables namely temperature of reaction, time of operation and mixing speed were significant. The interactions between mixing speed and temperature of operation and that between mixing speed and time of operation respectively were significant whereas interaction between temperature and time of operation was non-significant. The variables with positive coefficients and significant effect are temperature with +11.18, time of operation with coefficient +10.90 and mixing speed with +2.92. Consequently, reaction temperature and time of operation were more impactful on biodiesel yield than mixing speed. A quadratic model fitted by Design Expect 6.06 version with R-square value of 0.9690 successfully predicted the expected yield.

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