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Optimization of Biodiesel Production from Jatropha Oil

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Abstract: The most important factors that influence biodiesel production are temperature, molar ratio, catalyst amount, time and degree of agitation. This study investigated the effects of temperature, molar ratio and degree of agitation and their interactions on the yield and purity of biodiesel produced from Jatropha oil. Factorial design and response surface methodology (RSM) were used to predict yield and purity of biodiesel as functions of the three variables. Interactions of all the factors were found to be significant on both yield and purity responses. Temperature and molar ratio main effects were found to be significant on the yield whereas only temperature main effect was significant on the purity of the biodiesel. The optimum conditions of operations were; temperature of 54 °C, molar ratio of methanol to oil of 6:1 and stirring speed of 660 rpm. Using these conditions, biodiesel yield of 95% (wt) was obtained with a purity of 97%. This model can be used to predict the yield and purity of biodiesel from jatropha oil within the ranges of temperature (30 – 60°C), stirring rate (300 -900 rpm), and molar ratio (3 – 9 mol/mol) studied.

Introduction

There is a growing interest in biodiesel as an alternative diesel fuel due to the diminishing source of petrodiesel and the high cost and environmental problems associated with it. Other factors that contributed to the promotion of biodiesel include the European Union (EU) Directive 2003/30/EC which established a minimum content of 2% and 5.75% of biofuel for all petrol and diesel used in transportation by December 2005 and December 2010 respectively and the EU Directive 2003/96/EC which directed exemptions on excise duties on biofuels so as to promote their use [1]. Also The US Energy Policy Act (EPAct) of 1992 was made to accelerate the use of alternative fuels for transportation. However, the cost of biodiesel production still makes it difficult to compete with petrodiesel since cost of refining the feedstock is high.

Biodiesel, which is defined as monoalkyl ester of long chain fatty acids is produced from vegetable oils and animal fats. The most commonly preferred method of production is transesterification of the oil or fat with an alcohol in the presence of a catalyst. Transesterification reaction is a reversible and consecutive reaction and usually excess alcohol is used for better results. Factors that influence transesterification reaction include; temperature, methanol to oil molar ratio, amount of catalyst, time and the degree of agitation, among others [2-3].

Numerous researches have been done to try and optimize the process conditions of biodiesel production from various oils; Gemma et al. [4-6] studied the methanolysis of sunflower using factorial design and Central Composite Design (CCD) with temperature and initial catalyst concentration as the main factors affecting biodiesel purity and yield. They found out that temperature and catalyst concentration have a positive influence on conversion; Bautista et al. [1]

investigated transesterification of waste cooking oil using factorial design and a CCD having fatty acid concentration, temperature and initial catalyst concentration as the main factors of influence on the purity and yield of biodiesel. They reported that initial catalyst concentration has a positive influence on purity and negative on yield while fatty acid concentration has negative influence on both purity and yield. Park et al. [3] investigated biodiesel production from lard using response surface methodology (RSM) with temperature, catalyst concentration and molar ratio of methanol to oil as the main factors affecting the yield of biodiesel. They reported an optimum condition using these factors for 97.8% yield. Lee et al. [7] studied heterogeneous process and transesterified palm oil using RSM and CCD to determine the effects of temperature, time, molar ratio and catalyst concentration on the yield of Palm Oil Methyl Ester (POME). Optimum conditions for 79.6% yield was reported. Aracil et al. [8] investigated production of biodiesel from brassica carinata using a factorial design and RSM with temperature and catalyst concentration as the major variables affecting the yield of ethyl ester. Temperature and catalyst concentration were found to have significant influence on conversion, Shao et al. [9] transesterified rapeseed soapstock using RSM with a Box and Behenken methodology to determine the effects of molar ratio, time and catalyst concentration on the yield of biodiesel and reported methanol as being more influential.

Although these factors have been much studied, the process conditions are still not well understood. The purity of biodiesel, for example, is affected by changes in the reaction compositions during transesterification and yield can be affected by side reactions like saponification and dissolutions of Fatty Acid Methyl Ester (FAME) in glycerol phase. It is therefore important to get more information on the various process variables that affect transesterification to be able to optimize the operating conditions and control the variables. Simulation allows prediction of the effects of the variables on the responses. In this work, a simulation approach using factorial design and RSM was used to determine the effects of the process variables, namely temperature, degree of stirring and molar ratio and their interactions on the purity and yield of biodiesel produced from Jatropha oil.

Simulation

In the simulation, a 2^{5-1} fractional factorial design was first used as a screening design to get the significant factors. Temperature, stirring rate and molar ratio were found to be significant. A 2^3 full factorial design with four centre points was then used with these significant factors. A curvature was found in the design; therefore it was not sufficient to represent the system. As a result a central composite was developed which could represent the system. The responses (dependent variables) were yield (Y) and purity (P) while independent variables were the temperature (X_1), the stirring rate (X_2) and the molar ratio (X_3). A mathematical model that correlates responses to the process variables through first and second order reaction and interactions was developed to predict yield and purity according to the following quadratic polynomial regression equation:

$$Y/P = B_0 + B_1X_1 + B_2X_2 + B_3X_3 + B_4X_1^2 + B_5X_2^2 + B_6X_3^2 + B_7X_1X_2 + B_8X_1X_3 + B_9X_2X_3. \quad (1)$$

Where: B_0, an intercept; B_1, B_2, B_3, first order coefficients; B_4, B_5, B_6, quadratic coefficients and B_7, B_8, B_9, interactions coefficients for variables X_1, X_2 and X_3 respectively.

The factor levels were selected taking in to account, the properties of the materials to be used in real practical work (Table 1). Temperature (30 – 60℃) implies room temperature up to a temperature below the boiling point of methanol (64.5℃). Stirring speed (300 – 900 rpm) was selected to try to eliminate the mass transfer controlled regime and to allow only the kinetically controlled regime [10-11]. Molar ratio (3 – 9 mol/mol) was followed from the stoichiometry of the reaction; three moles of alcohol reacts with one mole of oil to produce three moles of biodiesel and one mole of glycerine. To push the reaction to the product formation, a higher molar ratio of nine was selected. Time (10 – 120 minutes) was selected as reported in literature. Most of the alkaline catalyzed transesterification reactions are complete either within one or two hours [12-14] and the lower time of 10 minutes was chosen to determine the changes that take place at the initial stages of the reaction as reported in literature [13, 15]. Catalyst amount was selected based on data reported in literature [11-15, 17].

Table 1: Screening design summary showing factor levels

	Real		Coded	
Factor	Low	High	Low	High
Temperature [°C]	30	60	-1	+1
Stirring [rpm]	300	900	-1	+1
Molar ratio [mol/mol]	3	9	-1	+1
Time [min]	10	120	-1	+1
Catalyst Amount [%]	0.5	1.5	-1	+1

The goodness of fit was evaluated using correlation coefficient R^2 and ANOVA. Design Expert Version 6.0.6 Software, (STAT EASE inc. Minneapolis, USA) was used for the regression analysis to fit the quadratic polynomial equation.

Biodiesel yield is defined as the weight percent of the final product after purification, relative to the initial weight of the oil put in the reactor. Purity, on the other hand is defined as the weight concentration of fatty acid methyl ester (FAME) in the biodiesel. It is determined from gas chromatographic peak areas of FAME, relative to the standard peaks whose initial concentrations are known.

Materials and Methods

Materials: Jatropha seeds were obtained from Arusha in Tanzania. The seeds were sorted, dried and mechanically pressed to extract the oil. The obtained crude oil was water degummed and neutralized using calculated amount of analytical grade sodium hydroxide to reduce the free fatty acid (FFA) content before it was used for biodiesel production. All reagents used were of analytical grade.

Experimental procedure: Transesterification reaction was carried out in a 2–litre round bottom flask as a reactor. The reactor was fitted with a reflux condenser (to minimize alcohol loss), a mechanical stirrer, a thermometer and a sampling port. The oil was pre-heated to 110 °C for 30 minutes to drive off water that it might have contained before being charged into the reactor. Calculated amounts of methanol and sodium hydroxide were dissolved separately while the oil was drying. The methanol-sodium hydroxide mix was then transferred into the reactor while stirring started immediately. The reaction was allowed to continue for one hour.

Samples of approximately 15mls were drawn after every 10 minutes and placed in a pre-weighed beaker containing 5mls of tetrahydrofuran (THF) to dilute the system and stop the reaction. A previous study indicated that this treatment stops the reaction effectively. The sample was transferred into a centrifuge and run at 4500 rpm for 20 minutes. The extracted ester was washed four times with warm water (50°C), dried using sodium sulphate anhydrous, filtered, weighed and kept for analysis. Gas Chromatography (GC) analysis was done using EN14142 standard method for the determination of FAME concentrations in the samples.

Statistical Analysis: Factorial design was used to simulate optimum conditions of operations using the regression analysis in Equation (1) and the operating conditions given in Table 1. The effects of main factors on the process and the interactions among them were studied. RSM was applied to optimize the process. The general flow chart used is shown in Figure 1. A 2^3 full factorial and four centre points were added to evaluate experimental error and to study the effects of curvature. Application of this method requires adequate selection of responses, factors and levels.

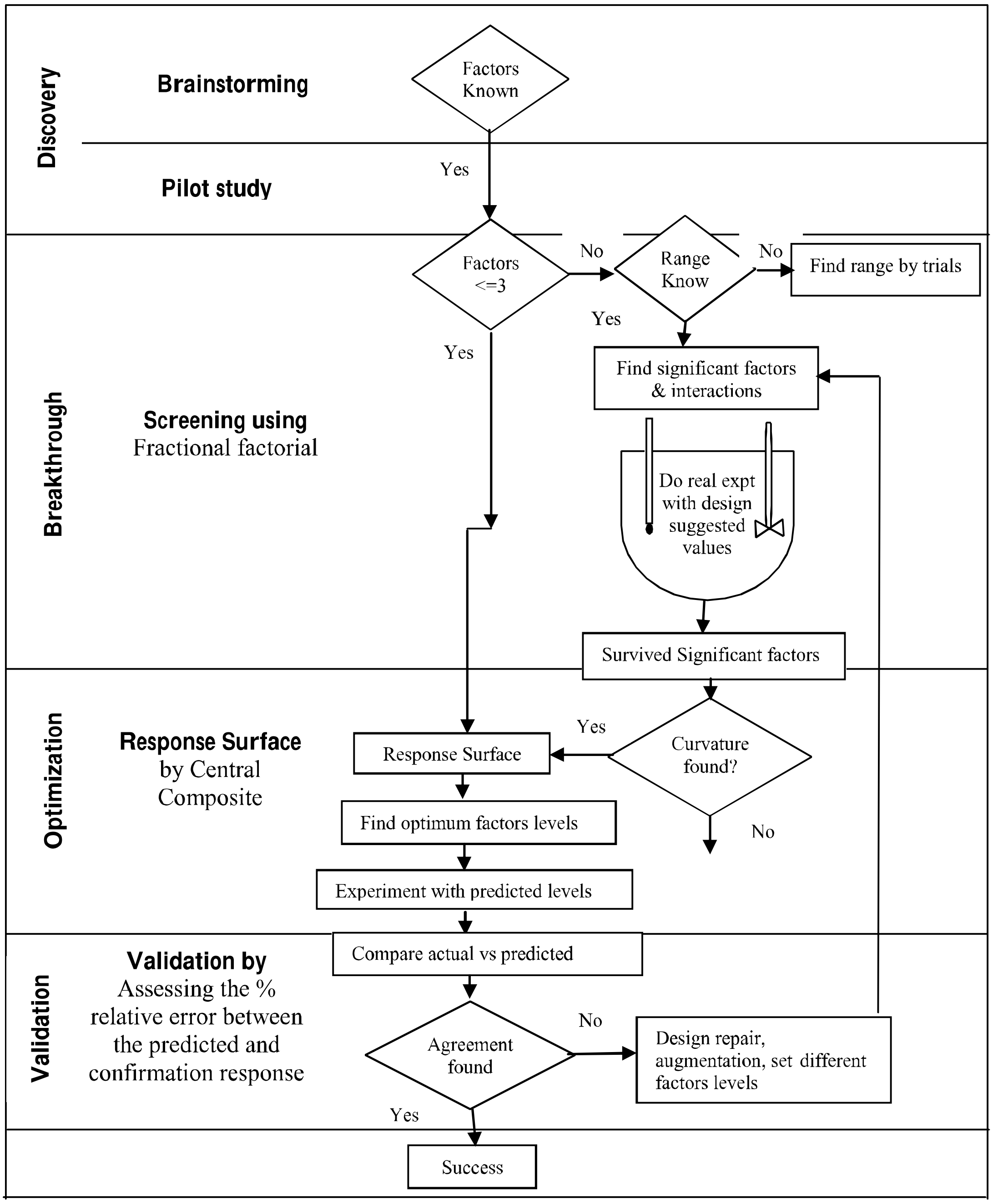


Figure 1: Flow chart for central composite design (adopted and modified from [17])

Results and Discussion

Linear stage: The design parameters of the 2^3 full factorial designs are shown in Table 2 and the matrix in Table 3. Experiments were randomized to minimize unforeseeable errors on the variables.

Table 2: 2^3 Design Summaries

Factor	Name	Units	Actual low	Actual high	Coded low	Coded high
X_1	Temperature	[°C]	30	60	-1	+1
X_2	Stirring	[rpm]	600	900	-1	+1
X_3	Molar ratio	[mol/mol]	4.5	7.5	-1	+1

Table 3: Design Matrix Coded Values

Std	Run	X_1:Temp.	X_2:Stir	X_3:MR	Yield[%]	Purity[%]
1	12	-1	-1	-1	89.0	94.0
2	9	1	-1	-1	94.0	97.0
3	11	-1	1	-1	90.0	95.5
4	8	1	1	-1	97.5	89.0
5	7	-1	-1	1	98.7	87.1
6	5	1	-1	1	97.0	97.5
7	4	-1	1	1	95.0	97.2
8	1	1	1	1	98.0	98.1
9	10	0	0	0	98.0	96.0
10	3	0	0	0	97.0	95.3
11	2	0	0	0	97.6	98.1
12	6	0	0	0	96.8	98.5

MR = molar ratio

Temperature and molar ratio main effects were significant on yield while only temperature main effect was significant on biodiesel purity. All the interaction effects were significant on both yield and purity responses. Experimental results were fitted to a linear model and the equations obtained were as follow:

Statistical models (obtained from coded factor levels):

$$\text{Yield} = +94.90 + 1.72X_1 + 0.22X_2 + 2.27\,X_3 + 0.90X_1X_2 - 1.40\,X_1X_3 - 0.90X_2X_3. \quad (2)$$

$$\text{Purity} = +94.38 + 1.00X_1 + 0.50X_2 + 0.50X_3 - 2.37X1X_2 + 1.88X_1X_3 + 2.13X_2X_3. \quad (3)$$

Industrial Models (obtained from real factor levels):

$$\text{Yield} = +58.20 + 0.188T + 7.50E\text{-}003ST + 7.317MR + 4.0E\text{-}004TST - 0.06TMR - 4.0E\text{-}003STM. \quad (4)$$

$$\text{Purity} = +116.25 + 0.36T - 5.83E\text{-}003ST - 10.50MR - 1.06E\text{-}003TST + 0.08TMR + 9.44E\text{-}003STMR \quad (5)$$

Statistical models were obtained from the coded factor levels and Industrial models from the real factor values. Equations (2 – 5) are only valid within the experimental range considered. The obtained R^2 for both models are 0.98 for yield and 0.93 for purity.

The statistical significance of curvature was studied to determine whether the factorial design was sufficient to describe the transesterification of Jatropha oil. The curvature effect is defined as the difference between centre point mean responses and factorial mean responses and the values were

2.47 for yield response and 2.54 for purity response respectively. For a 95% confidence level, the curvature confidence intervals were ±1.17 and ±2.85 for yield and purity responses respectively. Curvature effect was significant for yield although for purity it was not, there was need to use a more complex design, a central composite rotatable design was chosen for this purpose.

Non- Linear Stage: Due to the significance of curvature found in the linear model, a second order model was required according to RSM. Six star points were added on to the eight factorial and four centre point design of Table 3, making it a Central Composite Rotatable Design (CCRD). The star points are encoded as $+\alpha$ (1.682) and $-\alpha$ (-1.682) as shown in Table 4 columns two - four. Alpha (α) is the distance from the centre point to the star point and its value is given by $2^{n/4}$, where n is the number of factors. The model is a complete quadratic surface between responses and factors.

Table 4: Six star points

Run	X_1	X_2	X_3	Stir	Temp	MR	Yield[%]	Purity[%]
13	-1.682	0	0	600	45	6	96	98
14	1.682	0	0	900	45	6	96	98
15	0	-1.682	0	750	30	6	90	90
16	0	1.682	0	750	60	6	94	98
17	0	0	-1.682	750	45	4.5	93	96
18	0	0	1.682	750	45	7.5	96	98

The coefficients were obtained by multiple regression analysis which included all the independent variables and their interactions. The best-fit response surfaces are given by the following equations:

Statistical models:

$$\text{Yield}=90.00 - 0.25 X_1 + 0.83X_2 + 1.36X_3 + 2.10X_1^2 + 0.69X_2^2 + 1.57 X_3^2 – 1.68 X_1X_2 + 0.95X_1X_3 + 0.19X_2X_3. \quad (6)$$

$$\text{Purity}=92.27 + 0.21X_1 + 2.22X_2 + 1.06X_3 + 1.84X_1^2 + 0.52X_2^2 + 1.57X_3^2 – 0.64X_1X_2 - 0.61X_1X_3 - 0.51X_2X_3. \quad (7)$$

Industrial models:

$$\text{Yield}=304.95 - 0.38ST + 0.75T - 32.20MR + 2.64E\text{-}004ST^2 + 8.64E\text{-}003T^2 + 1.97MR^2 - 2.11E\text{-}003STT + 0.01STMR + +0.02TMR. \quad (8)$$

$$\text{Purity}=207.78 - 0.26ST + 0.65T - 13.89MR + 2.31E\text{-}004ST^2 + 6.49E\text{-}003T^2 + 1.98MR^2 - 7.98E\text{-}004STT – - 7.68E\text{-}003STMR - 0.06TMR. \quad (9)$$

The statistical model is obtained from encoded levels giving the real influence of each variable on the process and industrial model is obtained from the real values of the variables. Equations (6 – 9) describe the influence of temperature, stirring rate and molar ratio on the yield and purity of biodiesel within the studied experimental ranges. The obtained R^2 for both models are 0.91 for yield and 0.87 for purity.

For each response, the second order models can be plotted in three response surface plots as a function of two of the three factors at the centre point value of the operating condition of the third factor, Figure 2 shows the response surface graph of biodiesel yield predicted for the experimental range of temperature and stirring rate at molar ratio of methanol to oil of 6:1. This response surface shows the real values of the operating conditions as it is represented in Equation (8).

In the same way, response surfaces of temperature and stirring can be ploted for purity of biodiesel at molar ratio 6:1 as shown in Figure 3.

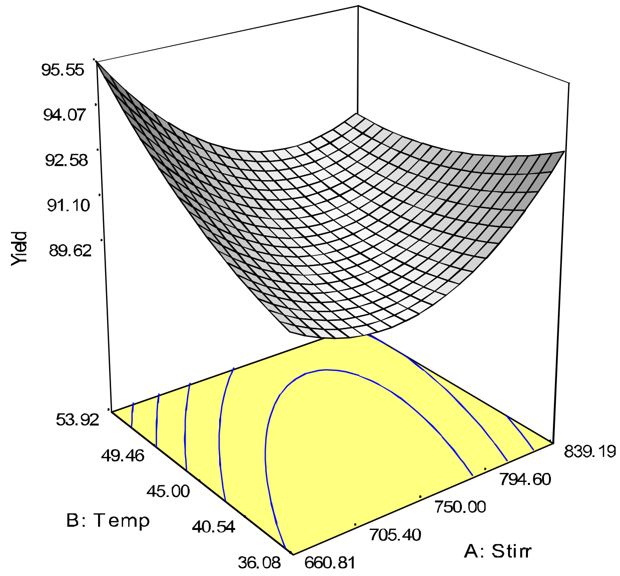


Figure 2: Response surface plots of biodiesel yield as a function of temperature and stirring. Molar ratio of methanol to oil is 6:1

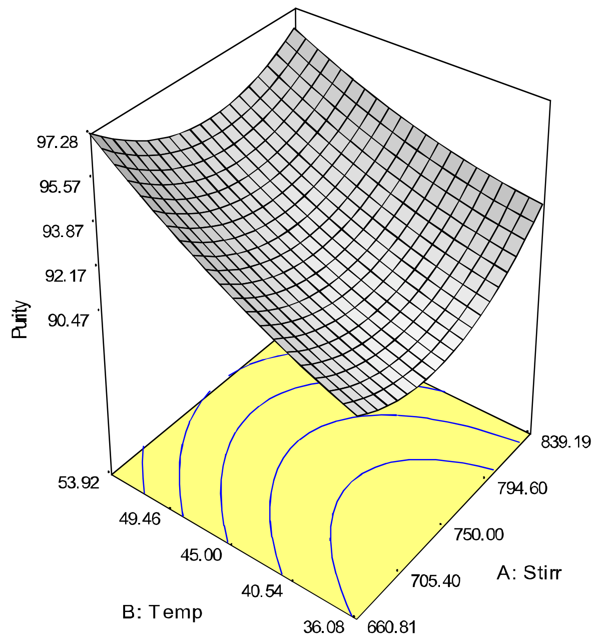


Figure 3: Response surface plots of biodiesel purity as a function of temperature and stirring. Molar ratio 6:1.

Predicted vs actual response plots of yield and purity are shown in Figure 4.

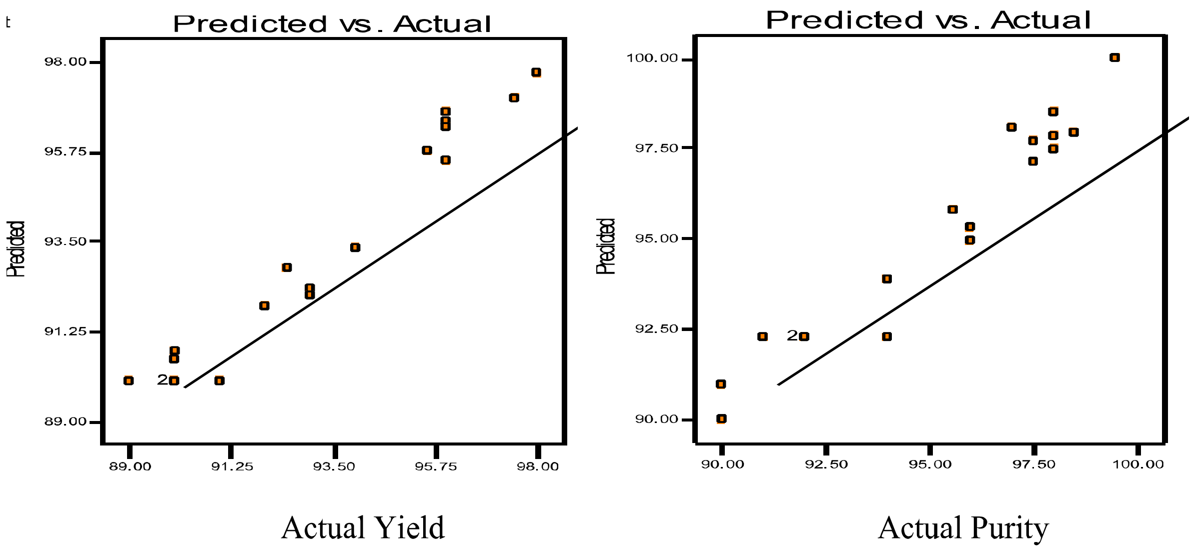
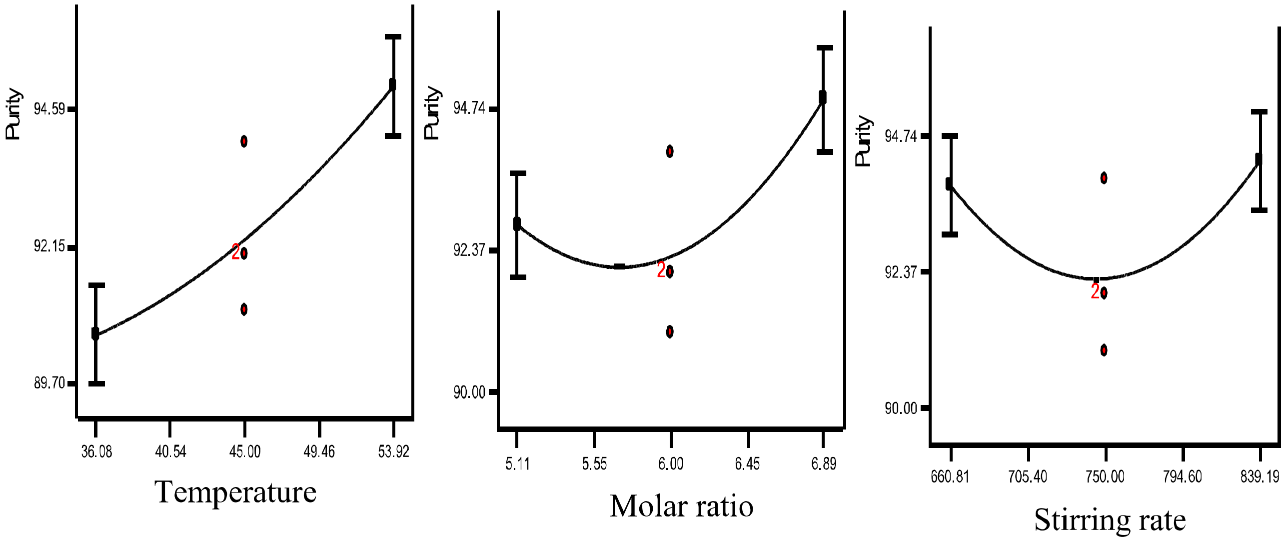


Figure 4: Predicted vs. actual response plots for yield and purity

Effects of the variables: The effects of temperature, stirring rate and molar ratio of methanol to oil on the yield and purity are now discussed using the statistical model Equations (6) and (7). The main effects and interaction plots on biodiesel purity and yield are given in Figures 5 and 6 respectively.

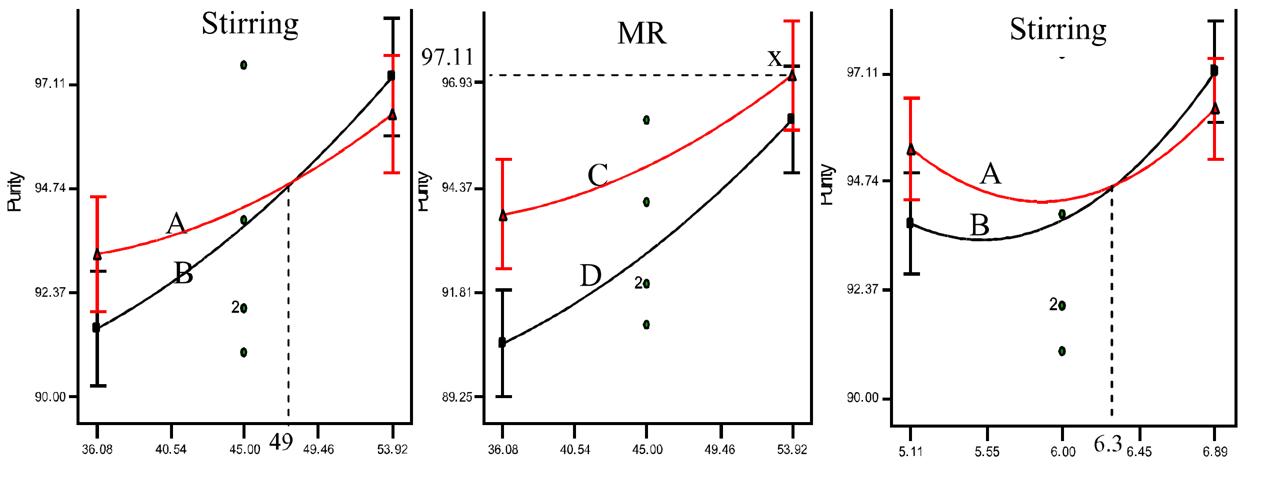
Influence of operating variables on the purity of biodiesel: Analysis of the studied range of experimental conditions shown in Figure 5a indicates that temperature is the most important factor on the purity followed by molar ratio and then stirring rate. This is because the slopes of the corresponding curves decrease in that order. On the interactions, high stirring speed of 839 rpm (curve A) favours the purity of biodiesel up to a temperature of about 49°C when low stirring speed of 661 rpm (curve B) becomes better (Figure 5b (i)). For Temperature-Molar ratio interactions Figure 5b (ii), high molar ratio of 6.89 (curve C) and high temperature of 54°C (point x) produces maximum purity of biodiesel of 97%. Figure 5b (iii) shows that high stirring speed of 839 rpm (curve A) has a negative effect on purity up to a molar ratio of about 6.3 when low stirring speed of 661rpm becomes better and purity of 97% is obtained at a molar ratio of 6.89. When the amount of methanol increases, the oil becomes more soluble and therefore low stirring rate is sufficient for the reaction.

The thick black dots in the centres of the figures are design points returned by the software which are not used in this discussion. The number 2 before the dot means that two different runs produce the same response of purity or yield, for example run numbers 2 and 18 (not shown here) produce purity of 92% with molar ratio of 6.0 in Figure 5b (iii.



(i) (ii) (iii)

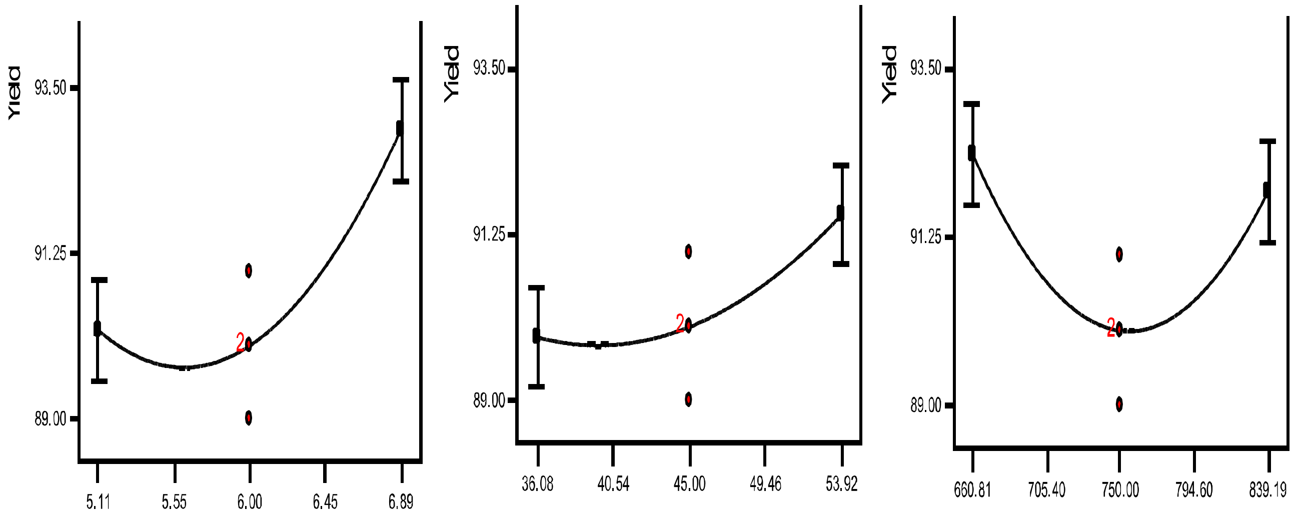
Figure 5a: Plots of main effects of: (i) Temperature, (ii) Stirring rate and (iii) Molar ratio on biodiesel purity



(i).Temperature-Stirring rate (ii) Temperature-Molar ratio (iii) Molar ratio-Stirring rate

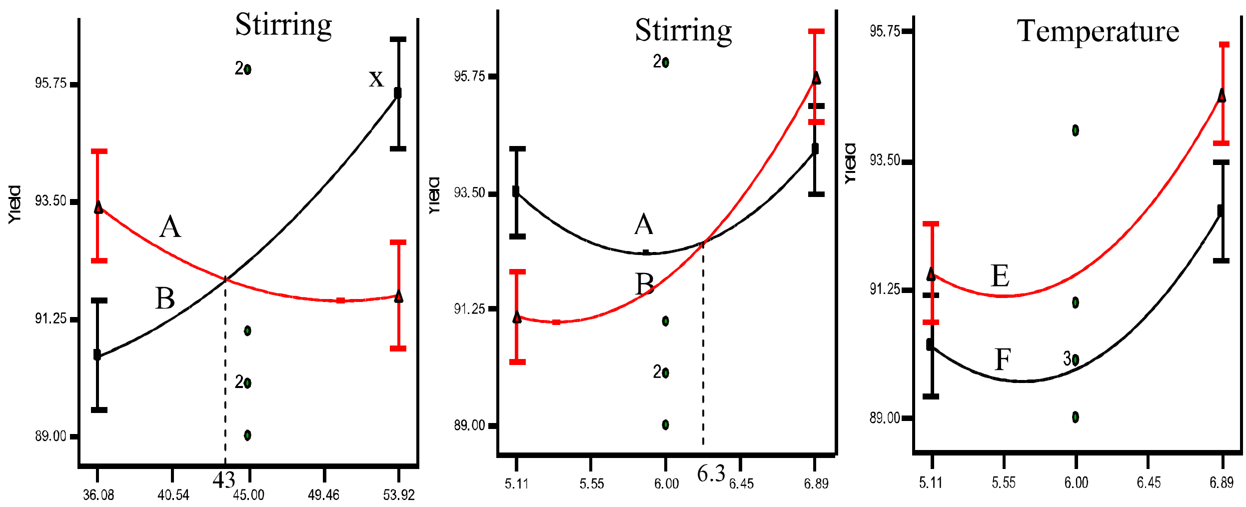
Figures 5b: Interactions on purity of biodiesel (Curve A: Stirring speed = 839 rpm; Curve B: Stirring speed = 661 rpm; Curve C: Molar ratio = 6.89; Curve D: Molar ratio = 5.10)

Influence of the variables on the biodiesel yield: Analysis of the variables on yield of biodiesel within the studied range indicates that molar ratio has greater effect on the yield than temperature and stirring (Fig. 6a). Increasing molar ratio and increasing temperature (i) and (ii) affect the yield positively; the effect of molar ratio being more pronounced than that of temperature while increasing stirring (iii).negatively affects the yield. Interactions of the variables on the yield are now discussed in Figures 6b.



Molar ratio (i) | Temperature (ii) | Stirring rate (iii)

Figure 6a: Plots of main effects of: (i) Molar ratio, (ii) Temperature and (iii) Stirring on the yield of biodiesel.



Temperature (i) Temperature-Stirring rate | Molar ratio (ii) Molar ratio- Stirring rate | Molar ratio (iii) Molar ratio-Temperature

Figures 6b: Interaction effects on the yield of biodiesel (Curve A: Stirring speed = 839 rpm; Curve B: = 661 rpm; Curve E: Temperature = 54℃; Curve F: Temperature = 36℃)

Figure 6b (i) shows that high stirring rate (curve A) does not favour increase in yield, whereas lower impeller speed of 661 rpm (curve B) is more favourable from a temperature of about 43 ℃ (dashed line) when the yield is 95.75% at 53.92℃ (point x). This is probably due to the fact that mass transfer no longer controls the reaction but only chemical reaction. Molar ratio- stirring effects indicate that both impeller speeds are favourable but lower speed (661 rpm) becomes better after a molar ratio of about 6.3, producing a yield of 95.75% at 6.89 MR (Figure 6b (ii)). Temperature-molar ratio shows that high temperature of 54 ℃ (curve E) is favourable giving a yield of 95.75%. The quadratic effects of all the variables have significant positive influence on both yield and purity responses, however, their absolute values are less than their corresponding main effects.

Analysis of the responses: The optimum operating conditions for the sodium hydroxide-catalysed transesterification of jatropha oil are those that obtained the highest biodiesel purity (methyl ester concentration) in the FAME phase and the highest yield in this phase. Biodiesel specification in terms of glyceride level, neutralization number, glycerol and methanol contents are quite rigorious and all of these terms are related to the purity (ester contents) of biodiesel. According to the European Union EN14214, the ester content should be greater than or equal to 96.5% (wt.) In this study, the limit of biodiesel purity of 97.28% (wt), from surface response plot of purity was chosen also having in mind economic factors. The order of priority is therefore; moderate temperature, moderate impeller speed and small quantity of methanol. The optimum conditions derived from the response surfaces in Figures 2 and 3 were as follow: temperature of 54 ℃, molar ratio of 6:1 and stirring speed of 661 rpm. Under these conditions, the yield that can be obtained is 95% and a purity of 97%.

Conclusion

In this work, a simulation has been done using a full central composite design to optimize the transesterification process of jatropha oil for biodiesel production using sodium hydroxide as a catalyst. The design was followed to optimize the variables that determine the maximum FAME yield and purity. A 2^3 full factorial design was not able to fit the data to the two responses; yield and purity. A CCRD was then used to predict the responses as functions of temperature, stirring rate and molar ratio.

Response equations were obtained for FAME yield and purity and from them it is possible to accurately predict the operating conditions required to obtain a given value of yield or purity. The optimum values of temperature, degree of agitation and molar ratio obtained in this work were; 54 ℃, 661 rpm and 6:1 respectively. Using these conditions, biodiesel yield of 95.55% (wt) can be obtained with a purity of 97% .

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