FUTA JEET Vol 11 Issues 1&2 December 2017

Journal of Engineering and Engineering Technology

ISSN 1598-0271

School of Engineering and Engineering Technology,
The Federal University of Technology, Akure, Nigeria

FUTA JOURNAL OF ENGINEERING AND ENGINEERING TECHNOLOGY (FUTAJEET Vol 11 Issues 1&2

Journal of Engineering and Engineering Technology

Development of a Model For Predicting the Yield of Biodiesel During Biodiesel Production

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Key words:
Biodiesel,
Central Composite Design,
Optimization,
Transesterification,
Yield.

A B S T R A C T

The production of methyl esters using vegetable oil or animal fat requires selective catalysis and controlled process conditions to meet biodiesel specifications and optimum yield. In this work, a suitable model for the optimization of biodiesel yield as a function of five independent process parameters namely: reaction time, reaction temperature, stir speed, catalyst concentration and methanol-oil ratio was developed using the central composite design and response surface methodology. Alkali transesterification was used for the conversion of palm olein virgin oil to biodiesel with 99.8% pure methanol and sodium hydroxide (NaOH) as catalyst, methanol being in excess to oil ratio (4:1 to 9:1). The designed experiment was carried out using a four-level-five factor central composite design model and response surface methodology to study the interaction of the independent variables; reaction time (1-5 hours), temperature (40-90oC), stir speed (200-400 rpm), catalyst concentration (1-2 wt%) and methanol-oil ratio (4:1 to 9:1) on the biodiesel yield with the response in terms of percentage yield. A predictive model was formulated which correlates the yield of biodiesel to the five process variables. The regression model was found to be highly significant at 95% confidence level as correlation coefficient R (0.985), R-Squared (0.9700), adjusted R-Squared (0.9686) and predicted R-Squared (0.9653) was very close to 1. This is an indication that very small deviation exists between the experimental and predicted values. Results obtained from the model output was in good agreement with experimental values hence the developed model can be employed to predict the yield of biodiesel.

1. Introduction

Biodiesel is a methyl or ethyl ester of fatty acid made from renewable biological resources such as vegetable oil or animal fat (Dermibas, 2010). Transesterification has been widely used to reduce the high viscosity of triglycerides (Meher et al., 2006) and to convert the triglycerides to biodiesel. Transesterification is considered today to be the most efficient process to produce biodiesel (IPTS, 2002) for several reasons including: low temperature (60°C) and pressure (1500 kPa) of the process, high conversion with minimal side reactions and reaction time and reduction in viscosity of the oil produced. Transesterification is defined as the chemical reaction of a vegetable oil or animal fat with short chain alcohol such as methanol or ethanol to produce esters (Collusi et al., 2005). The production of methyl esters using vegetable oil or animal fat requires selective catalysis and controlled process conditions to meet biodiesel specifications. Optimum conditions for biodiesel production strongly depends upon the properties of the raw oil used (Dorado et al., 2004). The reaction requires the presence of catalyst, which depends on the nature of feedstock and produces new chemical compounds called methyl-esters otherwise known as biodiesel (Van Gerpen et al., 2004). Catalysts are employed to speed the exchange of glycerol with methanol resulting in methyl esters of fatty acid (biodiesel). Typically, world biodiesel production is carried out employing methanol (Zhang et al., 2003 (a) & (b); West et al., 2008; Helwani et al., 2009; Apostolakou et al., 2009; Pokoo-Aikins et al., 2009; Diaz et al., 2009; Glisic and Skala 2009; Al-Zuhair et al., 2010). Methanol is preferred for rapid reaction rates (Zhang et al., 2009). Bello et al. (2013) carried out transesterification on used and unused oil using acid catalyzed and base catalyzed transesterification process respectively. The high free fatty of the used oil inhibits acid transesterification, this resulted in lower yield (60%) compared to the higher yield (80%) obtained from alkali transesterification. This was the reason for increasing the concentration of hydrogen chloride to oil weight

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so as to facilitate quick exchange of glycerol with methanols. The presence of FFA and other impurities likely to form soap is the main reason for increasing the temperature of acid transesterification compared to alkali transesterification. Also, the molar ratio of alcohol to oil increased significantly so as to drive the reaction to completion.

The most important considerations within a reactor are the extent of reaction of the reactants, which is known as conversion and the selectivity of the reaction to the desired products. According to Van Gerpen et al. (2004), key reactor variables that dictate conversion and selectivity are temperature, pressure, reaction time (residence time) and degree of mixing. In general, increasing the reaction temperature increases the reaction rate and hence, the conversion for a given reaction time. For batch reactor and CSTRs the degree of mixing is directly related to the amount of energy introduced through the impeller. It was reported that increasing the speed of the impeller, which increases the energy input to the reactor, increases the degree of mixing, which improves the performance of the reactor. This work therefore looks at the optimization of biodiesel yield via the use of a predictive model.

2. Experimental Design

The physical experiment was carried out at Afe Babalola University, Ado Ekiti, Nigeria. Test for the presence of free fatty acid (FFA) was carried out on the oil as the FFA content should not be more than 1% by mass, so as prevent saponification and hydrolysis which can reduce biodiesel yield (Daramola et al., 2015; Muazu et al., 2013; Mathiyazhagan and Ganapathi 2011; Liu 1994; Mittelbach et al., 1994; Freedman et al., 1984; Bello et al., 2016). The amount of free fatty acid was determined from equation 1

$$FFA = \frac{B-A}{W} N(28.2) \qquad (1)$$

where;

B is the blank (ml), A is the titre value of fuel sample (ml), W is the weight of sample (g) and N is normality

Alkali transesterification was used for the conversion of palm olein virgin oil to biodiesel with 99.8% pure methanol and sodium hydroxide (NaOH) as catalyst, methanol being in excess to oil ratio (4:1 to 9:1) NaOH at concentrations ranging between 1-2 wt.% was first dissolved as a catalyst in methanol and mixed in the mixing tank for 30 minutes to form sodium methoxide. Pre-heated oil was transferred into the reactor where sodium methoxide from mixing tank was added to it. Continuous mixing of the mixture was done in the reactor at temperatures ranging between 40-90^{o}C and stir speed ranging between 200-400 rpm for 1-5 hours. This ensured the reaction was driven to completion with phase separation into two layers: the top biodiesel and the bottom glycerol.

The methyl ester formed was washed with water in a volume ratio of 3:1 to remove other impurities after which it was dried by passing it through anhydrous sodium sulphate (Na2SO4). Both the dried biodiesel and glycerol was then transferred to the respective storage tanks for storage. Optimization of the process variable and prediction of biodiesel yield was carried out with Design-Expert® (version 7) software 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 (1-5 hours), temperature (40-90^{o}C), stir speed (200-400 rpm), catalyst concentration (1-2 wt.%) and methanol-oil ratio (4:1 to 9:1) on the biodiesel yield with the response in terms of percentage yield (Table 1).

A mathematical model was formulated from the central composite design using responses (i.e. Biodiesel yield) and the process variables which correlates the two set of variables through first and second order as well as interactive terms according to the second order polynomial (Equation 2)

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i x_i + \sum_{i>j}^{k} \sum_{j}^{k} \beta_{ij} x_i x_j \qquad (2)$$

where,

Y is the response variable (Biodiesel Yield), βi linear regression coefficient, βii is the quadratic coefficient, βij is the interactive effect coefficient, βo is the regression coefficient, xi,xj is the independent process variables and k is the number of factors studied and optimized in the experiment.

The average classification error was used to determine the degree of agreement between the actual and predicted values from the central composite design. The root mean square error for the actual and the predicted value of the biodiesel yield is given by equation 3

$$E_{r.m.s} = \sqrt{\frac{\sum_{i=n}^{n} (Actual - Predicted)^2}{n}} \qquad (3)$$

.

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	℃	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

The root mean square value for the biodiesel yield is given by equation 4

$$A_{r.m.s} = \sqrt{\frac{\sum_{i=n}^{n}(Actual)^2}{n}} \quad (4)$$

The ratio of the two values given in equations 3 and 4 gives the average classification error given in equation 5.

$$E_C = \frac{E_{r.m.s}}{A_{r.m.s}} \quad (5)$$

A predictive model for estimating the biodiesel yield in terms of the process parameters is given in equation 6.

$$Yield = 78.00 + 6.59 * A + 1.34 * B - 0.10 * C + 14.36*D - 2.56*E - 5.10*A*B + 4.49*A*C - 12.83*A*D + 4.71*A*E - 3.96*B*C + 2.18*B*D - 7.19*B*E \quad (6)$$

where; A denotes the reaction time (hours), B is the reaction temperature (°C), C is the stir speed (rpm), D is the catalyst concentration (wt.%) and E is the methanol-oil ratio.

Table 2 compares the percentage yield from experimental response to the percentage yield from values predicted by the model using central composite design and response surface methodology. The percentage optimum yield of biodiesel was found to be 91.6117 at a reaction time of 3 hours, reaction temperature of 58°C, stir speed of 305.5 rpm, catalyst concentration of 1.4 wt.% and methanol-oil ratio of 5.36.

The validation of the predictive model from the central composite design was done using arbitrary values within the range of process parameters given in Table 3. The biodiesel yield from the experimental response and the predictive model were found to be in agreement.

Table 2: Experimental and Predicted Yield of Biodiesel Using Central Composite Design

Run	Factor 1 A:Time (Hours)	Factor 2 B:Temperature (°C)	Factor 3 C:Stir speed (rpm)	Factor 4 D:Catalyst concentration (wt. %)	Factor 5 E: Methanol to Oil ratio	Yield (%) from Experimental response	Yield (%) from Predicted response	% Error
1.	1	40.00	400.00	2.00	9.00	88.80	87.9998	0.8002
2.	1	40.00	400.00	2.00	4.00	89.90	90.2999	0.3999
3.	5	40.00	200.00	1.00	9.00	90.00	91.6002	1.6002
4.	5	90.00	400.00	1.00	4.00	81.50	82.3999	0.8999
5.	**3**	**58.03**	**305.50**	**1.40**	**5.36**	**91.09**	**91.6117**	**0.5217**
6.	1	90.00	400.00	2.00	9.00	84.20	85.3410	1.1410
7.	1	40.00	200.00	2.00	9.00	91.00	89.6076	1.3924
8.	5	40.00	200.00	2.00	4.00	70.00	71.4980	1.4980
9.	5	90.00	200.00	2.00	9.00	78.00	79.9784	1.9784
10.	5	40.00	400.00	2.00	4.00	86.70	86.5012	0.1988
11.	1	40.00	200.00	2.00	4.00	90.20	91.6000	1.4000
12.	3	65.00	300.00	1.50	6.50	78.00	79.2310	1.2310
13.	5	90.00	400.00	2.00	4.00	90.00	88.9001	0.0999
14.	5	90.00	200.00	1.00	4.00	82.80	81.6231	1.1769
15.	5	90.00	400.00	2.00	9.00	81.00	82.0011	1.0011

Table 3:: Experimental and Predicted Yield of Biodiesel Using Central Composite Design

Run	Factor 1: A	Factor 2: B	Factor 3: C	Factor 4: D	Factor 5: E	Yield (%) from Experimental response	Yield (%) from Predicted response
1.	1	50.00	400.00	1.20	8.00	89.80	87.9998
2.	2	40.00	300.00	2.00	5.00	88.90	90.2999
3.	5	40.00	250.00	1.00	7.00	90.06	91.6002
4.	4	60.00	200.00	1.60	4.00	80.50	82.3999
5.	3	55.00	320.00	1.40	5.00	91.01	91.6117
6.	3	90.00	400.00	1.80	6.00	86.20	85.3410
7.	1	40.00	220.00	2.00	6.00	91.09	89.6076
8.	5	45.00	200.00	2.00	4.00	72.00	71.4980
9.	4	65.00	300.00	1.30	8.00	78.01	79.9784
10.	5	75.00	400.00	2.00	8.00	85.70	86.5012
11.	1	70.00	280.00	1.70	4.50	90.26	91.6000
12.	2	60.00	350.00	1.50	6.50	78.01	79.2310
13.	5	70.00	300.00	1.00	7.00	90.20	88.9001
14.	3	45.00	200.00	2.00	6.00	82.65	81.6231
15.	1	50.00	400.00	1.00	8.00	81.04	82.0011

From Table 3, the percentage optimum yield of biodiesel was found to be 91.6117. The root mean square error for the actual and predicted values was calculated as 4.829 from equation 3, while the root mean square value for the experimental values was found to be 329.31 from equation 4. The average classification error was also calculated as 0.0146 from equation 5.

Analysis of variance (ANOVA) was also used to further test and validate the model developed using response surface methodology for adequacy and the regression model was found to be highly significant at 95% confidence level. This is shown in Table 4.

Figure 1 shows the actual and predicted values of biodiesel yields or 15 experimental runs using the response surface methodology. The predicted values have good correlation with the experimental values (Figure 1). There was good agreement between the two, this indicates that the developed model is suitable to describe the process and predict the biodiesel yield based on the input process parameters.

Table 4 Statistical Analysis of Developed Model (Response surface methodology)

S/N	Correlation coefficient R	Predicted R-squared	R-squared	Adjusted R-squared
1	0.985	0.985	0.9700	0.9686

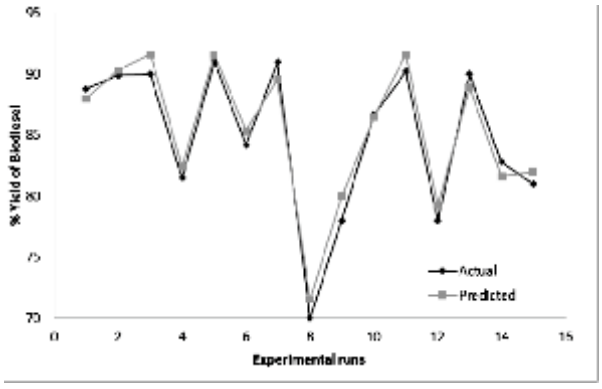

Figure 1: Percentage Yield of Biodiesel for 15 Experimental runs (Response surface methodology)

4. Statistical Analysis of Developed Model

Analysis of variance (ANOVA) was used to test and validate the developed model for adequacy and the regression model was found to be highly significant at 95% confidence level as correlation coefficient R (0.985), R-Squared (0.9895), adjusted R-Squared (0.9263) and predicted R-Squared (0.9653) was very close to 1 (Table 4). This is an indication that very small deviation exists between the actual and predicted values. From the coefficient of correlation R (0.985), the model is highly viable with the correlation of 98.5% resulting from the interaction of process variables with negligible 0.015% 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. The degree of correlation is a measure of correlation coefficient R.

The correlation coefficient is close to 1, meaning that the model is adequately efficient. This also indicates that there is good correlation between the input and output parameters. The developed model correlates and predicts optimum process parameters with high degree of coherence. From Tables 2 and 3, predicted values were very close to the actual values from experiment, thus confirming conclusively that the developed model is a viable correlation and prediction tool for optimizing process parameters for transesterification process.

5. Conclusion

The developed model predicted an optimum yield of biodiesel as 91.6117% for a reaction time of 3 hours, temperature (58oC), stir speed (305.5 rpm), catalyst concentration (1.4 wt.%) and methanol oil ratio (5.36). The developed model fitted to the results from physical experimental with the predictions of the developed model correlating with the experimental results (Table 3). This indicates that the developed model could adequately predict the biodiesel yield within the limits of the process variables investigated. The ANOVA also indicates that the variation in process parameters: reaction time, temperature, stir speed, catalyst concentration, methanol to oil ratio has influence on the yield of biodiesel.

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Journal of Engineering and Engineering Technology

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