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Optimization of Biodiesel Production from Spent Palm Cooking Oil Using Fractional Factorial Design Combined with the Response Surface Methodology

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Abstract: Fractional Factorial Design (FFD) of the experiments combined with the response surface methodology was used to determine the optimum conditions for the production of biodiesel from Used Cooking Oil (UCO) in a batch reactor. Spent palm olein cooking oil was used as a raw material for biodiesel production as it is commonly used in most of the restaurants in Bahrain. The data indicate that the catalyst (NaOH) concentration and reaction temperature are the most influential factors, while the reaction time and molar ratio of methanol to oil (M:O) have only modest effects on the biodiesel yield. A 2^{nd} order polynomial model was used to predict the biodiesel yield as a function of the catalyst amount and temperature. For the production of alkyl esters, the optimum conditions are 50°C, 0.30 wt % of NaOH catalyst, 1h reaction time and 9:1 feed molar ratio. These optimum values agree well with the literature values. At these optimum values, the alkyl esters content is 89.3 vol%, which is in good agreement with the predicted biodiesel yield (~ 91% by volume). Also, the biodiesel produced was characterized based on the flashpoint, viscosity, density, pH, percentage conversion of oil and yield.

Keywords: Biodiesel, Used Cooking Oil, Fractional Factorial Design, Response Surface Methodology, Renewable Energy

Introduction

A fuel like a biodiesel which is mainly derived from animal fats and vegetable oils. It is composed of long chain fatty acids e.g., mono-alkyl esters (Ma and Hanna, 1999; Mamun *et al*., 2013). Because of its biodegradability and non-toxicity, it has great potential as an alternative fuel. In addition, it can be directly used or blended with commonly available petrodiesel for the unmodified diesel engines. As compared to conventionally available petrodiesel, biodiesel has less sulfur, *c.a.* 10-11 wt% of oxygen, high value of cetane number and negligible amount of aromatics (Canakci and Gerpen, 2001). Based on these properties, biodiesel seems to be a good candidate in reducing the particulate matter, the emission of CO_2 and other greenhouse gases into the environment.

The main hurdle in the biodiesel commercialization, obtained primarily from virgin vegetable oils, is its high manufacturing cost. The feedstock costs constitute about 70-90% of the total biodiesel production cost (Connemann and Fischer, 1998). Thus Used Cooking Oil (UCO) or algae oil (Yaakob *et al*., 2013; Arjun *et al*., 2008; Knothe *et al*., 2009; Galadima and Muraza, 2014; Andersson *et al*., 2014; Nautiyal *et al*., 2014), which are cheap or free to use, can be employed as biodiesel feedstock to reduce the biodiesel production cost (Canaki, 2007). Huge amounts of UCOs are available throughout the world particularly in the developing countries such as the Kingdom of Bahrain. A small portion of UCOs is used for the production of soap and main portion of it is either used as a feedstock for animals or simply discharged into the surroundings, which causes environmental issues i.e., land resources

and water contamination. Hence, the utilization of UCOs to produce biodiesel as an alternative for petrodiesel offers significant merits due to the alleviation of environmental pollution as well as providing a method to manage used oils.

The most conventional method used for the biodiesel production is the transesterification process. This process can be carried out using acid (Zhenga *et al*., 2006; Patil *et al*., 2012), base (Hossain and Mazen, 2010; Singh *et al*., 2006; Semwal *et al*., 2011; Muciño *et al*., 2014; Srilatha *et al*., 2012; Tanga *et al*., 2014; Boeya *et al*., 2012; Yan *et al*., 2008; Tang *et al*., 2013) and enzyme (Zhao *et al*., 2015; Yu *et al*., 2013; Haigh *et al*., 2014) catalysts. The long reaction time and the requirement of a high reaction temperature are the main disadvantages of the acid catalyzed transesterification reaction. High cost makes the enzyme catalysts a limited commercial success. In contrast, base catalysts are relatively cheap and requires low temperatures to efficiently promote the transesterification reaction. The parameters which greatly affect the transesterification reaction rate are M:O molar ratio, catalyst amount, reaction time and temperature etc. (Ma and Hanna, 1999; Meher *et al*., 2006; Freedman *et al*., 1984; Ma *et al*., 1998). However, optimum combinations of aforementioned parameters are very important to make biodiesel as a viable alternative over commonly available diesel fuel. Over the past three decades, some studies have been conducted to determine the optimal process parameters to maximize the biodiesel yield (Sahoo and Das, 2009; Math and Irfan, 2007; Antolin *et al*., 2002; Math *et al*., 2011; Zabeti *et al*., 2009). However, in the open literature, trial and error method or response surface methodology alone has been reported to obtain optimal process parameters. This procedure is often not conveniently implemented or is not practical due to the high number of experiments/treatments (Sahoo and Das, 2009; Math and Irfan, 2007; Antolin *et al*., 2002).

In this study, the fractional factorial design in combination with the central composite rotatable design for optimizing the process parameters was used. The fractional factorial design was used to determine the most influential parameters with a minimum number of treatments. Once the most influential parameters were found, optimum values of the variables affecting the process were measured by the application of the central composite rotatable design and response surface analysis. Overall, this approach enables the following: (i) identification of the most influential parameters; (ii) obtaining the knowledge of the relationships between variables which include temperature, reaction time, M:O molar ratio and NaOH amount and the response (yield wt%); and (iii) identification of the best conditions for biodiesel synthesis. Also, the biodiesel produced was characterized using standard analytical techniques.

Materials and Methods

Materials and Apparatus

The raw material employed to produce biodiesel is used cooking oil (palm olein oil). It was received from a restaurant located in the University of Bahrain. A comparison of the physio-chemical properties (ASTM Standard Methods were used to determine properties) of spent palm oil with other used cooking oil is shown in Table 1 (ASTM Standard Methods, 1991). CH_3OH (anhydrous, 99.9 wt %), H_2SO_4 (97.3 wt %), NaOH and C_3H_8O were purchased from Sigma Aldrich. A batch reactor (500 mL) equipped with a controllable heater along with a stirrer was used in this study.

Pre-Treatment of UCO

UCO contains contaminants such as food debris and salts that may cause unnecessary side reactions during biodiesel production. Therefore, pretreatment procedures were used to remove the contaminants prior to biodiesel production. Firstly, UCO was filtered by a fine-screen tissue paper to minimize suspended solid particles and other food waste up to great extent. The oil was then washed by stirring with water (10% of oil volume) at 80°C and 400 rpm followed by the separation of water from the oil using a separating funnel. Secondly, esterification was used to minimize free fatty acids, salts and saponification. The conditions used for the esterification reaction are M:O molar ratio 9:1, 0.35 wt % of sulfuric acid, an operating temperature of 50°C with stirring at 400 rpm for 2 h (Muciño *et al*., 2014). The mixture was then washed with water at 50°C to eliminate alcohol and H_2SO_4 from the oil phase using a separation funnel.

Transesterification Reaction

The pretreated oil was subjected to transesterification in a batch reactor with a working volume of 300 mL. A mixture of NaOH and CH_3OH was then added to the purified oil at the desired temperature (measured using a thermometer placed in the reactor) with stirring at 400 rpm. The reaction conditions were optimized using an appropriate statistical experimental design to maximize production. When the transesterification reaction is completed the NaOH was centrifuged from the product (methyl ester + glycerin) by centrifugation. The vacuum evaporation process was used to evaporate the residual methanol.

Statistical Design of the Experiment

Design of Experiments (DOE) is an important tool to optimize the outputs (either maximize or minimize) based on the process inputs (Montgomery, 2000; Montgomery and Runger, 2006; Box *et al*., 1978).

Table 1. Comparison of physio-chemical properties of spent palm oil used in this study with other used cooking oils

Properties of oil [Country]	Palm oil [Bahrain]	Corn oil [Pakistan] (Zahir *et al*., 2014)	Palm oil [Columbia] (Carlos *et al*., 2011)	Mustard oil [Pakistan] (Zahir *et al*., 2014)	Canola oil [Malaysia] (Ullah *et al*., 2014)
Density (g/mL)	0.92±0.05	0.87±0.03	0.9216	0.87±0.04	0.9±0.05
Kinematic viscosity (centi St)	41±2	33± 2	44.78	40±2	35±2
Flash point (^{o}C)	223±3	140±5	-	170±5	165±3
Saponification value (mg KOH/g oil)	170±5	-	195.87	-	-
Free Fatty Acid, FFA (%)	3±0.3	-	-	-	-

Table 2. The coded levels of the control parameters of the treatments used in the Fractional Factorial Design (FFD)

Treatment	M:O (molar ratio) (A)	Time (h) (B)	Temperature (°C) (C)	Catalyst (wt %) (D)
1	-	-	-	-
2	+	+	-	-
3	+	-	+	-
4	-	+	+	-
5	+	-	-	+
6	-	+	-	+
7	-	-	+	+
8	+	+	+	+

Table 3. Level of each parameter with actual values used in the FFD

Factors	Levels +	Levels -
M:O (molar ratio) (A)	12:01	6:01
Time (h) (B)	2	1
Temperature (°C) (C)	60	45
Catalyst, NaOH (wt %) (D)	1	0.5

In this study, initially, four parameters, namely the NaOH amount, reaction time and temperature and the M:O molar ratio were selected. In order to determine the influential parameters which affect the biodiesel production process, a 2^{k-1} fractional factorial design with resolution IV was used. This was achieved by using a general linear interaction model i.e., Equation 1, that relates the independent factors with their interaction effects (Zahir *et al*., 2014; Carlos *et al*., 2011; Ullah *et al*., 2014):

$$y = \beta_0 + \sum_{i=1}^{N} \beta_i x_i + \sum_{i=1}^{N} \sum_{J=1}^{N} \beta_{ij} x_i x_j \quad (1)$$

Where:

y = Anticipated response
N = Number of parameters
x_i = Coded variables
β_o = Intercept term
β_i = Linear effect
β_{ij} = Interacting effect

The coded levels of the control parameters used in the treatments and the levels of each parameter are shown in Table 2 and 3, respectively.

A Central Composite Design (CCD) with 4 factorial, 4 star (axial) and 5 center points was also implemented to optimize the most influential variables (obtained from FFD) (Zahir *et al*., 2014; Carlos *et al*., 2011; Ullah *et al*., 2014). The axial points distance from a center point is represented by the following relation i.e., $\alpha = 2^{n/4}$ (α = ±1.414 is for two factors). The behavior of the process is described by an equation, Equation 2, that contains all interaction terms despite of their significance:

$$y = \beta_0 + \sum_{i=1}^{N} \beta_i x_i + \sum_{i=1}^{N} \beta_{ii} x_i^2 + \sum_{i<j} \sum \beta_{ij} x_i x_j \quad (2)$$

Where:

x_i = Coded variables
y = Predicted response
β_o = Ntercept term
β_{ij} = interaction effect
β_i = Linear effect
β_{ii} = Squared effect

For the calculations, the equation is used as follows (Zahir *et al*., 2014):

$$x_i = \left(X_i - X_o\right) / \Delta X,\ i = 1,2,3 \ \ N \quad (3)$$

Where:

x_i = Coded value of the true variable X_i
X_o = The center point value of X_i
ΔX = The step change

The experiments were conducted in duplicate. Analysis of variance (ANOVA) was implemented to determine the statistical significance (95% confidence level). The regression equation can be optimized to find the best set of the independent variables. This was achieved either by taking partial derivatives with respect to the x_i's and equating them to zero or graphically, using a prediction profile plot. Minitab (version 14.0) was used for graphical and regression analysis, including developing the prediction profile plot.

Biodiesel Characterization

Several properties e.g., kinematic viscosity, density, flash point, pH and Free Fatty Acids (FFA) were used to

characterize the biodiesel by applying ASTM Standard Methods (1991). Above mentioned properties were measured in duplicate and the average values were obtained for the final results.

Biodiesel production conditions were optimized based on the yield, which was calculated taking into account the purity of the product (Vujicic *et al*., 2010):

$$Yield\ (\%) = \frac{Mass\ of\ ester\ layer\ after\ separation}{Total\ mass\ of\ reactanta\ at\ reaction\ start} \times purity\ (\%) \quad (4)$$

where, purity is the fractional part of esters in the biodiesel and it was determined by using Gas Chromatography-Mass Spectrometry (GC-MS). The analytical results were obtained using a PERKIN-ELMER CLARUS 600°C gas chromatograph equipped with a Mass Selective Detector (MSD) and a capillary column (30 m long ×0.25 mm internal diameter and a layer thickness of 0.25 μm). As a carrier gas (4 mL/min), pure Argon was used. The conditions used in the GC-MS analysis are as follows; column temperature 150°C, detector temperature 280°C, injector temperature 250°C, sample size 1 μL, split ratio (1:50) and a temperature program of 80-240°C at a fixed heating rate of 5°C/min followed by holding the temperature at 240°C for 4 min. The Fatty Acid Methyl Esters (FAME) were identified using a mixture of FAME (chromatographic reference) of known composition.

Results and Discussion

Screening Parameters for Biodiesel Production

Four control parameters, namely the reaction temperature, M:O molar ratio, NaOH amount and reaction time were selected to be optimized for biodiesel production. A Fractional Factorial Design (FFD) was used to design the experiments to determine the most influential parameters among the four. The experiments were based on a 2^{4-1} FFD with resolution IV implemented using MINITAB statistical software. A total of sixteen experiments at different conditions were conducted. The levels and treatments or experimental runs are shown in the experimental section. The effects of different experimental conditions can be better understood by examining the physicochemical properties (e.g., flashpoint, viscosity, density and pH) of biodiesel (Table 4). In addition, the fractional part of esters in the biodiesel was determined by GC-MS technique to calculate the yield, which is shown in Fig. 1.

The values of flash point, viscosity, density and pH of biodiesel obtained using some production conditions are within the acceptable range of biodiesel standard (NREL, 2009), while some production conditions yield biodiesel with values outside the acceptable range. In addition, percentage yield obtained with some conditions are very low compared to others (Table 4).

Figure 2a shows a graphical representation of the magnitude of the main effects of each factor as well as the nature of the effect, i.e., positive or negative. The factors of different levels affect the response differently. It is important to note that the magnitude of the main effect is greater if the line is steeper. The data indicate that the lines associated with the reaction temperature and catalyst concentration are steeper than the others, indicating that they are the most influential parameters for biodiesel production. In addition, the main effects of M:O molar ratio (A) and temperature (C) are positive while time (B) and catalyst concentration (D) are negative, indicating that the treatment with a low level of B and D and a high level of A and C can probably maximize the yield. Figure 2b shows the interactive effects of M:O molar ratio Vs. time and M:O molar ratio vs. temperature. Figure 2c depicts the normal probability plot of the effects. The effects are negligible or less important, which lie along the line. On the other hand, the effects which are away from the line are considered to be important. The important main effects are time (B), temperature (C) and catalyst concentration (D), in which B is less dominant than factors C and D. The important interactions are AB (M:O molar ratio Vs. time) and AC (M:O molar ratio Vs. temperature). From this Figure it is also obvious that the treatment with high level of C (temperature) and low levels of B (reaction time) and D (catalyst concentration) can maximize the yield. Similar results were observed previously in Fig. 2a. Figure 2d represents the residual or error plot for the yield. The normal probability plot is one of the residual plots for assessing whether a data set is uniformly distributed or not. Data points on this plot show linear trend, indicating that the data are normally distributed. The pattern of the histogram also supports these phenomena. The residual Vs. fitted values and residual Vs. order of the data show a random pattern, indicating a good fit for the model. However, these plots do not explain whether the variables and interaction are significant or not. Therefore, an analysis of variance (ANOVA) is required to determine the most significant variables and interactions. As such, ANOVA was performed for 95% confidence interval and is shown in Table 5. The p values for both temperature and NaOH catalyst (wt %) are 0.0, indicating that these two parameters are the most influential parameters in the production of biodiesel. The third most influential parameter is the reaction time ($p = 0.002$). While p value is 0.095 for parameter M:O molar ratio indicating that this factor has an insignificant effect or a very low effect on the yield. The interactive effects of M:O molar ratio Vs. time and M:O molar ratio Vs. temperature are highly significant ($p<0.01$), while M:O molar ratio Vs. catalyst (wt %) is of low significance ($p = 0.064$).

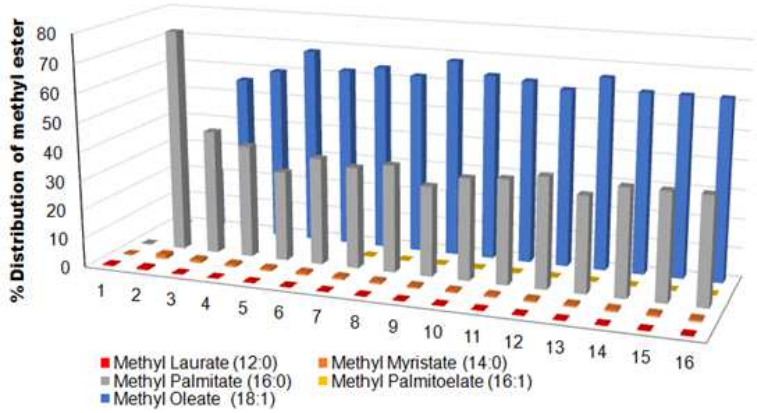

Fig. 1. Percentage distribution of methyl esters resulting from the transesterification of spent palm cooking oil in the sixteen experiments determined by GC-MS analysis

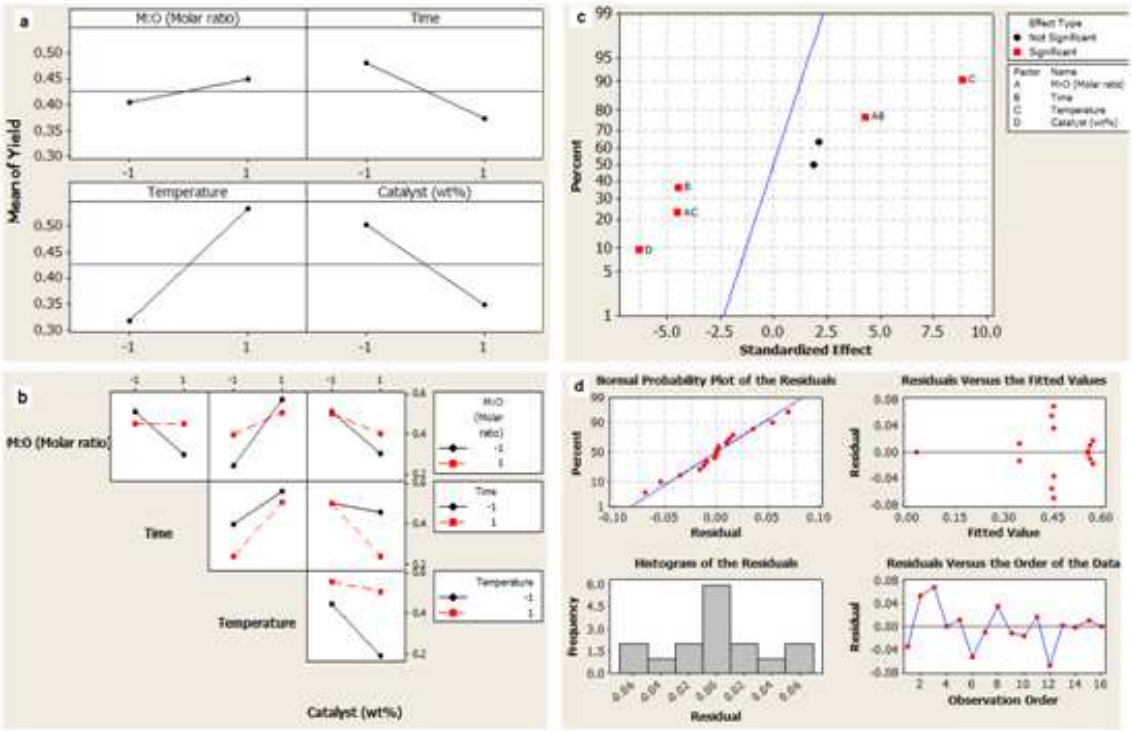

Fig. 2. (a) Main effects plot for the biodiesel yield, (b) Interaction plot for the yield, (c) Normal probability plot for the standardized effects and (d) Residual plots for the yield

Table 4. Properties of biodiesel with the experimental and predicted values of yield (%) obtained from FFD

Treatment	Flash point (°C)	Kinematic viscosity, 40°C (cst)	Density (g/mL)	pH	Experimental yield (%)	Predicted yield (%)
1	150	8.3	0.78	9.3	78.6	74.9
2	170	4.9	0.88	9.6	69.1	74.6
3	-	6.3	0.80	9.6	68.1	75.1
4	170	9.4	0.89	9.3	63.4	64.6
5	170	4.1	0.79	9.4	87.3	86.3
6	181.5	4.1	0.86	9.5	88.8	87.1
7	> 200	5.7	0.86	9.5	85.3	85.5
8	> 200	3.9	0.86	9.4	32.7	32.6
Standard value		> 130	3.5-6	0.87-0.89	> 8	

Table 5. ANOVA of FFD for the linear regression model

Term	Effect	Coef	SE Coef	T	P
Constant		0.42619	0.0123	34.64	0.000
M:O (Molar ratio)	0.04649	0.02325	0.0123	1.89	0.095
Time (h)	-0.1093	-0.0547	0.0123	-4.44	0.002
Temperature	0.21742	0.10871	0.0123	8.84	0.000
Catalyst (wt%)	-0.1551	-0.0776	0.0123	-6.3	0.000
M:O (Molar ratio)*Time	0.10626	0.05313	0.0123	4.32	0.003
M:O (Molar ratio)*Temperature	-0.1113	-0.0557	0.0123	-4.52	0.002
M:O (Molar ratio)*Catalyst (wt%)	0.05276	0.02638	0.0123	2.14	0.064

S = 0.0492109 R-Sq = 95.85% R-Sq(adj) = 92.22%

Table 6. Experimental matrix for the central composite design together with the biodiesel yield

Treatment	Temperature (°C)	Catalyst (wt %)	Coded temperature (C)	Coded catalyst (D)	Experimental yield (%)	Predicted yield (%)
1	45.00	1.00	0.00	0.00	52.5400	45.1600
2	55.00	1.50	1.00	1.00	20.0000	21.2782
3	59.14	1.00	1.41	0.00	44.1908	45.6200
4	45.00	0.29	0.00	-1.41	89.2000	91.1527
5	45.00	1.00	0.00	0.00	42.9480	45.1600
6	45.00	1.00	0.00	0.00	38.0000	45.1607
7	45.00	1.00	0.00	0.00	45.0000	45.1607
8	35.00	0.50	-1.00	-1.00	85.4335	85.9713
9	45.00	1.00	0.00	0.00	47.3121	45.1607
10	55.00	0.50	1.00	-1.00	77.3699	74.6025
11	35.00	1.50	-1.00	1.00	28.7572	33.3406
12	45.00	1.71	0.00	1.41	20.0000	16.2313
13	30.85	1.00	-1.41	0.00	65.4335	62.1884

Table 7. Analysis of Variance (ANOVA) of CCD for the non-linear regression model

Term	Coef	SE Coef	T	P
Constant	45.161	2.17	20.816	0.000
Temperature	-6.554	1.715	-3.821	0.007
Catalyst Amount	-27.680	1.715	-16.138	0.000
Temperature*Temperature	4.545	1.839	2.471	0.043
Catalyst Amount*Catalyst Amount	5.139	1.839	2.794	0.027
Temperature*Catalyst Amount	1.218	2.426	0.502	0.631

S = 4.851 R-Sq = 97.6% R-Sq(adj) = 95.9%

Linear Regression Model and its Validation

The experimental and anticipated values of the biodiesel yield (%) is presented in Table 4. The coefficients of the equation and their values are listed in Table 5. Based on FFD predictions and the results of experiments, the linear regression model for predicting biodiesel yield can be written as:

$$Y = 0.42619 - 0.0547\text{B} + 0.1087C - 0.0776D + 0.05313\text{AB} - 0.0557AC \quad (5)$$

The determination coefficient (R^2) was evaluated to check the model validity. The adjusted R^2 is 92.22%, indicating that the model can explain 92.22% of the variability and the remaining (7.78%) variations cannot be elucidated by the model, which reflect the goodness of fit and confirm the adequacy of the regression model. Adequacy of the model is also indicated by the good concordance between experimental and anticipated values of the response variable, as shown in Table 4.

Second-Order Regression Model and its Validation

As the reaction temperature (C) and catalyst concentration (D) seemed to be the most influential factors (very strongly significant) for biodiesel synthesis, these two factors were optimized further using the CCD, while keeping the reaction time (1 h) and the feed molar ratio, M:O (9:1) constant. A total thirteen experiments at different experimental conditions were conducted for this purpose. Experimental runs were conducted in a fully random order in order to refrain bias. The experimental matrix corresponding to the CCD together with the biodiesel yield (%) are shown in Table 6. Table 7 shows the summary of the ANOVA for non-linear regression, where the coefficients of the equation and their values are listed. The low p values for both NaOH catalyst (wt %) and reaction temperature indicate that these two factors significantly affect the biodiesel yield.

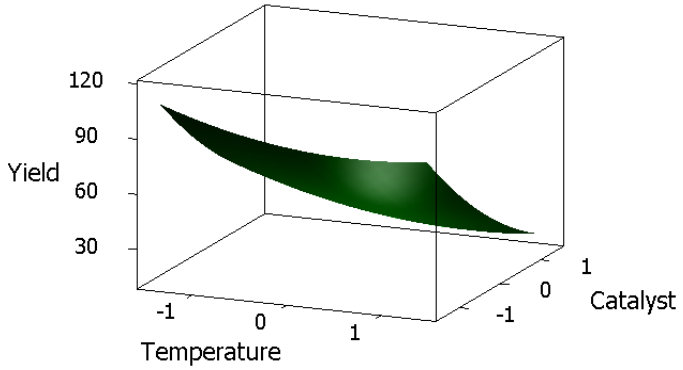

Fig. 3. Surface plot of yield Vs. catalyst amount and temperature

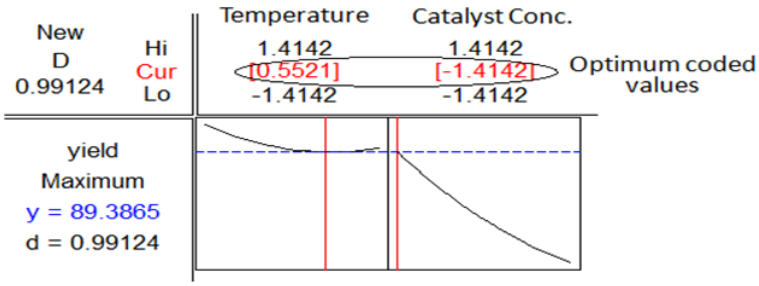

Fig. 4. Response optimization plot

The value of adj. R^2 of the non-linear model is 95.9%, which is greater than that of the linear regression model (adj. R^2 = 92.22%). The results indicate that the second order model fits well with the experimental data.

Multiple regression analysis was used to determine the factors of the non-linear model. From coded levels, expression for the statistical model is as follows:

$$Y = 45.16 - 6.55C - 27.68D + 4.55\ C^2 + 5.14D^2 \quad (6)$$

The results of this model are plotted as a 3-D surface in Fig. 3, which depicts the response (yield) as a function of the two parameters (temperature and NaOH amount). A response optimizer (using MINITAB software) was used to determine the optimum biodiesel production conditions, which are shown in Fig. 4. The optimum coded values for temperature and catalyst concentration are 0.55 and 1.414, which are equivalent to 50°C and 0.30 wt % of the NaOH catalyst, respectively, predicting an optimum yield of around 91%. These values agree well with the literature values, indicating that the optimized biodiesel production method used in this study can successfully produce biodiesel (Math *et al*., 2011; Vicente *et al*., 1998).

Conclusion

A two-step reaction was employed for the production of biodiesel. First, UCO was esterified to reduce the amount of fatty acids to refrain saponification in the consecutive transesterification reaction. The process was optimized by the application of the FFD in combination with the RSM. Reaction temperature and catalyst concentration are the most influential factors, while reaction time and M:O molar ratio have only modest effects on the response. A non-linear model was used to anticipate the conversion levels as a function of NaOH amount and temperature. The model describes the experimental range studied adequately. The process conditions used to predict the optimum yield of ~ 91 wt % are 50°C, 0.30 wt % of NaOH catalyst, reaction time of 1 h and M:O molar ratio of 9:1. These optimum values are well concord with the literature. In addition, the biodiesel produced was characterized based on the flashpoint, viscosity, density, pH, percentage conversion of oil and yield. Thus, the present study provides a simple platform for biodiesel production. As UCO is used as the feedstock, the platform can decrease the biodiesel production cost and aid the disposal of UCO with significant concurrent environmental benefits.

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Author's Contributions

S.M. Zakir Hossain: Formulated the study, wrote the protocol, wrote the first draft, gave answer to reviewer's comments, helped in the revising the manuscript and approved the final version to be submitted.

Nahid Sultana: Managed the literature searches, interpreted the data and approved the final version to be submitted.

Muhammad Faisal Irfan: Helped in literature searches, experimental part and data analyses, gave responses to reviewer's comments, edited the revised version of the manuscript and approved the final version to be submitted.

Elamin Mohammed Ali Elkanzi: Reviewed the whole manuscript critically for significant intellectual content and approved the final version to be submitted.

Yousuf Ahmed Mirza Al-Aali: Managed the experimental process and gathered data and approved the final version to be submitted.

Ahmed Taha: Managed both experimental procedure and data analysis for GC-MS and approved the final version to be submitted.

Sk Manirul Haque: Helped in literature searches and data analysis and approved the final version to be submitted.

Ethics

This article is original and contains unpublished material. The corresponding author confirms that all of the other authors have read and approved the manuscript and no ethical issues involved.

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