

Optimization of Process Parameters for Biodiesel Production Using Response Surface Methodology

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Abstract: The effect of five process parameters namely: reaction time, reaction temperature, stir speed, catalyst concentration and methanol-oil ratio on the transesterification process of waste frying oil to biodiesel were investigated. Optimization of the five process parameters and their quadratic cross effect was carried out using a four level-five factor central composite experimental design model and response surface methodology with each factor varied over four levels. Taking the biodiesel yield as the response of the designed experiment, the data obtained were statistically analysed to get a suitable model for optimization of biodiesel yield as a function of the five independent process parameters. The optimization produced 30 feasible solutions whose desirability equals to 1 and the selected (most desirable) condition was found to be: reaction time (3 hrs), reaction temperature (58°C), stir speed (305.5 rpm), catalyst concentration (1.4 wt%) and methanol to oil ratio (6:1), while the optimum yield of biodiesel for this condition was found to be 91.6%. The developed model was tested and validated for adequacy by substituting random experimental values as input parameters and the output parameters from the developed model were close to the experimental values. The biodiesel properties were characterized and the results obtained were found to satisfy the standard for both the ASTM D 6751 and EN 14214.

Keywords: Biodiesel, Central Composite Design, Optimization, Transesterification, Yield

1. Introduction

Biodiesel is one of such renewable alternative fuel derived from triglycerides by transesterification of vegetable oils and animal fats (Nie, *et al.*, 2006; Shibasaki-Kitakawa *et al.*, 2007; Aworanti *et al.*, 2013)

Biodiesel is sustainable, renewable, biodegradable, safe to handle and simple to use, environmental friendly, non-toxic, and essentially free of sulphur and aromatics. (Monyem *et al.*, 2001, Highina *et al.*, 2012). Chemically, biodiesel is a fuel composed of mono-alkyl ester of long chain fatty acid derived from vegetable oil or animal fat, designated as B100 and meeting the requirements of ASTM (American Society for Testing and Materials) D 6751 or EN (European Norm) 14214 (Hai, 2002, European Biodiesel Board, 2006). It can

be used either in the pure form (B100) or as blends with fossil diesel in diesel engines (Canacki and Van Gerpen, 2005; Basiron and May, 2005). According to Bello (2008), biodiesel can be produced from used frying oil, coconut oil, palm oil etc. however high demand of diesel fuel and the availability of waste cooking oil indicate that biodiesel from used oil cannot completely replace fossil diesel fuel but can contribute to reduce the dependency on petrol based diesel (Martin and Grossman, 2011).

2. Physical Experiment

Waste cooking oil was obtained from Afe Babalola University, Ado Ekiti restaurants in Ado Ekiti, Nigeria and was filtered and pre-heated to remove impurities. Reagents namely methanol (99.8% purity), hydrogen chloride (HCl),

sodium hydroxide (NaOH) and anhydrous sodium sulphate (Na_2SO_4) of analytical grade were purchased from Brilliant Chemical store in Lagos, Nigeria. Methanol was the choice of alcohol because it is cheap and it is a short chain alcohol that reacts faster. The catalyst employed for both acid and alkali transesterification were hydrogen chloride (HCl) and sodium hydroxide (NaOH) prepared at different concentration ranging from 1.0wt% - 2.0wt%. Acid transesterification was used for converting used cooking oil to biodiesel using 99.8% pure methanol, HCl and NaOH as catalyst. Waste cooking oil was filtered and pre-heated at a temperature of 100°C to remove water and other volatile impurities. HCl at a concentration of 1.4 wt% was added to methanol with methanol in excess in a molar ratio of 6:1 to oil. The mixture was thoroughly stirred in the mixing tank for 1 hour to form methanolic HCl. The pre-heated oil was thereafter transferred to the reactor alongside with methanolic HCl and the mixture was stirred at 305 rpm and a temperature of 60°C for 2 hours. The product was discharged into the separating funnel and allowed to settle for 24 hours. After the settling, the product is observed to have separated into two distinct layers. The top layer being a mixture of water and methanol while the bottom layer the transesterified oil. This is the first process aimed at breaking the long chain of fatty acid from used oil. This process is followed by alkali transesterification. The transesterified oil was transferred back into the reactor where prepared sodium hydroxide (NaOH) at a concentration of 1.4 wt% and methanol at a molar ratio of 6:1 to oil (Methanol in excess to oil) forming sodium methoxide was added. Continuous stirring of the mixture was done in the reactor at 305 rpm at 60°C for 3 hours. The product was thereafter discharged into the separating funnel and allowed to settle for 24 hours after which the products separated into two distinct layers. The lighter biodiesel at the top and the heavier glycerol at the bottom. The methyl ester (biodiesel) was then washed with distilled water at a volume ratio of 3:1 by stirring gently. The methyl ester was dried by passing it through anhydrous sodium sulphate (Na_2SO_4). The dried biodiesel was stored in a refrigerator to prevent oxidation.

Waste cooking oil was obtained from Afe Babalola University, Ado Ekiti restaurants in Ado Ekiti, Nigeria and was filtered and pre-heated to remove impurities. Reagents namely methanol (99.8% purity), HCl, Sodium hydroxide, anhydrous sodium sulphate of analytical grade were purchased from Brilliant Chemical store in Lagos, Nigeria. Methanol was the choice of alcohol because it is cheap and it is a short chain alcohol that reacts faster. The catalyst employed for both acid and alkali transesterification, HCl and sodium hydroxide was prepared at different concentration ranging from 1.0 wt% - 2.0wt%. Acid transesterification was used for converting used cooking oil to biodiesel using 99.8% pure methanol, HCl and NaOH as catalyst. Waste cooking oil was filtered and pre-heated at a temperature of 100°C to remove water and other volatile impurities. HCl at a concentration of 1.4 wt% was added to methanol with methanol in excess in a molar ratio of 6:1 to oil. The mixture was gently stirred in the mixing tank for 10

minutes to form methanolic HCl. The pre-heated oil was thereafter transferred to the reactor alongside with methanolic HCl and the mixture was stirred at 305 rpm and a temperature of 60°C for 3 hours. The product was discharged into the separating funnel and allowed to settle for 24 hours. After the settling, the product separated into two distinct layers. The top layer being a mixture of water and methanol while the bottom layer the transesterified oil. This is the first process aimed at breaking the long chain of fatty acid from used oil. This process is followed by alkali transesterification. The transesterified oil was transferred back into the reactor where prepared sodium methoxide was added. NaOH is at a concentration of 1.4 wt% and methanol at a molar ratio of 6:1 (Methanol in excess to oil). Continuous stirring of the mixture was done in the reactor at 305 rpm at 60°C for 3 hours. The product was thereafter discharged into the separating funnel and allowed to settle for 24 hours after which the products separated into two distinct layers, the lighter biodiesel at the top and the heavier glycerol at the bottom. The methyl ester (biodiesel) was then washed with distilled water at a volume ratio of 3:1 by stirring gently. The methyl ester was dried by passing it through anhydrous sodium sulphate (Na_2SO_4). The dried biodiesel was stored in a refrigerator to prevent oxidation.

3. Numerical Experiment

Modelling and Optimization of the production process variables were carried out with Design-Expert® (version 7) software for experiment design using a four-level-five factor central composite design model and response surface methodology to study the effect of independent variables such as reaction time (hours), temperature (°C), stir speed (rpm), catalyst concentration (wt%) and methanol-oil ratio on the biodiesel yield with the response in terms of percentage yield. The Response Surface Methodology (RSM) is a viable statistical tool for the optimization of process variables as it simplifies complex nature of many experimental runs. Besides, it studies the interactive effect of two or more variables and the effect on the response (target). The following input process parameters were varied.

- i. Reaction time: 1-5 hours.
- ii. Reaction temperature: 40-90°C.
- iii. Stir Speed: 200-400 rev/min.
- iv. Catalyst concentration: 1-2 wt%.
- v. Methanol to Oil ratio: 4:1-9:1.

4. Central Composite Design

The Central Composite Design is the most widely used RSM model. Response surface methodology (RSM) is a mathematical/statistical based technique which is useful for analyzing the effects of several independent variables on the response (Box and Drapper, 1987; Enweremadu and Rutto, 2015). It is used to investigate the quadratic cross effect of five input process parameters namely: time, temperature, stir speed, catalyst concentration and methanol-oil ratio on biodiesel yield. Each numeric factor is

varied over 4 levels: plus and minus alpha (axial point). Plus and minus 1 (factorial point). Uosukainen *et al.*, (1999), Ghadge and Raheman *et al.*, (2006), Kansedo *et al.*, (2009), Salamatina *et al.*, (2013 a & b), Jeong and Part (2009), Jeong and Park *et al.*, (2009), Silva *et al.*, (2006), Aworanti *et al.*, (2013) and Enweremadu and Rutto (2015),

were some of the authors who have investigated and reported the optimization of biodiesel production using the response surface methodology. However, the quadratic cross effect of five process parameters has not been investigated.

Table 1. Numeric Factors and Levels.

s/n	Factor	Name	Unit	-1 Level	+1 Level	-alpha	+alpha
1.	A	Reaction time	hours	1	5	0.00930244	5.9907
2.	B	Reaction temperature	°C	40	90	27.6163	102.384
3.	C	Stir speed	rpm	200	400	150.465	449.535
4.	D	Catalyst concentration	wt%	1	2	0.752326	2.24767
5.	E	Methanol to oil ratio		4	9	4.74302	0.256977

Table 1 shows the input values for process parameters denoted as numeric factors over 4 levels. This generated a runs of 15 experiments and the data obtained was statistically analysed with Design-Expert® (version 7) software to get a suitable model for biodiesel yield (%) as a function of the independent variables. The biodiesel yield is taken as the response of the designed experiment for the

transesterification process.

Table 2 summarizes the experiment designed in terms of study type which is response surface using central composite as initial design and a quadratic design model. The numbers of experimental runs was 50. From Table 2, the mean and standard deviation for each process variable as well as biodiesel yield was calculated.

Table 2. Summary of Design for Numerical Experimentation Analysis.

Design Summary			
Study type	Response surface	Runs	50
Initial design	Central composite	Blocks	No blocks
Design model	Quadratic		

Factor	Name	Units	Type	Low Actual	High Actual	Low Coded	High Coded	Mean	Std. Dev.
A	Time	hours	Numeric	1.00	5.00	-1.000	1.000	3.000	1.708
B	Temperature	°C	Numeric	4.00	90.00	-1.000	1.000	65.000	21.352
C	Stir speed	rpm	Numeric	200.00	400.00	-1.000	1.000	300.000	85.407
D	Catalyst concentration	w%	Numeric	1.00	2.00	-1.000	1.000	1.500	0.427
E	Methanol-oil ratio		Numeric	4.00	9.00	-1.000	1.000	6.500	2.135

Response	Name	Units	Obs.	Analysis	Minimum	Maximum	Mean	Std. Dev.	Ratio	Trans	Model
Y1	Yield	%	15	Polynomial	70.000	91.600	84.140	5.916	1.300	None	None

Mathematical model was formulated from each response (Biodiesel yield) which correlates the response (Yield) to the process variables through first and second order as well as interactive terms according to equation 1

$$Y = \beta_0 + \sum_{i=1}^k \beta_i x_i + \sum_{i>j}^k \beta_{ij} x_i x_j \quad (1)$$

where

Y=Response (Biodiesel Yield)

β_i =Linear regression coefficient

β_{ij} =Quadratic coefficient when $i=j$ and interactive effect

coefficient when $i \neq j$

β_0 =Regression coefficient

x_i, x_j = Independent process variables (uncoded)

k=Number of factors optimized in the experiment

Biodiesel was also produced from the developed pilot plant at optimum transesterification process condition. Yield of biodiesel from used and unused oil from transesterification process is calculated as shown in Table 3. Table 3 shows the feasible combinations of the five process variables with their yield determined experimentally for the first 15 runs according to equation 2

$$\text{Yield} = \frac{\text{Weight of biodiesel}}{\text{oil weight}} \times 100\% \quad (2)$$

Table 3. Process Variables and Yield.

s/n	Standard deviation	Block	A: Reaction time (hrs)	B: Reaction temperature (°C)	C: Stir speed (rpm)	D: Catalyst concentration (wt. %)	E: Methanol to Oil	Yield (%)
1	29	1	1.00	40.00	400.00	2.00	9.00	88.8
2	13	1	1.00	40.00	400.00	2.00	4.00	89.9
3	18	1	5.00	40.00	200.00	1.00	9.00	90
4	8	1	5.00	90.00	400.00	1.00	4.00	81.5
5	36	1	3.00	102.38	300.00	1.50	6.50	80
6	31	1	1.00	90.00	400.00	2.00	9.00	84.2
7	25	1	1.00	40.00	200.00	2.00	9.00	91
8	10	1	5.00	40.00	200.00	2.00	4.00	70
9	26	1	5.00	90.00	200.00	2.00	9.00	78
10	14	1	5.00	40.00	400.00	2.00	4.00	86.7
11	9	1	1.00	40.00	200.00	2.00	4.00	90.2
12	45	1	3.00	65.00	300.00	1.50	6.50	78
13	16	1	5.00	90.00	400.00	2.00	4.00	90
14	4	1	5.00	90.00	200.00	1.00	4.00	82.8
15	32	1	5.00	90.00	400.00	2.00	9.00	81

5. Statistical Analysis

Using response surface quadratic model, the values of standard error, Variance Inflation Factor (VIF), and standard deviation were gotten for possible combination of process parameters as shown in Table 4.

Table 4. Design Matrix Evaluation for Response Surface Quadratic Model.

s/n	Coded Factor	Actual Factor	Standard Error	VIF	0.5% Standard deviation	1% Standard Deviation	2% Standard deviation
1	A	Time	0.17	1.00	30.9	83.1	99.9
2	B	Temperature	0.17	1.00	30.9	83.1	99.9
3	C	Stir speed	0.17	1.00	30.9	83.1	99.9
4	D	Catalyst concentration	0.17	1.00	30.9	83.1	99.9
5	E	Methanol-oil ratio	0.17	1.00	30.9	83.1	99.9
6	AB	Time × temperature	0.18	1.00	27.7	78.0	99.9
7	AC	Time × stir speed	0.18	1.00	27.7	78.0	99.9
8	AD	Time × catalyst concentration	0.18	1.00	27.7	78.0	99.9
9	AE	Time × methanol-oil ratio	0.18	1.00	27.7	78.0	99.9
10	BC	Temperature × stir speed	0.18	1.00	27.7	78.0	99.9
11	BD	Temperature × catalyst concentration	0.18	1.00	27.7	78.0	99.9
12	BE	Temperature × methanol-oil ratio	0.18	1.00	27.7	78.0	99.9
13	CD	Stir speed × catalyst concentration	0.18	1.00	27.7	78.0	99.9
14	CE	Stir speed × methanol-oil ratio	0.18	1.00	27.7	78.0	99.9
15	DE	Catalyst concentration × methanol-oil ratio	0.18	1.00	27.7	78.0	99.9
16	A ²	Time ²	0.29	1.31	38.0	91.1	99.9
17	B ²	Temperature ²	0.29	1.31	38.0	91.1	99.9
18	C ²	Stir speed ²	0.29	1.31	38.0	91.1	99.9
19	D ²	Catalyst concentration ²	0.29	1.31	38.0	91.1	99.9
20	E ²	Methanol-oil ratio ²	0.29	1.31	38.0	91.1	99.9

Table 4 shows, the five process parameters namely: reaction time, temperature, stir speed, catalyst concentration and methanol-oil ratio are represented by variables A, B, C, D and E. The polynomial analysis and errors were also calculated. The standard errors are similar within type of coefficient. The Smaller values are most significant. The Ideal Variance Inflation Factor (VIF) is 1.0, the result obtained is significant. VIF's above 10 depicts poor model indicating coefficients are poorly estimated due to multicollinearity. Also, Ideal R-squared is 0.0, high R-squared means terms are correlated with each other, possibly

leading to poor models. The predictive capability and significance of the model is further validated with the value of R-Squared (0.9895) and the Adjusted R-Squared (0.9263) close to 1. R-squared is a multiple correlation coefficient indicating the degree of relationship of the response variable to the combined linear predictor variable. The correlation coefficient values being a measure of goodness of fit of the model indicates high degree of correlation between the observed and predicted values (Aworanti, *et al.*, 2013)

The final equation in terms of coded factors is given as:

$$\begin{aligned} \text{Yield} = & 78.00 + 6.59 * A + 1.34 * B - 0.10 * C + 14.36 * D - 2.56 * E - 5.10 * A * B + 4.49 * A * C - 12.83 * A * D \\ & + 4.71 * A * E - 3.96 * B * C + 2.18 * B * D - 7.19 * B * E \end{aligned} \quad (3)$$

The desirability equals to 1 indicating the significance of the process variables to amount of yield and viability of the developed model for prediction.

Design-Expert® Software

Yield



X1 = A: Time

X2 = B: Temperature

Actual Factors

C: Stir speed = 300.00

D: Catalyst concentration = 1.50

E: Methanol-Oil ratio = 6.50

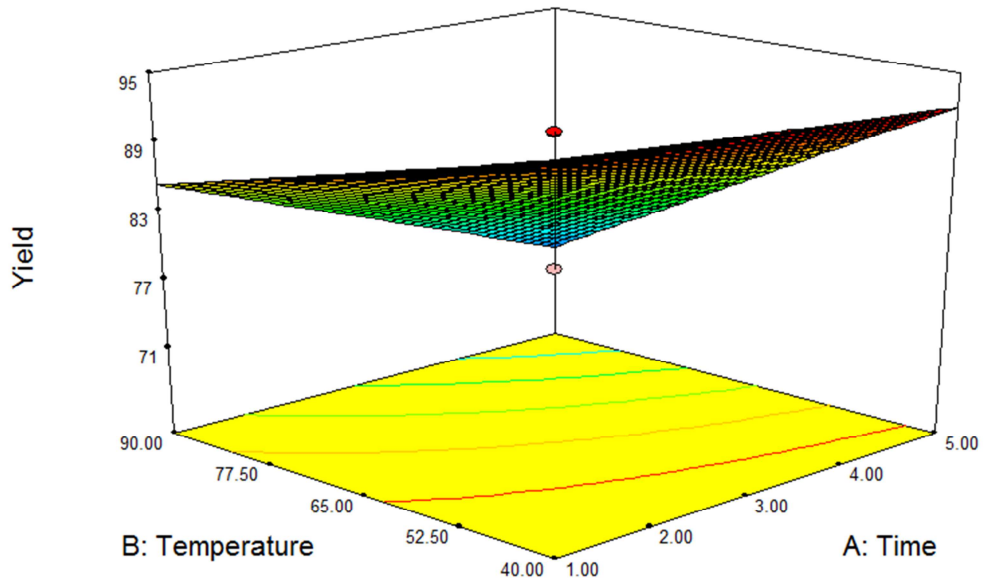


Figure 1. Effect of Interaction of Time and Temperature on Biodiesel Yield.

Figure 1 is a 3D Response Surface plot of the interaction effect of time and temperature on biodiesel yield when catalyst concentration is 1.50, methanol-oil ratio: 6.50 and stir speed: 300 rpm. From Figure 1, biodiesel yield increased significantly with increase in temperature and reaction time until a sharp decrease in yield above 58°C and 3 hours. This may be due to evaporation of methanol or reverse glycerolysis.

Design-Expert® Software

Yield



X1 = A: Time

X2 = C: Stir speed

Actual Factors

B: Temperature = 65.00

D: Catalyst concentration = 1.50

E: Methanol-Oil ratio = 6.50

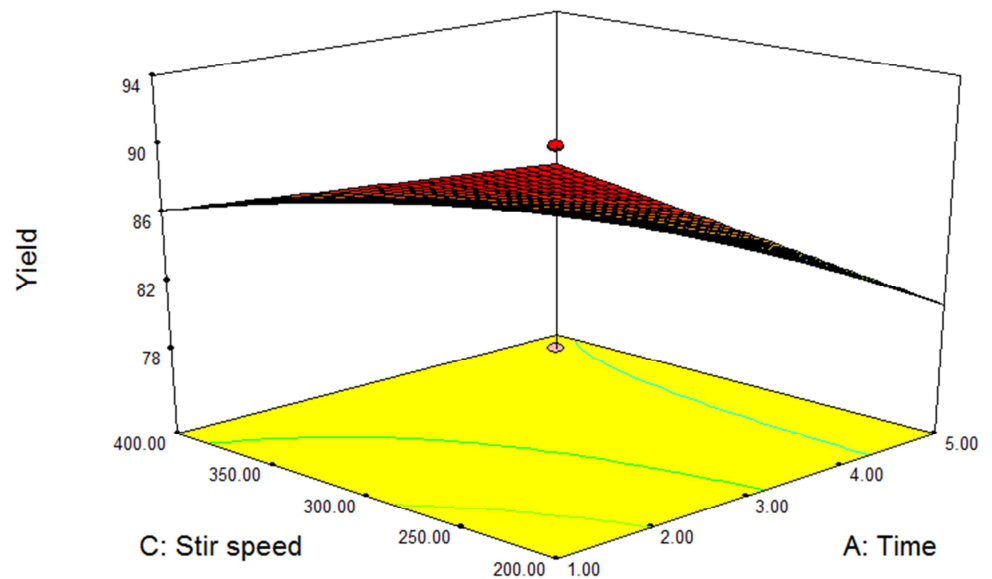


Figure 2. Effect of Interaction of Time and Stir Speed on Biodiesel Yield.

Figure 2 is a 3D response surface plot of the interaction effect of time and stir speed on biodiesel yield when catalyst concentration is 1.50, methanol-oil ratio: 6.50 at 65°C. From Figure 2, biodiesel yield increased significantly with increase in reaction time and stir speed until a decrease in yield above 3 hours and 305.5 rpm. This may be due to excessive agitation.

Design-Expert® Software

Yield
91
70

X1 = A: Time
X2 = D: Catalyst concentration

Actual Factors
B: Temperature = 65.00
C: Stir speed = 300.00
E: Methanol-Oil ratio = 6.50

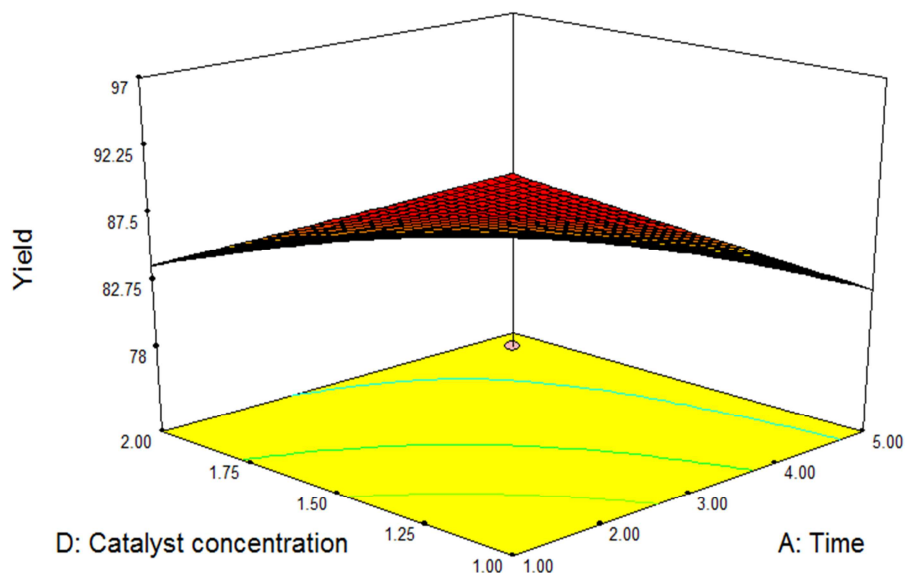


Figure 3. Effect of Interaction of Time and Catalyst Concentration on Biodiesel Yield.

Figure 3 is a 3D response surface plot of the interaction effect of time and catalyst concentration on biodiesel yield when methanol-oil ratio is 6.50, stir speed: 300 rpm at 65°C. Biodiesel yield increases with increase in catalyst concentration. Addition of excessive catalyst favours saponification reaction and reduces biodiesel yield (Goyal *et al.*, 2012).

Design-Expert® Software

Yield
91
70

X1 = A: Time
X2 = E: Methanol-Oil ratio

Actual Factors
B: Temperature = 65.00
C: Stir speed = 300.00
D: Catalyst concentration = 1.50

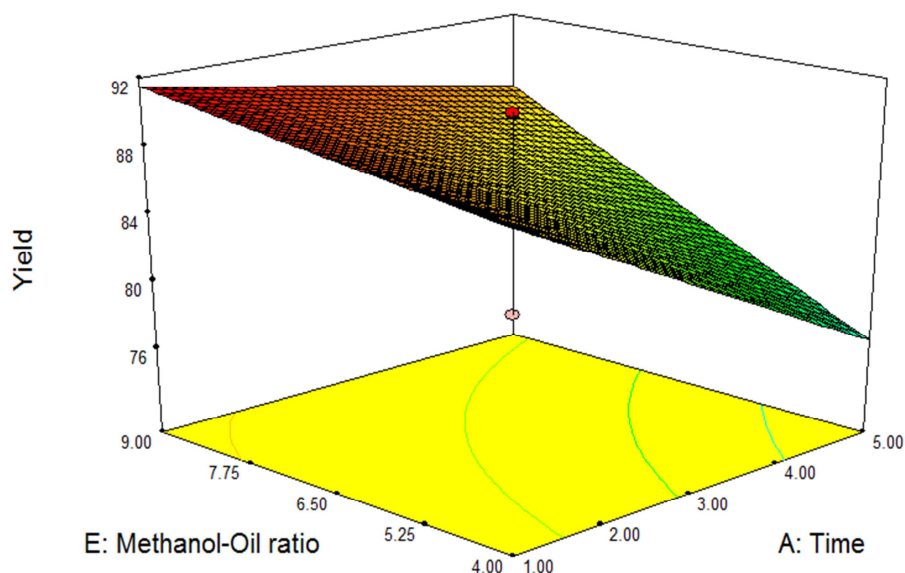


Figure 4. Effect of Interaction of Time and methanol-oil ratio on Biodiesel Yield.

Figure 4 is a 3D response surface plot of the interaction effect of time and methanol-oil ratio on biodiesel yield when catalyst concentration is 1.50, stir speed: 300 rpm at 65°C. Methanol-oil ratio of 6:1 (methanol in excess) for 3 hours brought about optimum yield of biodiesel while ratio greater than 6:1 brought about significant decrease in yield. Too much methanol reduces the flash point thus eroding an important advantage of biodiesel.

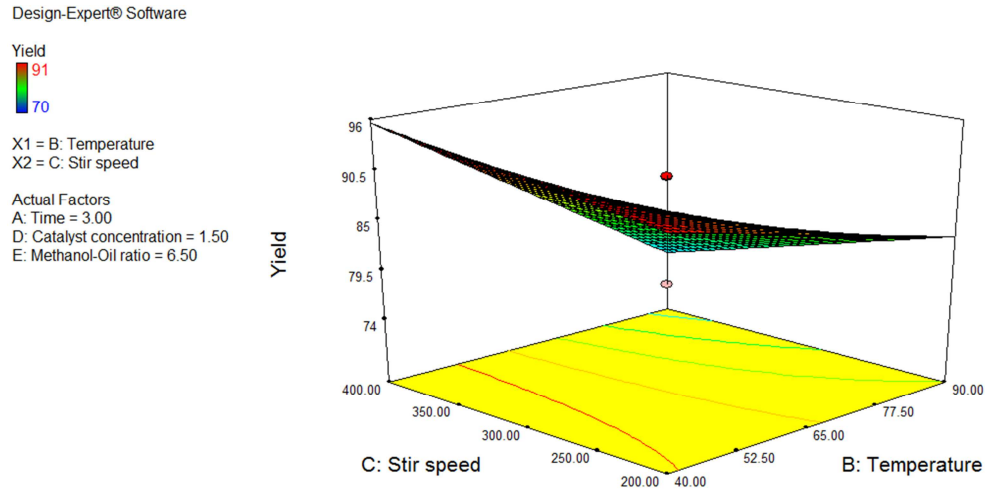


Figure 5. Effect of Interaction of Temperature and Stir speed on Biodiesel Yield.

Figure 5 is a 3D response surface plot of the interaction effect of temperature and stir speed on biodiesel yield when catalyst concentration is 1.50, methanol-oil ratio: 6.50 for 3 hours. Excessive agitation causes splashing and the mixture tend to foam which may result in cavitation corrosion.

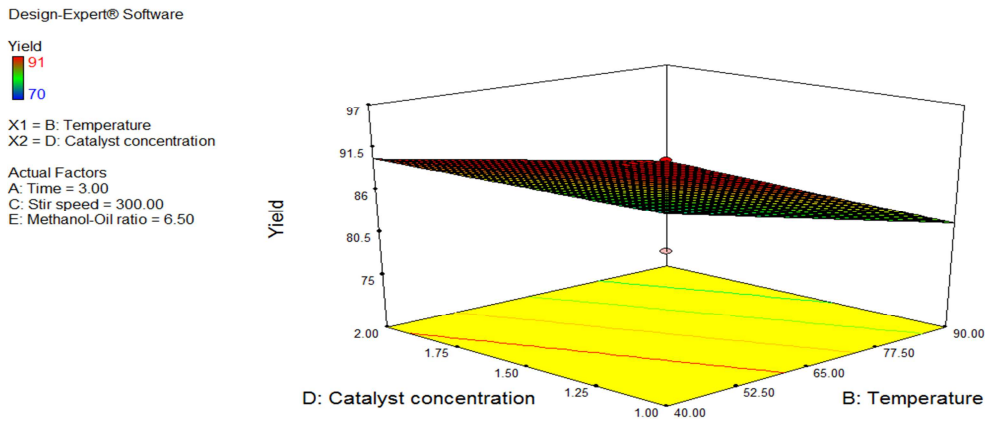


Figure 6. Effect of Interaction of Temperature and Catalyst Concentration on Biodiesel Yield.

Figure 6 is a 3D response surface plot of the interaction effect of temperature and catalyst concentration on biodiesel yield when methanol-oil ratio is 6.50 for 3 hours at a stir speed of 300 rpm. Excessive catalyst beyond 1.4 wt % decreases biodiesel yield as soap may be formed which prevents ester layer formation.

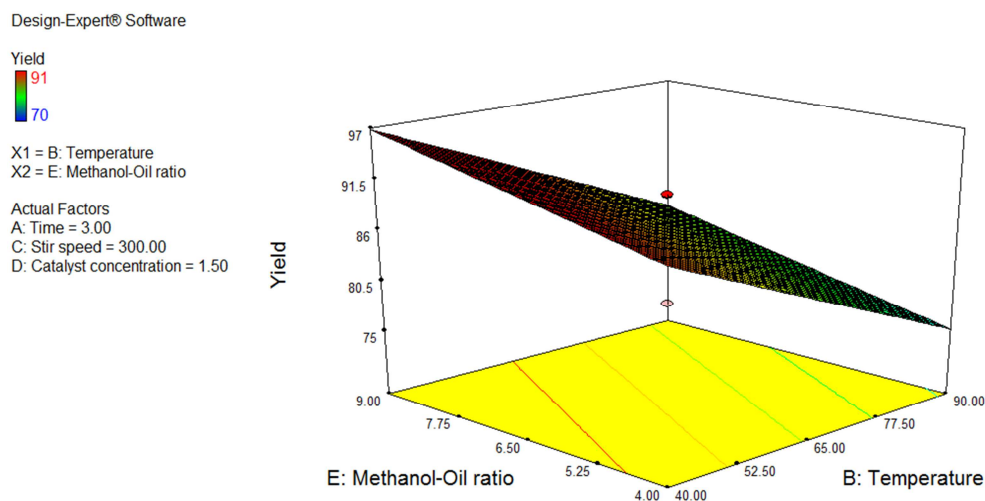


Figure 7. Effect of Interaction of Temperature and Methanol-oil ratio on Biodiesel Yield.

Figure 7 is a 3D response surface plot of the interaction effect of temperature and methanol-oil ratio on biodiesel yield when catalyst concentration of 1.50 for 3 hours at a stir speed 300 rpm. Biodiesel yield increases with increase in methanol oil ratio up to 6:1 after which the yield decreases with excessive temperature.

6. Result and Discussion

Taking the biodiesel yield as the response of the designed experiment, the data obtained were statistically analysed to get a suitable model for optimization of biodiesel yield as a function of the five independent process parameters. The optimization produced 30 feasible solutions whose desirability equals to 1 and the selected condition (most desirable) was found to be: reaction time (3 hrs), reaction temperature (58°C), stir speed (305.5 rpm), catalyst concentration (1.4 wt%) and methanol to oil ratio (6:1), the optimum yield of biodiesel for this condition was found to be 91.6%.

The developed model was tested and validated for adequacy by substituting random values (from Table 3) as input parameters and the output parameters from the developed model were close to the observed values from Table 3. The regression model was found to be highly significant at 95% confidence level as correlation coefficients for R-Squared (0.9895), adjusted R-Squared (0.9263) and predicted R-Squared (0.9653) was very close to 1 for variables CE, B, C and CD. This is an indication that very small deviation exists between the actual and predicted values. From the coefficient of correlation R (0.9947), the model is highly significant as it explains about 99.47% variance resulting from the interaction of process variables with negligible 0.53% which cannot be correlated. Since there is good agreement among values of correlation coefficient R, R-squared and predicted R-Squared, the selected model is adequate to describe the actual value as a more complicated model is not needed.

7. Conclusion

The following conclusions were drawn after the successful completion of this work:

- i. The results obtained showed that used frying oil can be transesterified to biodiesel which solves oil disposal problems in restaurants and converts waste to wealth.
- ii. A reaction time of 3 hours is sufficient to drive the transesterification reaction to completion with an optimum yield of 91.6%
- iii. Increase in temperature speeds up the rate of transesterification reaction and reduces the reaction time for the conversion of oil to biodiesel. Excessive increase in temperature above the evaporating temperature of methanol will subsequently reduce yield due to gradual evaporation of methanol. Hence, the higher the temperature up to 58°C, the higher the

biodiesel yield, the lower the temperature the lower the biodiesel yield. When the temperature is above 60°C, lower yield of biodiesel is obtained, because of evaporation of methanol.

- iv. The higher the stir speed up to 305.5 rpm, the higher the biodiesel yield, the lower the stir speed, the lower the biodiesel yield. This is because reaction occurs at the interface between the oil and methanol. Excessive increase in stir speed may cause splashing of raw materials due to excessive agitation hence decreasing the biodiesel yield.
- v. The optimum catalyst concentration was 1.4% wt. Soap and gel may be formed when catalyst amount increases beyond the optimum, this prevents ester layer separation.
- vi. The regression model was found to be highly significant at 95% confidence level as correlation coefficients for R-Squared (0.9895), adjusted R-Squared (0.9263) and predicted R-Squared (0.9653) was very close to 1 hence an indication of good correlation and predictive capabilities.

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