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Optimization of Process Parameters for Transesterification of Palm Kernel Oil to Produce Fatty Acid Ethyl Esters using Box-Behnken Design

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Abstract— *A Box-Behnken experimental design was used to investigate the parameters of the process that need optimization to produce ethyl esters of fatty acids (FAEE) biodiesel by transesterification of palm kernel oil (PKO). Two-step reaction which included the acid-catalyzed pretreatment of the esterification with transesterification using potassium hydroxide (KOH) and ethanol was used. The crude PKO contained 13mg of KOH/g of acid that was decreased to below 0.75mg of KOH/g by acid esterification with 2% H_2SO_4 catalyst at 60°C in 1 hour. The systematic investigation of three process variables of temperature (45-65°C), catalyst loading (0.75-1.50wt%), and ethanol-oil molar ratio (6:1-9:1) was carried out at three levels through 17 experimental runs. The ANOVA has shown that the quadratic model was statistically significant ($F = 9.30$, $p = 0.0038$), and the most important factors were catalyst loading ($p = 0.0004$), and temperature ($p = 0.0025$). The loading of catalyst and the ethanol-to-oil ratio (BC) also interacted ($p = 0.0126$). The yields of biodiesel were between 78.78-99.58%. The optimal conditions were determined in the form of 47.98°C, 0.937wt% KOH, and 7.92:1 ratio of ethanol and oil with a predicted yield of 97.9% and desirability of 1.000, respectively. The resulting biodiesel had a range of density of 816 -868 kg/m³ and an average kinematic viscosity of 4.00-7.68 mm²/s at 40°C. The regression equation ($R^2 = 0.9228$) was sufficiently representative of the correlation between the process variables and the biodiesel yield.*

Keywords— *Biodiesel, feedstock, esterification, free fatty acid, ethanol, optimization, RSM.*

I. Introduction

The world energy environment is experiencing serious change due to the fear of exhaustion of fossil fuels, energy security, and the destruction of the environment. Transesterification of vegetable oils and animal fats has resulted in biodiesel, which is a potential renewable energy source in place of petroleum-based diesel fuel. Recent reports have indicated that modern biofuels could be an effective long term renewable energy source with the potential of responding to the issue of environmental impact as well as security issue that is currently being experienced due to the dependency on fossil fuels [1].

Palm kernel oil (PKO) is a promising feedstock to biodiesel production, especially in tropical areas where the production of oil palm is common. Nigeria being one of the major producers of palm oil has great PKO that can be used to manufacture biodiesel. The typical fatty acid composition of PKO is high levels of lauric acid (about 48%), a large percentage of saturated fatty acids (84-85%), thus giving the resulting biodiesel an excellent oxidative stability as well as high cetane number [2]. Nevertheless, the raw palm kernel oil cannot be utilized immediately as diesel fuel, since its viscosity (26–30 mm²/s at 40°C) would be excessive to use in engines [3].

The most common process of biodiesel production using vegetable oils is the process of transesterification. It is done through reaction of triglycerides with an alcohol (usually methanol or ethanol) in the presence of a catalyst to form fatty acid alkyl ester (biodiesel) and glycerol as a co-product. The overall reaction of transesterification may be illustrated by:

$$\text{Triglyceride} + 3\,\text{ROH} \rightleftharpoons 3\,\text{RCOOR'} + \text{Glycerol}$$

A number of benefits are associated with using ethanol over methanol, such as that it is renewable when made using biomass, less toxic, and fatty acid ethyl ester (FAEE) has better cold flow properties than fatty acid methyl ester (FAME) [4]. Efficiency of the production of bio-diesel process is determined by a number of process variables, which include the reaction temperature, the catalyst type and concentration, the molar ratio of the alcohol and the oil, and the reaction time [5]. In the case of feedstocks that contain high levels of free fatty acid (FFA), a two-stage process is normally necessary [6]. Box-Behnken Design (BBD) and Response Surface Methodology (RSM) have been widely used in optimization in the production of biodiesel [7].

In Nigeria, a number of researches have been carried out on the transesterification of PKO. The highest percentage of yield was 95.8% on PKO biodiesel through the use of NaOH-catalyzed methanolysis. Ishola et al. [1] studied the production of biodiesel using the palm olein in Nigeria. Ayoola et al. [8] used the RSM and ANN analysis to optimize the crude PKO biodiesel production. Nevertheless, there are not enough systematic optimization studies which use RSM to optimize the process of PKO ethanolysis using KOH catalyst.

The proposed research is to optimize the process parameters in terms of transesterification of palm kernel oil using potassium hydroxide catalyst and ethanol by means of Box-Behnken experimental design. The targeted objectives are to: (i) describe the PKO feedstock and clean it in terms of FFA content by applying acid-catalyzed esterification pretreatment; (ii) examine the influence of temperature, catalyst concentration on biodiesel yield using BBD; (iii) formulate a quadratic regression model; (iv) determine optimal conditions by using

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numerical optimization; and (v) determine the fuel properties of the resulting biodiesel. Materials and Methods

II. Materials and Methods

A. Materials

The palm oil or crude palm kernel oil (PKO) was purchased at one of the local palm oil processing plants located in Warri, Delta State, Nigeria. The oil was dried and filtered at 105°C and left at 30 minutes to evaporate all the moisture[9].Anhydrous ethanol(99.5% purity), Anhydrous methanol(99.5% purity), potassium hydroxide(KOH, 85% purity), and concentrated sulfuric acid(H_2SO_4, 98% purity) were used. The chemicals were all of reagent grade.

B. Determination of Acid Value

The standard titration was used to determine the acid value of the PKO (ASTM D664). About 10g of oil was dissolved in 50 ml of neutralized ethanol-diethyl ether, phenolphthalein, and titrated against 0.1 N KOH. The acid value was determined by applying Equation (1):

$$\text{Acid Value (mg KOH/g)} = (56.1 \times N \times V) / m \quad (1)$$

and 56.1 is the molar weight of KOH (g/mol), N is the normality of KOH solution (eq/L), V is volume of titrant (mL) and m is the weight of oil sample (g). The amount of fatty acids was determined as the free fatty acid (FFA) by Equation (2):

$$\text{FFA (\%)} = \text{Acid Value} / 1.99 \quad (2)$$

The acid value of the first PKO was 13.0 ± 0.3 mg KOH/g (FFA = 6.5) and was higher than the allowable concentration of direct base catalysis (<2mg KOH/g) [10]. Thus, it was necessary that acid-catalyzed esterification pretreatment was carried out

C. Pretreatment Esterification Pretreatment by Acid-Catalyst

The process of the esterification changes the free fatty acids to fatty acid methyl esters as shown in the Equation (3):

$$RCOOH + CH_3OH \rightleftharpoons RCOOCH_3 + H_2O \quad (3)$$

The procedure: 500g of PKO was refluxed in a 3-neck flask of 1000mL at 55°C with reflux condenser. methanol and H_2SO_4 catalyst (2% w/w of oil) were mixed separately. methanol to FFA molar ratio was held at 6:1. The reaction was stirred and allowed 1 hour at 55°C. The mixture was poured into a separatory funnel after completion and allowed to rest in the funnel during 2 hours. The water layer was pumped out and the oil was washed thrice using warm distilled water (50°C) in order to eliminate the leftover acid. The dried oil was heated to 105°C in vacuum over 30 minutes in getting rid of moisture. Postpretreatment acid value was 0.75 g + 0.05mg KOH/g which was able to decrease FFA levels to below the critical point (<0.5%) [11].

D. Transesterification Procedure

The process of transesterification is as follows: The transesterification reactions were done in batch mode using three necked flask of 500 ml with magnetic stirrer, thermometer and reflux condenser. The pretreated PKO concentration was 50g per run and heated to the necessary temperature [12]. The amount of ethanol and KOH needed were calculated by using equation (4) and (5):

$$m_{EtOH} = (r_{EtOH} \times m_{oil} \times M_{EtOH}) / M_{TG} \quad (4)$$

$$m_{cat} = (w_{cat} \times m_{oil}) / 100 \quad (5)$$

where m_{EtOH} is the mass of ethanol (g), r_{EtOH} is the ethanol-to-triglyceride molar ratio, m_{oil} is the mass of oil (50 g), M_{EtOH} is the mass of the ethanol (46.07 g/mol), M_{TG} is the average mass of the triglyceride of PKO (873.0 g/mol) and m_{cat} is the mass of the catalyst (g) and w_{cat} is the percentage loading of the catalyst. Sample calculations for Run 1 (7.5:1 molar ratio, 1.125 wt% catalyst):

Ethanol mass = (7.5 × 50 × 46.07) / 873.0 = 19.79 g

Catalyst mass = (1.125 × 50) / 100 = 0.562 g

The KOH was dissolved in ethanol to form potassium ethoxide, added to preheated oil with stirring at 400 rpm for 90 minutes under reflux. After reaction, the mixture was transferred to a separatory funnel for 4 hours. The glycerol layer was drained, crude biodiesel was washed three times with warm water, and dried at 105°C under vacuum.

E. Box-Behnken Experimental Design

The design was a three-factor, three-level Box-Behnken design that researched the impact of a temperature (A), catalyst loading (B), and ethanol-to-oil molar ratio (C) on biodiesel yield. The levels of factors are presented in Table 1.

TABLE 1: Independent variables and their coded levels for Box-Behnken design

Factor	Symbol	-1 (Low)	0 (Center)	+1 (High)
Temperature (°C)	A	45	55	65
Catalyst loading (wt%)	B	0.75	1.125	1.50
Ethanol:oil molar ratio	C	6:1	7.5:1	9:1

The design was designed to comprise of 17 experimental runs with 12 factorial points and 5 center point replicas to estimate the error of the experimental. The runs were performed randomly.

F. Biodiesel Yield Calculation

The biodiesel yield was calculated on a mass basis using Equation (6) [13]:

$$\text{Yield (\%)} = (m\_biodiesel / m\_oil) \times 100 \quad (6)$$

G. Statistical Analysis

Experimental data were analyzed using Design-Expert® software (Version 13). A second-order polynomial equation (Equation 7) was fitted [14]:

$$Y = \beta_0 + \Sigma\beta_i x_i + \Sigma\beta_{ii} x_i^2 + \Sigma\Sigma\beta_{ij} x_i x_j + \varepsilon \quad (7)$$

Where Y is the response to be predicted, β_0 indicates the intercept, β_i are the linear coefficients, β_{ii} are quadratic coefficients, β_{ij} are interaction coefficients and e is the random error. ANOVA was done at 95% confidence. The desirability function method was used in carrying out numerical optimization.

III. Results and Discussion

A. Experimental Results

Table 2 presents the complete Box-Behnken design matrix with calculated reactant quantities and experimental responses. Biodiesel yields ranged from 78.78% (Run 14) to 99.58% (Run 15).

B. Analysis of Variance (ANOVA)

Table 3 presents the ANOVA results for the response surface quadratic model. The model F-value of 9.30 ($p = 0.0038$) indicates statistical significance. There is only 0.38% probability that such a large F-value could occur due to random noise [14].

TABLE 2: Box-Behnken design matrix with experimental responses

Run	T (°C)	Cat (%)	EtOH:Oil	Cat (g)	EtOH (g)	Yield (%)	ρ (kg/m³)	ν (mm²/s)
1	55	1.125	7.5:1	0.562	19.79	90.20	843	5.38
2	55	1.500	6.0:1	0.750	15.83	85.08	828	4.00
3	65	1.125	9.0:1	0.562	23.75	84.42	842	4.58
4	45	1.125	6.0:1	0.562	15.83	97.70	833	4.58
5	65	1.125	6.0:1	0.562	15.83	91.44	830	7.36
6	45	1.125	9.0:1	0.562	23.75	95.70	841	5.20
7	55	1.125	7.5:1	0.562	19.79	94.78	845	5.16
8	45	1.500	7.5:1	0.750	19.79	86.24	868	6.26
9	55	0.750	6.0:1	0.375	15.83	89.66	862	6.32
10	55	1.125	7.5:1	0.562	19.79	91.70	839	5.44
11	45	0.750	7.5:1	0.375	19.79	98.38	840	6.36
12	55	1.125	7.5:1	0.562	19.79	91.70	840	5.41
13	55	1.125	7.5:1	0.562	19.79	91.02	854	5.38
14	55	1.500	9.0:1	0.750	23.75	78.78	816	5.21
15	55	0.750	9.0:1	0.375	23.75	99.58	868	7.68
16	65	0.750	7.5:1	0.375	19.79	87.82	867	5.33
17	65	1.500	7.5:1	0.750	19.79	82.62	835	5.82

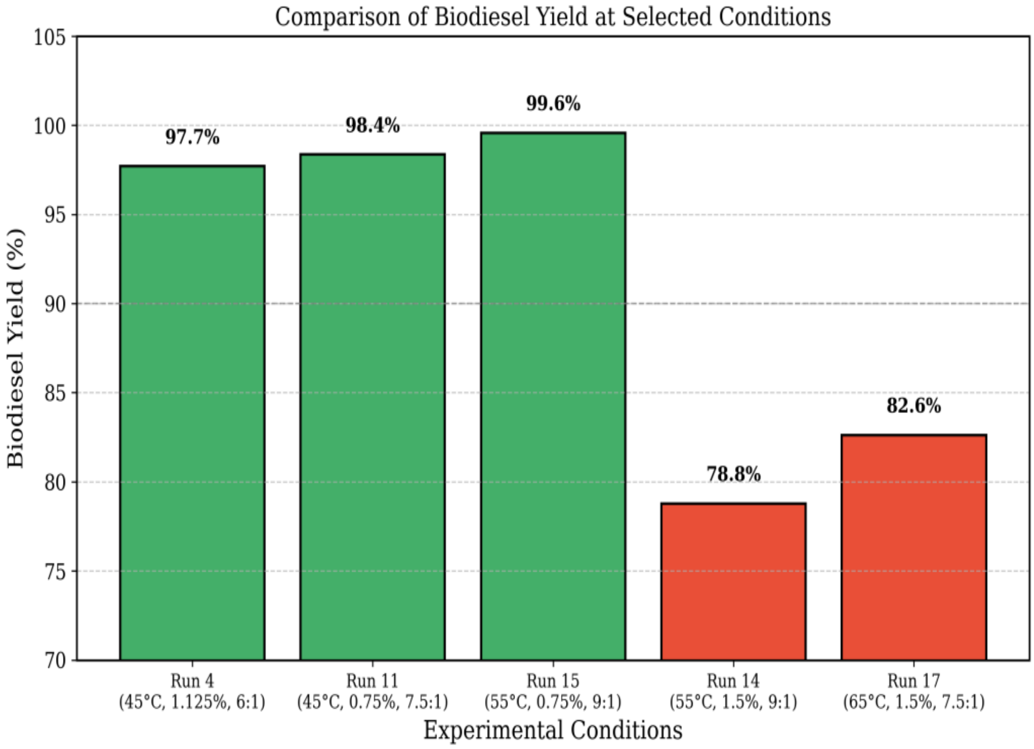

Figure 1: Comparison of biodiesel yield at selected experimental conditions

TABLE 3: ANOVA for quadratic model (Response: Biodiesel Yield)

Source	Sum Sq.	df	Mean Sq.	F	p-value	Signif.
Model	496.03	9	55.11	9.30	0.0038	Significant
A-Temperature	125.77	1	125.77	21.21	0.0025	Significant
B-Catalyst	228.12	1	228.12	38.48	0.0004	Significant
C-EtOH:oil	3.64	1	3.64	0.61	0.4587	Not significant
BC	65.77	1	65.77	11.09	0.0126	Significant
B^2	53.89	1	53.89	9.09	0.0195	Significant
Lack of Fit	29.46	3	9.82	3.26	0.1415	Not significant
$R^2 = 0.9228$	Adj $R^2 = 0.8235$	-	-	-	-	Prec. = 11.57

The significant terms are: temperature (A, $p = 0.0025$), catalyst loading (B, $p = 0.0004$), BC interaction ($p = 0.0126$), and B^2 ($p = 0.0195$). The lack-of-fit is not significant ($p = 0.1415$), confirming adequate model fit. $R^2 = 0.9228$ indicates 92.28% of variability is explained by the model.

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C. Regression Model

The quadratic regression model in terms of actual factors is (Equation 8):

$$\text{Yield (\%)} = 47.478 - 0.798(A) + 71.620(B) + 12.445(C) + 0.463(AB) - 0.084(AC) - 7.209(BC) + 0.005(A^2) - 25.440(B^2) - 0.012(C^2) \quad (8)$$

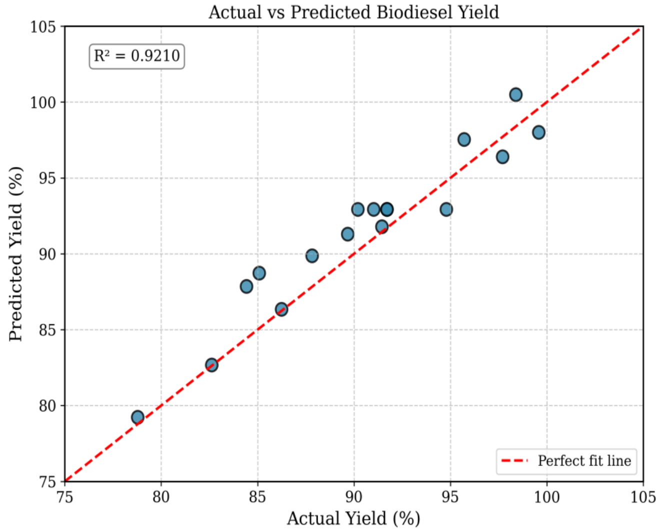

Figure 2: Actual versus predicted biodiesel yield ($R^2 = 0.9228$)

D. Effect of Process Parameters

Temperature exhibited a significant negative effect on biodiesel yield ($F = 21.21$, $p = 0.0025$). At 45°C, the average yield was 94.51%, decreasing to 91.88% at 55°C and 86.58% at 65°C. This inverse relationship is attributed to saponification side reactions at elevated temperatures [16]. Phan and Harvey [11] demonstrated that FAEE saponification proceeds at rates 3.5 times higher in ethanol-hydroxide systems compared to methanol systems.

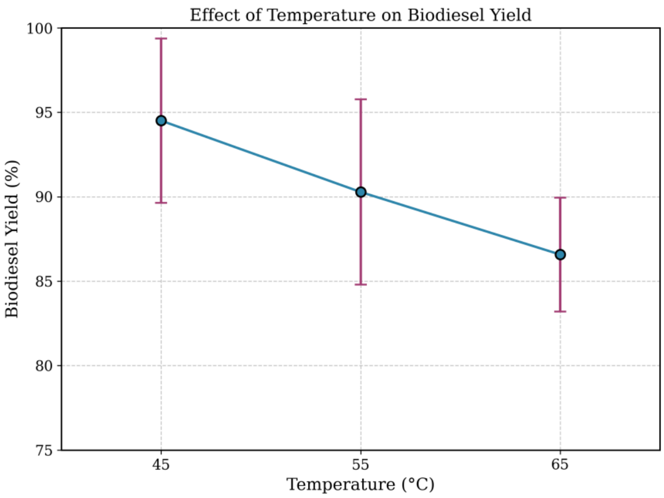

Figure 3: Effect of reaction temperature on biodiesel yield

Catalyst loading was the most significant factor ($F = 38.48$, $p = 0.0004$). At 0.75 wt% KOH, average yield was 93.86%, decreasing to 83.18% at 1.50 wt%. Excess catalyst promotes saponification [7], [17]. The significant quadratic term B^2 ($p = 0.0195$) confirms curvature in the response.

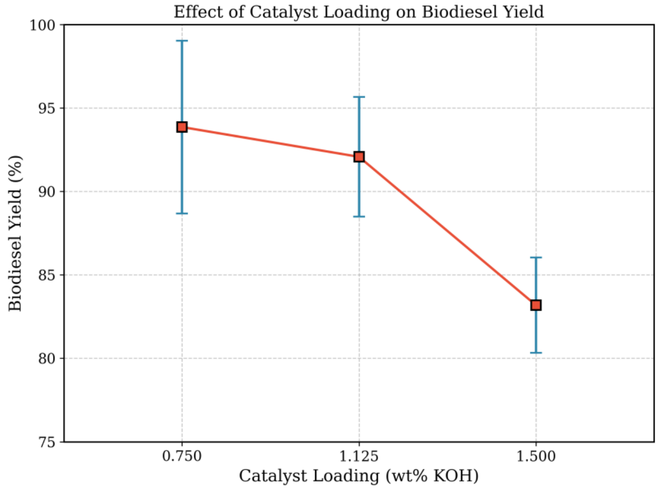

Figure 4: Effect of KOH catalyst loading on biodiesel yield

The ethanol ratio main effect was not significant ($p = 0.4587$), but its interaction with catalyst (BC) was significant ($p = 0.0126$). This is demonstrated by comparing Run 15 (0.75 wt%, 9:1, yield = 99.58%) with Run 14 (1.50 wt%, 9:1, yield = 78.78%) [18].

E. Response Surface Analysis

The relevance of the significant BC interaction is shown in Figure 5. At low catalyst (0.75 wt%), more ethanol ratio leads to higher yield, whereas at a high catalyst (1.50 wt%), higher ethanol ratio leads to dramatic reduction in yield due to greater solubility of soap.

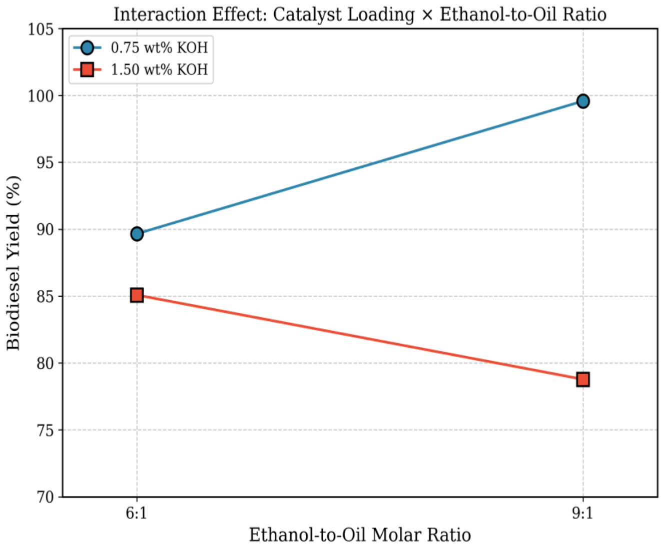

Figure 5: Interaction effect between catalyst loading and ethanol ratio at 55°C

The response surface in 3D (Figure 6) represents the yield as a function of the temperature and the amount of catalyst loaded at a constant ethanol ratio (7.5:1). Maximum yield is obtained at low temperatures and moderate-to-low catalyst loadings.

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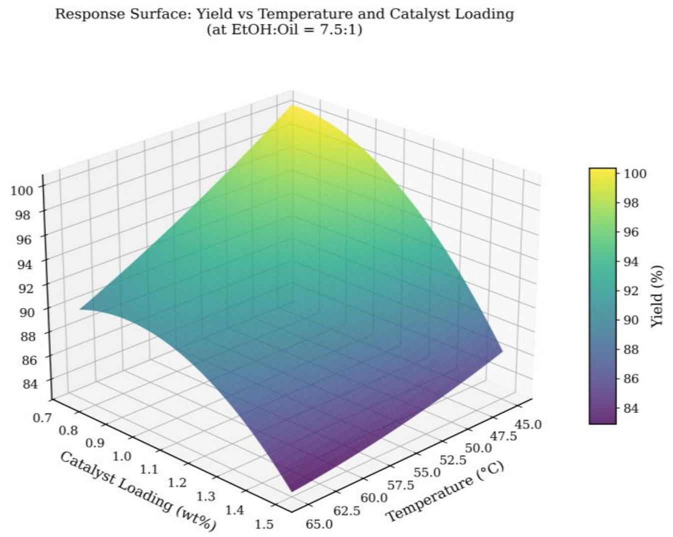

Figure 6: 3D response surface plot of biodiesel yield as function of temperature and catalyst loading

The contour plot (Figure 7) shows the yield as a function of the catalyst loading and the ethanol ratio at 55°C. Highest yields (> 95%) are obtained by low catalyst loading (0.75-0.90 wt%) with higher ethanol ratios (8-9:1).

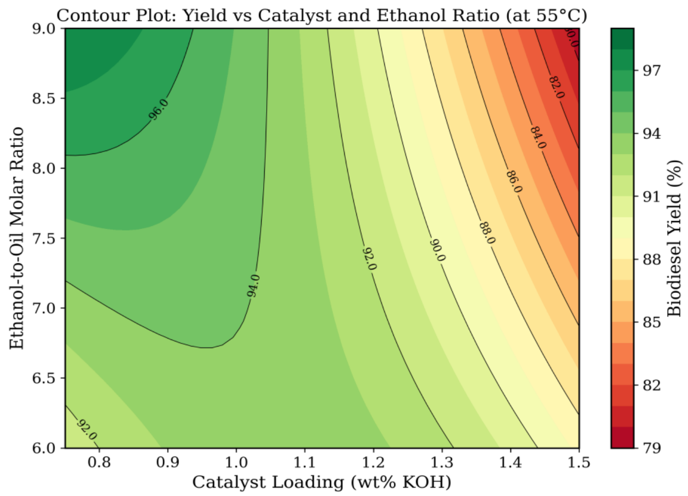

Figure 7: Contour plot of biodiesel yield as a function of catalyst loading and ethanol ratio

F. Numerical Optimization

Numerical optimization using the desirability function approach. determined the optimum (Figure 8). The software tested 100 solutions under the terms of the experiment.

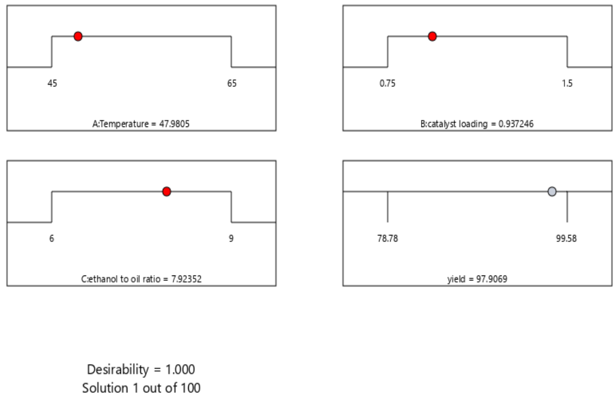

Figure 8: Optimization ramp from Design-Expert(r) (Desirability = 1.000)

TABLE 4: Numerical optimization optimal conditions

Parameter	Optimal Value
Temperature (A)	47.98°C
Catalyst loading (B)	0.937 wt% KOH
Ethanol-to-oil molar ratio (C)	7.92:1
Predicted yield	97.91%
Desirability	1.000

The desirability value of 1.000 means that the optimization goal was completely reached. The optimum temperature of 47.98°C balances saponification with minimum catalyst loading of 0.937wt% to balance the catalytic activity and excessive saponification. The ethanol ratio of 7.92:1 gives enough excess alcohol without too much dilution. These results are comparable to those of Nigerian PKO biodiesel by Sabapathy et al. [24] and Oloyede et al. [25].

G. Fuel Properties

Table 5 compares the produced PKO biodiesel properties with international standards [26].

The densities were between 816 to 868 kg/ m³, most of the samples were within the requirements of EN 14214. The kinematic viscosity was 4.00 to 7.68 mm²/s with 82% of samples passing ASTM D6751 [27]. The transesterification obtained about 75-85% decrease in viscosity relative to raw PKO (27.8 mm²/s).

TABLE 5: Comparison of PKO biodiesel properties with international standards

Property	PKO Biodiesel	ASTM D6751	EN 14214
Density at 15°C (kg/m³)	816-868	Not specified	860-900
Kinematic viscosity at 40°C (mm²/s)	4.00-7.68	1.9-6.0	3.5-5.0
Maximum experimental yield (%)	99.58	-	≥96.5 (ester)
Optimized predicted yield (%)	97.91	-	-

IV. Conclusion

This paper was able to demonstrate optimization of process parameters in the production of FAEE biodiesel from palm kernel oil using Box-Behnken design. Key findings:

1. Pretreatment of Crude PKO with acid value 7.0 mg KOH/g was done successfully using 2% H_2SO_4 at 60°C for 1 hour to decrease the acid value to 0.46mg KOH/g (reduced 93.4%).
2. The quadratic model (R^2 = 0.9228, Adj R^2 = 0.8235) satisfactorily explained the process with sufficient accuracy of 11.57 and an insignificant lack of fit (p = 0.1415).
3. The greatest significance was experienced in catalyst loading (F = 38.48, p = 0.0004) and then the temperature (F

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= 21.21, p = 0.0025). The interaction of the BC was also important (p = 0.0126).

4. Yields between 78.78 to 99.58 with a top yield at 55°C, 0.75 wt% KOH and 9:1 ethanol.
5. The optimal conditions are temperature = 47.98°C, catalyst = 0.937 wt% KOH, ethanol ratio = 7.92:1, and predicted yield = 97.91% and desirability= 1.000.
6. PKO biodiesel had density 816 - 868 kg/ m^3 and viscosity 4 -7.68 mm^2/s, most of them falling within the ASTM D6751 limits.

These results offer lead in production of renewable biodiesel using the locally produced feedstocks in Nigeria and other palm oil producing nations. The future work ought to explore storage stability, validation experiments, engine performance testing and techno-economic analysis.

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