

# Assessment of the Transesterification Stage of Biodiesel Production II: Optimisation of Process Variables Using a Box-Behnken Design

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**Abstract** Renewable energy policies, in tandem with favourable market conditions, have culminated in large increases in biodiesel use globally. With increasing volumes of biodiesel and a broader range of suitable feedstocks, including used oils, stabilisation is becoming ever more important. The quality of biodiesel depends on the feedstock, purity of the milled oils, the storage conditions and the transesterification process. In this study, process variables in transesterification were optimised using a Box-Behnken design. The results of linear and non-linear (quadratic) functions and interaction terms (catalyst-FFA (Free fatty acid), stirring-catalyst, FFA-stirring pairs) indicate complex relationships between the set of factors and biodiesel yield. At the 95% significance level, all the linear terms were statistically significant unlike the quadratic term in which only the catalyst was significant. Furthermore, the joint effects of the FFA\*Catalyst and the Stir\*Catalyst interaction terms were statistically significant whereas the interaction between stirring and FFA had no influence on the yield of biodiesel. The yield distributions for varying combinations of the factors show that the optimum factor combinations for a percentage yield greater than 98%

is low ( $-1/0.5$  g) FFA, low ( $-1/400$  rpm) stirring rate and high catalyst amount ( $1/4$  g). Overall, the multivariate model accounted for 86.4% of the total variance in the data.

**Keywords** Biodiesel · Transesterification · Canola · Experiment · Box-Behnken · Optimisation

## Introduction

The production of biodiesel has seen tremendous growth in the last 25 years worldwide especially in the European Union, US and Canada. The biodiesel industry in the US reached a key milestone in 2011 when it crossed the billion-gallon production mark for the first time. This trend was repeated the following year with more than 1.1 billion gallons and a new record was set in 2013 when nearly 1.8 billion gallons was produced, according to statistics from the US Environmental Protection Agency [17]. Since 2005, about 4.6 billion gallons of biodiesel has been produced in the US and this has cumulatively reduced lifecycle greenhouse gas emissions by 25.5 billion kilograms—the equivalent of removing almost 5 million passenger vehicles from the roads in the US. The growth in biodiesel production is understandable given significant policy changes over time and the fact that biodiesel is nontoxic and biodegradable, and it significantly reduces harmful emissions from petroleum diesel such as particulate matter, hydrocarbons and carbon monoxide [17].

In order to optimise biodiesel production and ensure diffusion of biodiesel-based technologies, previous research has sought to ascertain the most favourable feedstock, feedstock catalyst combination and operating conditions [11, 12, 16, 18, 20]. These parameters are dependent on

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the availability of the feedstock, cost of production and the yield of biodiesel obtained [1, 4, 7].

It is discernible from the literature that most studies have focused only on a few factors that affect the yield of biodiesel [18, 21, 23, 24]. Therefore, there are significant gaps in our existing understanding of the full set of parameters that influence the yield of biodiesel and this provides a motivation for this study. Objective of this study is to develop a model for the prediction of yield of biodiesel, alcohol to oil molar ratio, reaction temperature, catalyst concentration and reaction time, based on the experimental data; further the model will be validated with different set of experimental values and surface plots will be generated to explain the trend of achievable yield, under specific combination of process parameters. Ultimately this approach is useful in understanding the influence of process parameters and the resulting output parameters.

This paper is the second in a series of studies in which the assessment of the process of transesterification was carried out using Canola oil as feedstock. In a complementary paper, the process variables in the transesterification stage of biodiesel production were selected within the Plackett-Burman experimental design [18]. In the present study however, the process variables were optimised using a Box-Behnken design. We contribute to the literature by taking most of the parameters that systematically vary with biodiesel yields into consideration in this study.

Transesterification is a defining characteristic of biodiesel production. In the transesterification reaction, triacylglycerol (TAG) is reacted with an alcohol (either methanol or ethanol), in the presence of a catalyst (base or acid) to produce glycerol and fatty acid alkyl esters [2, 14]. The entire biodiesel production process is summarized by this reaction and hence various reaction parameters are monitored to ensure that maximum yield (and/or conversion) and purity is achieved. These include the alcohol to oil molar ratio, reaction temperature, catalyst concentration and reaction time [12].

Various experiments have been conducted that propose the optimum values for the parameters stated above. These parameters (60 °C reaction temperature and 6:1 methanol: oil molar ratio, 1 wt% catalyst) have become a standard for methanol-based transesterification [9]. Some reports also state that the transesterification reaction is more dependent on the alcohol to oil ratio, catalyst concentration and reaction time than on temperature [8, 10].

From previous analysis done in our laboratory using the Plackett-Burman design [18], it was determined that within some specified intervals, the FFA amount, the rate of stirring and the amount of catalyst affect the yield of biodiesel more than the other factors [6]. Studied the effect of methanol-oil molar ratio on yields from 1:1 to 6:1 molar ratios. It was found that the maximum conversion (98%)

was obtained at 6:1. They also observed that depending on the alcohol used, reactions at 60 °C to 100 °C resulted in 80–99% conversions in 2–60 min. [5, 6] also found that reaction is only highly affected by temperature in the initial stages of reaction. These observations are consistent with the findings of our previous work, hence the temperature, reaction time and methanol-oil ratio were kept constant at 60 °C, 1 h and 6:1, respectively [19, 23].

## Materials and Method

The Box-Behnken method was used for the present study. It is a spherical design with all the points lying on a sphere of radius  $\sqrt{2}$ . This design does not contain any points at the vertices of the cubic region created by the upper and the lower limits of each variable [15]. Box-Behnken are incomplete three-level factorial designs and are constructed by merging two-level factorial designs with incomplete block designs in a specific way [3]. Its fundamental utility resides in its capacity to limit the sample size as the number of parameters increases. The sample size is maintained at a value which is appropriate for the estimation of the coefficients in a second degree least squares approximating polynomial [3]. In this design, a block of samples conforming to a two-level factorial design is recurrent over different sets of parameters. According to [3], the parameters which are excluded from the factorial design persist at their mean level throughout the block. The type (full or fractional), the size of the factorial, and the number of blocks which are evaluated, depend on the number of parameters and it is chosen so that the design meets, exactly or approximately, the criterion of rotatability [3]. In this context, the experimental design is said to be rotatable if the variance of the predicted response at any point depends only on the distance from the central point. The typical justification for employing the Box-Behnken design is to evade the situation where the corner points in the central composite design are very extreme, i.e. they are at the highest level of several factors [15]. The Box Behnken may look a lot more desirable since there are more points in the middle of the range and they are not as extreme. In this study, this design was preferred to the central composite design because the axial and corner points were thought to be extreme and that such high levels were not typical. This in effect reduced the number of runs for the Box-Behnken design [26].

Canola oil (certified organic) with FFA concentration of less than 0.10% was obtained from Sigma-Aldrich. Anhydrous potassium carbonate (min. 99%) supplied by Caledon Laboratories was obtained from the chemical store. Anhydrous grade methanol (99.8%) and technical grade linoleic acid (about 65%) were also purchased from Sigma-Aldrich. Analytical grade methyl nonadecanoate ( $\geq 98.0\%$ ) and

toluene were obtained from Sigma Aldrich for the GC analysis [24].

### Experimental Set-up and Design

The experimental set-up is described in detail in [18]. Exactly 100 g of canola oil was measured out and poured into the reactor. The reaction parameters were modified for each run from the design. The design is as shown in Table 1. For a reaction time of 1 h, the temperature was kept constant at 60 °C and methanol-oil molar ratio at 6:1. The -1 (s), 0 (s) and 1 (s) are coded variables used to denote the high, center and low points of each variable. The actual values are given in Table 2 below. The values used for each of the factors were based on findings from the Plackett-Burman (PB) design. Also, [25] have noted that transesterification would not occur if the FFA amount in the oil was over 2%. This explained the low yields of biodiesel that were obtained in the PB design with FFA amounts of 6% wt of oil [18]. Hence, the FFA amount was decreased to 2% and used for the Box-Behnken design.

The amounts to use for each run was calculated (for 100 g of oil) based on how the reaction proceeds. The reaction was reduced to as follows;



**Table 1** Factor calculations

Notation	Factors	Low (-1)	Center (0)	High (+1)
A	FFA amount (wt% oil)	0.5	1.25	2
B	Stirring speed (rpm)	400	600	800
C	Catalyst amount (wt% oil)	1	2.5	4

**Table 2** Results (in % yield) from the Box-Behnken design experiment

Standard order	FFA/wt%	Stir/rpm	Catalyst/wt%	Average yield	Standard deviation	Confidence interval
1	0.50	400	2.5	92.9	0.1	92.7–93.1
2	2.00	400	2.5	86.3	0.7	85.3–87.3
3	0.50	800	2.5	94.2	0.6	93.4–95.0
4	2.00	800	2.5	90.4	0.3	90.1–90.8
5	0.50	600	1.0	82.3	1.4	80.3–84.2
6	2.00	600	1.0	0.0	0.0	
7	0.50	600	4.0	97.5	0.1	97.3–97.6
8	2.00	600	4.0	93.2	0.0	93.1–93.2
9	1.25	400	1.0	1.5	0.1	1.4–1.6
10	1.25	800	1.0	71.8	2.7	68.1–75.5
11	1.25	400	4.0	94.6	1.4	92.7–96.5
12	1.25	800	4.0	95.3	0.8	94.2–96.3
13	1.25	600	2.5	88.4	1.7	86.0–90.8
14	1.25	600	2.5	88.3	0.5	87.6–89.1

where TAG and FAME are triacylglycerol and fatty acid methyl ester, respectively. The 6:1 methanol–oil ratio was calculated to be 21.762 g.

Two replicates of each run were done and the corresponding yield determined. The runs were also randomised to remove any correlations that may be present. At the end of each run, the mixture was transferred into a separating funnel and then left to settle (by gravity) overnight. The resulting layers were then decanted into separate bottles for analysis.

### Gas Chromatography Analysis

The yield determination was done using gas chromatography. The method used is outlined in EN 14103.

Exactly 100 mg of each sample was measured using a high precision balance and put into a 12 mL vial. This was followed by the addition of 100 mg of internal standard (C19) to each sample. The masses in each case were recorded to the nearest 0.1 mg. The sample and standard were then dissolved in 10 mL of toluene. The resulting mixture was allowed to settle; it was then mixed thoroughly for 15 min after which three samples were drawn from each vial for GC analysis.

The yield of FAME is computed as:

$$C = \frac{\sum A - A_{IS}}{A_{IS}} \times \frac{C_{IS} \times V_{IS}}{m} \times 100\%$$

where  $\sum A$  is the total peak area from C14 to C24,  $A_{IS}$  is the internal standard peak area,  $C_{IS}$  is the concentration of internal standard in mg/mL,  $V_{IS}$  is the volume of internal standard in mL,  $m$  is the mass of sample in mg.

Prior to this step, a FAME mixture (C8–C24) obtained from Agilent Canada was run and the resulting retention times used to aid in identification of the methyl esters.

## Results and Discussion

In this study, process variables in transesterification were optimised using a Box-Behnken design. The results (in % yield) from the Box-Behnken design are summarised in Table 2. Three factors were analysed for a base run of 14. For two replicates of each run, 28 runs were considered in total (2 center points). The average values have been calculated for each yield and the corresponding standard deviation and confidence intervals also calculated.

The assumption of normality was checked with the residual plots generated in Minitab. Normality requires that the data have zero mean and constant variance. This is necessary in order to apply the hypothesis tests. It can be seen from Table 3 that the data met this assumption.

The analysis of variance table, also generated in Minitab, is shown below. At the chosen level of significance, the high F value ( $F_{\text{model}} = 20.01$ ) and the very low probability value ( $P < 0.0001$ ) indicates the high significance of the fitted model. The same can be said for the linear, quadratic and interaction terms. At 95% significance level, all the linear terms were significant but only the catalyst quadratic term was significant. It can also be seen that the FFA\*Catalyst and the Stir\*Catalyst interaction terms were significant while that between stirring and FFA was not significant. All the terms are further discussed below. The coefficient of determination ( $R^2$ ) was found to be 90.91%

( $R\text{-Sq.}(\text{adj}) = 86.37\%$ ), indicating that the fitted model accounted for 86.37% of the total variation of the process and that only 13.63% was not explained by the model when all the factors in the model have been accounted for.

From the estimation of effects (Table 4), increasing the amount of FFA has a high negative effect on the yield of biodiesel. From the ANOVA table, it can also be seen that there is a significant linear relationship between the yield and the FFA amount ( $F = 16.49$ ,  $P = 0.01$ ) but there is no quadratic relationship between the two ( $F = 0.29$ ,  $P = 0.60$ ) at the 95% level of significance.

The effect of FFA amounts on biodiesel yields is well documented and known. [22], mentions that increased FFAs react with the basic catalyst added for

**Table 4** Estimated coefficients and effects for yield

Term	Coefficient	SE COEF	T	P
Constant	88.370	5.964	14.818	0.000
Linear				
FFA	-12.109	2.982	-4.061	0.001
STIR	9.550	2.982	3.203	0.005
CATALYST	28.122	2.982	9.431	0.000
Quadratics				
FFA	2.520	4.715	0.535	0.600
STIR	0.074	4.715	0.016	0.988
CATALYST	-22.664	4.715	-4.807	0.000
Interactions				
FFA*STIR	0.701	4.217	0.166	0.870
FFA*CATALYST	19.487	4.217	4.621	0.000
STIR*CATALYST	-17.387	4.217	-4.123	0.001

**Table 3** Analysis of variance for yield

Source	DF	SEQ SS	ADJ SS	ADJ MS	F	P
Regression	9	25618.9	25618.9	2846.5	20.01	0.000
Linear	3	16458.9	16458.9	5486.3	38.56	0.000
FFA	1	2346.1	2346.1	2346.1	16.49	0.001
STIR	1	1459.3	1459.3	1459.3	10.26	0.005
CATALYST	1	12653.5	12653.5	12653.5	88.94	0.000
Square	3	3699.6	3699.6	1233.2	8.67	0.001
FFA*FFA	1	270.9	40.6	40.6	0.29	0.600
STIR*STIR	1	141.5	0.0	0.0	0.00	0.988
CATALYST*CATALYST	1	3287.3	3287.3	3287.3	23.11	0.000
Interaction	3	5460.3	5460.3	1820.1	12.79	0.000
FFA*STIR	1	3.9	3.9	3.9	0.03	0.870
FFA*CATALYST	1	3037.8	3037.8	3037.8	21.35	0.000
STIR*CATALYST	1	2418.5	2418.5	2418.5	17.00	0.001
Residual error	18	2560.8	2560.8	142.3		
Lack-of-fit	3	2544.8	2544.8	848.3	798.27	0.000
Pure error	15	15.9	15.9	1.1		
Total	27	28179.7				

transesterification resulting in the formation of soap. As a consequence, one part of the catalyst is neutralised and is therefore not available for the transesterification reaction.

The results also show that increasing the catalyst amount from 1 to 4 wt% oil greatly affects the yield of biodiesel. The catalyst has significant linear ( $F=88.94$ ,  $P<0.001$ ) and quadratic ( $F=23.11$ ,  $P<0.001$ ) terms. It is observed that the quadratic term has a negative coefficient. This indicates that there is a possible point of inflexion after which increasing the amount of catalyst will have a rather negative effect on the yield. This is particularly true since there will be an increase in the rate of the catalyst-FFA side reaction which leads to soap formation.

Stirring has a positive effect on the yield of biodiesel even though it is not as pronounced as the other factors. Ma et al. [13] notes that the effect of stirring speed and time is only pronounced in the first 10 min. After a homogenous mixture is obtained, mixing does not affect the process that much. Stirring only has a linear relationship with yield and a smaller coefficient compared to that of catalyst and FFA amounts. This could explain why increasing the rate of stirring from 400 to 800 rpm does not affect the yield as much as the FFA and catalyst amounts do, even though stirring is necessary to start the reaction [21].

Interactions exist between the catalyst amounts and the rate of stirring and the amount of FFA. Interaction generally implies that the effect on the yield produced by one variable depends on the level of another variable. The FFA\*Stir interaction term is insignificant ( $F=0.03$ ,  $P=0.87$ ). The surface plots (Fig. 1), make it easier to observe the

interaction effects on the yield. In the FFA\*Catalyst plot, it is noticed that if the FFA amount is changed from say 0 to 1, the change in yield at 1 level of catalyst is not as pronounced as that at catalyst levels of  $-1$ . Similar analogy can be drawn for the Stir\*Catalyst surface plot [16].

The contour plots below (Fig. 2) show the yield distributions for varying combinations of the factors. By holding one value constant at its middle, the percentage yield is determined for two factor combinations. From the Catalyst\*FFA plot, the yield increases with increasing catalyst amount for a given FFA amount. Similar deductions can be made from the other plots [11].

Using the surface optimiser in the Minitab® software package, it is found that the optimum factor combinations for a percentage yield greater than 98% is low ( $-1/0.5$  g) FFA, low ( $-1/400$  rpm) stirring rate and high catalyst amount ( $1/4$  g) [20].

## Conclusion

The Box-Behnken method has effectively been used in determining the linear and quadratic relationships between the yield and the three factors considered for the study. It was realised that the amount of FFA and catalyst amounts affected the yield greatly even though they had opposite effects.

Although the Box-Behnken design is almost fully rotatable it contains regions of poor prediction quality, which may be nonetheless useful when the experimenter intends

**Fig. 1** Surface plot of yield against factors

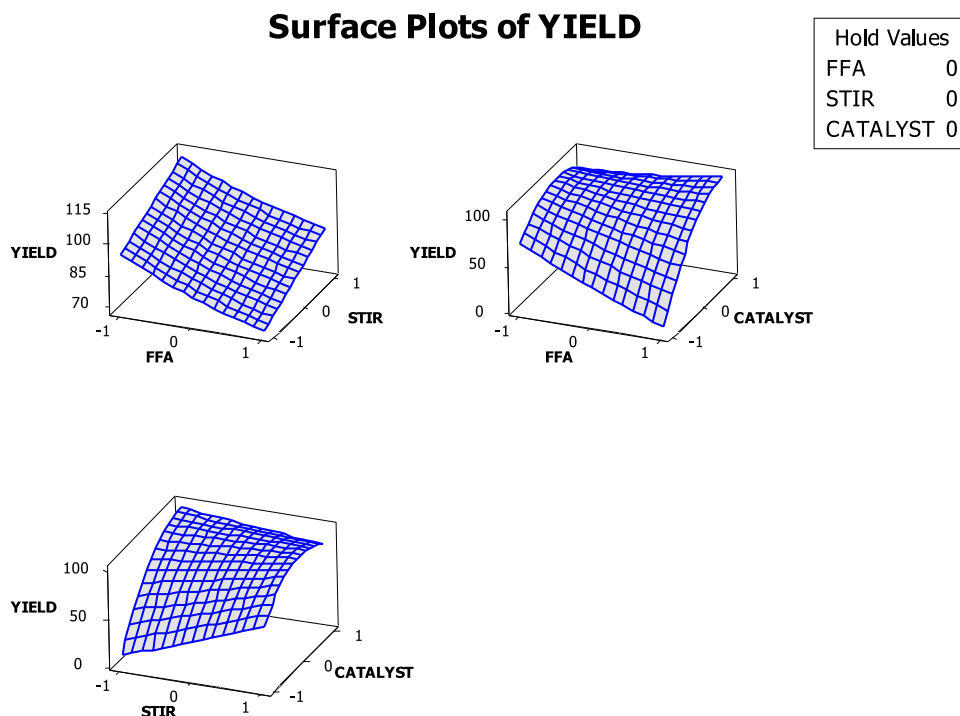
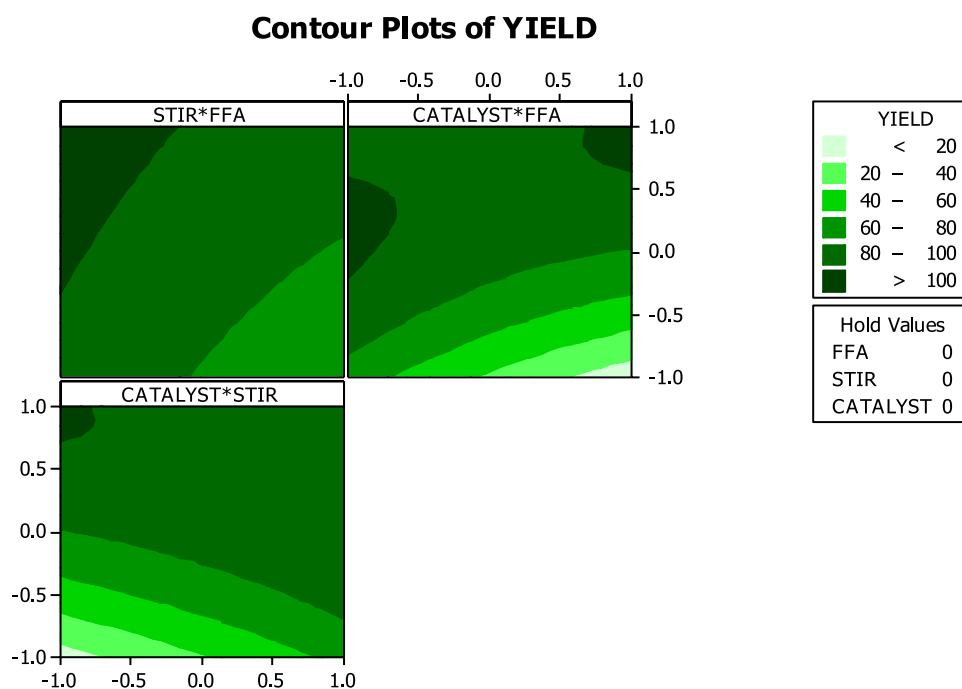


Fig. 2 Contour plot of yield



to avoid combined factor extremes. This property prevents a potential loss of data in those cases. In this study, the response surface models evolved for responses show the effect of each input parameter and its interaction with other parameters, depicting the trend of response. Verification of the Fitness of each model using ANOVA, indicates that all the models can be used with confidence level of 0.95, for navigating the design space. Further validation of the models done with the additional experimental data collected demonstrates that the models have high reliability for adoption within the chosen range of parameters. Surface plots generated show the trend of different responses by varying the 2 input parameters keeping the 3rd parameter constant. Specifically, the contour and surface plots give a graphical representation of the relationship between the factors and the yield. This helps in easily identifying optimum factor combinations for production. Based on the results from the surface optimiser, it is suggested that for optimum yield, the amount of FFA be reduced to the barest minimum; 0.5 g in this case. Also, the stirring can be kept at 400 rpm for the process since it is only needed at the initial stages of the reaction. This also reduces cost that may accompany higher stirring speeds. The optimum catalyst amount was found to be 4 g. It should, however, be noted that increasing the catalyst amount also increases the saponification side reaction as was indicated by the quadratic relationship. With reduced number of experimental runs, fairly convincing, logical and acceptable results have been obtained. There is scope for expanding the frontiers of this study in future research work. In this context, multi-feedstock plants may be investigated by considering segregated and co-fed

raw materials. Also, a detailed kinetic study of the effect of methanol removal on rates of glycerol/biodiesel formation versus reverse reaction to monoglyceride and methanol is warranted. Furthermore, new reaction pathways and processing schemes may be explored by combining experimental and theoretical work to elicit a nuanced understanding of process optimization.

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