

OPTIMIZATION OF YELLOW OLEANDER BIODIESEL PRODUCTION PARAMETERS USING CENTRAL COMPOSITE DESIGN RESPONSE SURFACE METHODOLOGY

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Abstract

Response surface methodology was used for optimization of methyl ester production parameters with the two-step trans- esterification of Yellow oleander seed oil. The model equation obtained with R², 0.877 and coefficient of variation (CV), 3.21% shows the reliability of the model and adequately captured the correlation between the biodiesel yield and process parameters. The result suggested the best combination of the process variables for optimum biodiesel yield of 91.42% are: reaction temperature (46.61 °C); reaction time (90.52 min); amount of methanol (5.90 cm³/g oil) and catalyst concentration (11.44gm). Validation results show close agreement between the actual (90.85%) and predicted (91.42%) biodiesel yields.

Keywords: Optimization, Yellow oleander, Trans- esterification, Biodiesel, Central Composite design, Response Surface Methodology.

Introduction

World population is geometrically growing leading to high and rapid economic activities demand resulting in astronomical rise in energy consumption, particularly liquid fuels for transport and industries [1]. A large percentage of this energy demand is met by fossil fuel-based sources such as coal, petroleum, and natural gases [2]. Depleting crude oil reserves and environmental pollution due to extreme consumption of these fuels have forced mankind into searching alternatives which are cheaper and harmless to the environment [3]. Biofuels, liquid or gaseous fuels from biomass have been widely accepted as replacement of conventional energy in the transport sector [4] where they are blended with either gasoline or diesel. The most common types of liquid biofuels are bioethanol, straight vegetable oils and biodiesel [4]. Biodiesel is a mixture of mono alkyl esters of long chain fatty acids derived from renewable lipids and animal fats when reacted with alcohol in presence or absence of catalyst [5]. It is an environmentally friendly, nontoxic, biodegradable and renewable biofuel [6]. It has been proved as the best substitute to fossil-based diesel fuel in diesel engines offering similar engine emissions of particulate, hydrocarbon and carbon monoxide [7]. At present, edible plant oils constitute 95% of biodiesel production feedstock [8]. There are concerns that biodiesel feedstock may compete with food supply in the long run [6]. Non-edible plant oils have been found to be promising feedstock to produce biodiesel [5].

Yellow oleander, one of the non-edible plant oil sources belongs to the family of plants called Apocynaceae is a small tree or shrub with yellow, bell-shaped flowers. It originates in the Mediterranean regions of southern Europe and southwest Asia [9].

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To attain maximum biodiesel yield, optimization processes are usually employed [10, 11]. The most versatile method used by researchers for optimization of biodiesel production including process parameters is response surface methodology [12]. However, there is no elaborate work done or sufficient evidence concerning the optimization of biodiesel from yellow oleander seed oil.

A study on the effects of process parameters variations and optimization of biodiesel production from orange seed oil using raw and thermal clay as catalyst was reported by [13]. The results show that experimental/actual maximum optimal biodiesel yield was 79.53 and 94.58% v/v while the predicted biodiesel yield was 79.55 and 92.98% v/v. The set of conditions that caused these positive effects were established at Time of 150 minutes, Temperature 65°C, methanol /sample molar ratio of 12:1, catalyst concentration of 3.0 wt. % and agitation speed at 300 rpm respectively.

This study aims at finding the best combination of biodiesel production parameters which can give the highest biodiesel yield using the RSM of CCD. To do this, the effects of parameters such as reaction time, catalyst concentration and reaction temperature and methanol-to-oil ratio were investigated and optimized.

Methodology

Yellow oleander seed oil for this study was extracted from the seeds by the Soxhlet extraction process. Methanol and potassium hydroxide of technical grade were used for all experiments. The central composite design (CCD) matrix of response surface methodology (RSM) using Design Expert 7 software was applied for the design of the experiment to optimize the reaction variables. The model was validated by performing additional experiments using the optimum values of process parameters.

Procedure

A 100 ml of yellow oleander oil was poured into the flask and heated to 60 °C. Methanol (45% v/v) was mixed with the heated Yellow oleander oil and stirred for some minutes. Some quantity of 0.5% Sulfuric acid (H_2SO_4) was poured into the mixture and heated for 45 minutes at normal pressure. At completion of reaction, the mixture was poured into separating funnel to separate the excess alcohol, impurities and sulfuric acid which moved to top layer and were discarded. The esterified oil which remains at the lower layer was kept for further processing into methyl ester.

The esterified oil was poured into a flask and heated to 60°C. A 1% quantity of KOH was dissolved in 30% (6:1 M) methanol and added to the esterified oil. The mixture was then heated while stirring for 1 hour. At the end of reaction, the mixture was poured into separating funnel and left for 12 hours to settle. Glycerol and impurities settled in the lower layer while impure biodiesel containing traces of catalyst, glycerol and methanol remained in upper layer was drawn off, washed and dried

Single factor experiments

Single factor experiments in which one factor was varied while the other factors were kept constant to fine ranges of the independent parameters to be used in design of experiments was carried out. The results of the experiments were used to fix the range of each independent variable which influence biodiesel yield.

Experimental Design

A 2^k (number of factors $k = 4$) factorial central composite design (CCD) RSM was employed to optimize catalyst concentration (A), methanol- oil molar ratio (B), process temperature (C) and reaction time (D) for high biodiesel yield. Catalyst concentration, methanol-to-oil ratio, temperature and reaction time are independent variables while biodiesel yield is the dependent variable. Thirty experimental runs made up of 16 factorial experiments, 8-star points and 6 center

points were conducted. It was found from the preliminary production of biodiesel from yellow oleander seed oil that biodiesel yield will be most effective in the range of catalyst amount from 0.75 to 15 g, methanol- oil molar ratio 3: 1 to 12: 1, reaction temperature 40°C to 60°C and processing time in the range from 30 to 180 minutes (Fig. 1 to 4). The range values coded to lie at ± 1 for the factorial points, 0 for center points and $\pm \alpha$ for axial points were calculated using central composite circumscribed (CCC) method as shown in Table 1.

Table 1. Ranges and levels of independent process parameters using Central Composite Circumscribed (CCC) of CCD

Factor	$-\alpha$	- 1	0 (middle)	+1	$+\alpha$
Catalyst (A) (g)	0.75	4.31	7.88	11.44	15.00
Molar ratio (B)	3.00	5.25	7.50	9.75	12.00
Temperature(C) (°C)	40.00	46.25	52.50	58.75	65.00
Time (D) (minutes)	60.00	90.00	120.00	150.00	180.00

The relationship between independent parameters and yield was determined using second order polynomial equation [1]. Design-Expert 7 (Stat-Ease Inc., Minneapolis, MN, USA) software was used for analysis of variance (ANOVA) and calculation of modal coefficients. The experimental data was analyzed by the response surface regression procedure to fit to the following second-order polynomial equation of Equation.

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

Where Y is the predicted response, β_0 the model constant, β_i the linear coefficient, β_{ii} the quadratic coefficient, β_{ij} interaction coefficient and k number of factors considered for study. Table 2 shows the design summary for the optimization of biodiesel yields and production parameters.

Table 2. Design Summary for the optimization of biodiesel yields (study type: response surface; experiments :30; initial design: central composite blocks: no blocks; design model: quadratic)

Factor	Name	Units	Type	Low Actual	High Actual	Low Coded	High Coded
A	Catalyst	g	Numeric	4.31	11.44	-1.000	1.000
B	Molar Ratio	v/v	Numeric	5.25	9.75	-1.000	1.000
C	Temperature	°C	Numeric	46.25	58.75	-1.000	1.000
D	Time	mins	Numeric	90.00	150.00	-1.000	1.000

Results and Discussion

Single Factor Preliminary Experiment

The influences of each of the four factors selected for optimization on biodiesel yield as found from the preliminary experiments are discussed here. Fig. 1 to 4 show the variation in biodiesel yield with increase of each factor.

Influence of catalyst amount on biodiesel conversion rate

KOH in the range of 0.75 to 16g was taken for this experiment. Fig. 1 shows the variation in rate of biodiesel conversion with increased catalyst. Trans esterification of yellow oleander seed oil with 7.5g of potassium hydroxide catalyst gave the highest yield of biodiesel of 85% with 4.57g of the catalyst. A steep reduction in biodiesel yield was noticed with increased quantity of

catalyst. Excess catalyst in a reaction leads to formation of emulsions and gels which increased the viscosity thereby blocking the reaction resulting in decreased yield of biodiesel.

Influence of methanol-oil molar ratio on biodiesel conversion rate

A steady rise in biodiesel conversion can be seen (Fig. 2) with increase in molar ratios depicting proportional response as amount of methanol increases. An optimum yield of 84.75% at a molar ratio of 9:1 was achieved. However, when the molar ratio became higher, the increment of biodiesel yield reduced considerably. According to [14] and [15] higher molar ratio of alcohol to vegetable oil impedes glycerin separation as a result of increased insolubility. When glycerin remains in the solution, it causes a shift in equilibrium to the left, resulting in the lower yield of biodiesel. The range of values chosen in the case of methanol-oil molar ratio for optimization experiment 3:1 to 12:1

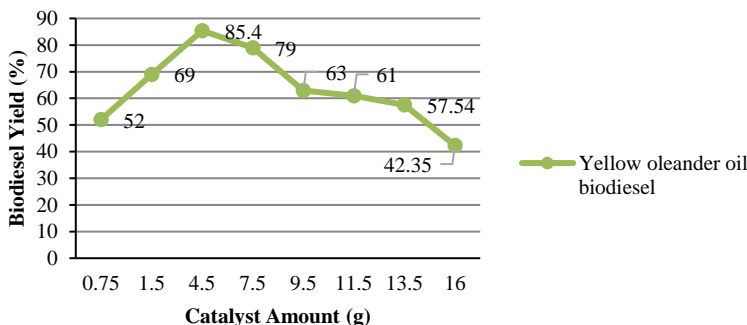


Fig. 1. Influence of Catalyst Amount on Biodiesel Conversion rate

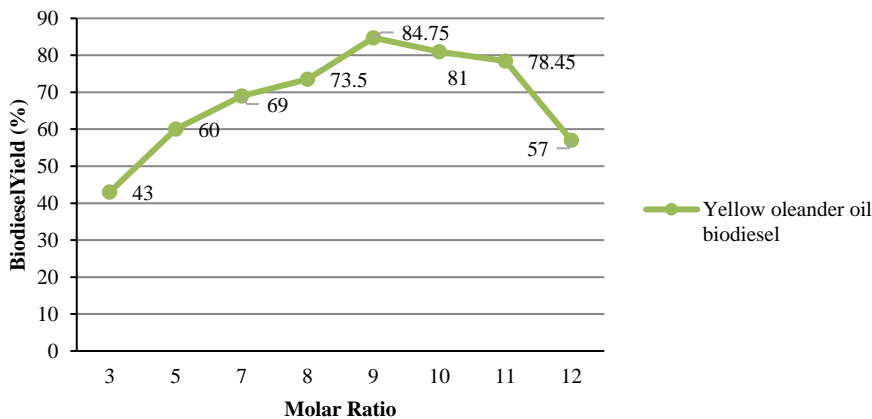


Fig. 2. Influence of Methanol-Oil Molar Ratio on Biodiesel Conversion rate

Influence of temperature on biodiesel conversion rate

Reaction temperature has positive influence on biodiesel yield (Fig. 3) provided it does not exceed the boiling point of methanol. The highest biodiesel yield of 85% was gotten at 61.5°C; further temperature increases decreased biodiesel yield. The reaction temperature above boiling point of methanol (65°C) was avoided in the choice of suitable range since at higher temperatures;

methanol evaporates resulting in soap formation by the alkaline catalyst with glycerides before completion of the reaction [14].

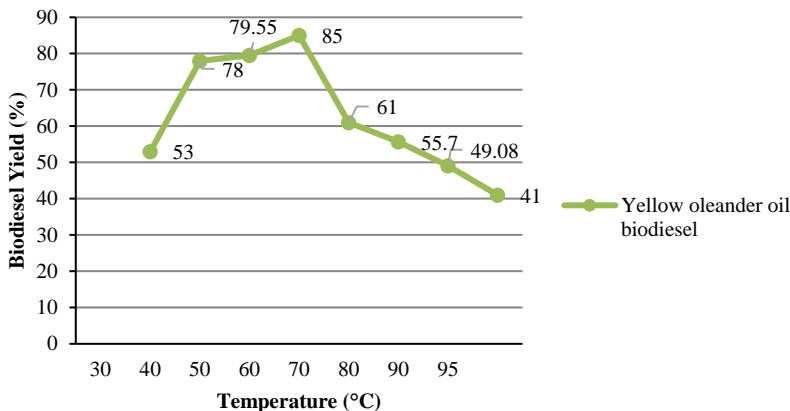


Fig. 3. Influence of Reaction Temperature on Biodiesel Conversion rate

Influence of reaction time on biodiesel conversion rate

For the experiment with time in the range of 30 to 180 minutes the influence of reaction time on conversion efficiency (Fig. 4). At the beginning, reaction was slow due to time taken for mixing of methanol and the yellow oleander triglycerides (16). However, the reaction increased steadily from 40 minutes of reaction. As depicted, though the increase in reaction time at 100 to 120 minutes engendered an increase in biodiesel yield of 82% maximum at 120 minutes, this subsequently declined with a further increase above 140 minutes to 51% at 180 minutes. The decline is attributed to the imperfect trans-esterification response between methanol and oil [16]. From this experiment it can be deduced that 150 minutes is enough for completion of the reaction.

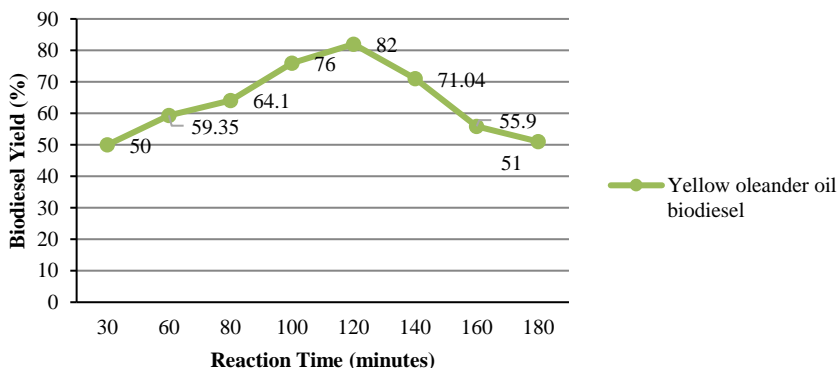


Fig. 4. Influence of Reaction Time on Biodiesel Conversion Efficiency

Statistical Analysis and Model Fitting

The experimental design matrices of the independent parameters investigated are on CCD basis with result of biodiesel yield shown in Table 3. Thirty experimental runs were conducted,

and the result analyzed by multiple regression. The predicted results were gotten by model fitting technique and are maximally correlated to observed values.

Equation 2 is a quadratic model equation in coded factors which relate biodiesel yield to various process parameters. The evaluated coefficients of the response surface of the model are provided by equation 1. The significance of each coefficient is checked from p-values (probability of error value), which also shows the level interaction of each parameter.

Final modal equation in coded factors;

$$\begin{aligned} \text{Yield}(\%) = & 85.0 - 7.08A + 11B - 0.743C + 0.0293D + 0.440AB + 0.0356AC \\ & + 0.00582AD - 0.0880BC - 0.0318BD + 0.00716CD + 0.0278A^2 \quad (2) \\ & - 0.393B^2 + 0.00348C^2 - 0.000559D^2 \end{aligned}$$

Where: A = Catalyst amount, B = Methanol molar ratio, C = Reaction temperature (°C) and D = Reaction time (minutes)

Table 3. Experimental design matrix and results for yellow oleander biodiesel production

Run	A: Catalyst	B: Molar ratio	C: Temperature °C	D: Time minutes	Yield %
1	11.44	5.25	58.75	150	93
2	7.88	7.5	52.5	60	85
3	7.88	7.5	52.5	120	91.45
4	4.31	5.25	46.25	90	92
5	11.44	9.75	58.75	90	91
6	15	7.5	52.5	120	90
7	11.44	5.25	58.75	90	75
8	7.88	7.5	52.5	120	88
9	7.88	7.5	52.5	120	89
10	11.44	5.25	46.25	150	80
11	11.44	9.75	46.25	150	90.09
12	7.88	7.5	52.5	120	92.1
13	4.31	5.25	46.25	150	93.85
14	7.88	7.5	65	120	93.11
15	7.88	7.5	52.5	180	96
16	0.75	7.5	52.5	120	97.86
17	7.88	3	52.5	120	80.09
18	7.88	7.5	52.5	120	91.25
19	4.31	9.75	46.25	90	90.53
20	4.31	9.75	46.25	150	91
21	4.31	5.25	58.75	150	97.9
22	4.31	9.75	58.75	150	91.33
23	7.88	7.5	40	120	93
24	11.44	5.25	46.25	90	73
25	11.44	9.75	46.25	90	93
26	11.44	9.75	58.75	150	91.03
27	7.88	7.5	52.5	120	96.75
28	4.31	5.25	58.75	90	90.43
29	4.31	9.75	58.75	90	88.95
30	7.88	12	52.5	120	89

The lack of fit test result for the model is in Table 4 and describes the variation in the data around any fitted model [17]. The lack of fit value of 0.85 means it is not significant and model fits the data very well. The large p value of 0.2182 of the lack of fit for biodiesel production model also means that it is not significant; implying the model adequately described the data. The F- value of 8.56 is further confirmation of significance of the model. Similarly, the p-value of 0.00017 of the shows very high significance of the model and its suitability in predicting the response values (Table 4). The reliability of the fitted model is proved by the low value coefficient of variation (CV) (3.21%). The high values of coefficient of determination (R^2) and adjusted

coefficient of determination (R^2 Adj) (0.763) further confirms the significance and quality of the model and shows that the developed model equations successfully captured the correlation between the process parameters and biodiesel yield as similarly reported by [18].

The terms AB, AC, AD, BC, BD and CD in equations 2 are interaction terms. Interaction terms show the effect of two factors on biodiesel yield.

The variables with the largest influence on biodiesel yield are interaction terms of catalyst amount and methanol- oil molar ratio (AB) (P- value of 0.00019) followed by the quadratic term of methanol- oil molar ratio (B) (with P- value = 0.0025) then the linear term of methanol molar ratio (B) (with P- value = 0.0041), the linear term of catalyst amount (A) (p-value = 0.0045), interaction terms of methanol- oil molar ratio and reaction time (BD) (p-value = 0.0094) and interaction terms of reaction temperature and reaction time (CD) (p-value = 0.0082).

The effect of the linear term of process temperature (C) (p- value = 0.65), interaction terms of catalyst amount and reaction temperature (AC) (P= 0.28), interaction terms of catalyst amount and reaction time (AD) (P= 0.40), interaction terms of methanol- oil molar ratio and reaction temperature (BC) (0.10), the quadratic terms A^2 , C^2 and D^2 (0.53, 80 and 0.37 respectively), are found to be insignificant (Table 5).

In terms of parameters that have significant effects on yellow oleander biodiesel yield equation [2] becomes

$$Yield(\%) = +85.0 - 7.08A + 11B + 2.35D - 0.393B^2 + 0.440AB - 0.303BD + 0.00716CD \quad (3)$$

Regression analysis of the experimental data also show that the linear terms of amount of catalyst (A) of yellow oleander seed oil have positive and negative linear influence respectively on yield. AB and A have linear effects of +0.440 and -7.08 on yield (Equation [3]).

Optimization of Process Parameters Conditions

The graphical representation of the regression equation 2 and effects of two independent parameters within the experimental ranges are depicted in 3D- surface plots and contour plots in Fig. 5 to 10. The shapes of the surface plots indicate different interactions between the variables. The contour curves describe the trends in the yields of biodiesels when process parameters are decreased or increased.

Interactive effects of process parameters on biodiesel yield

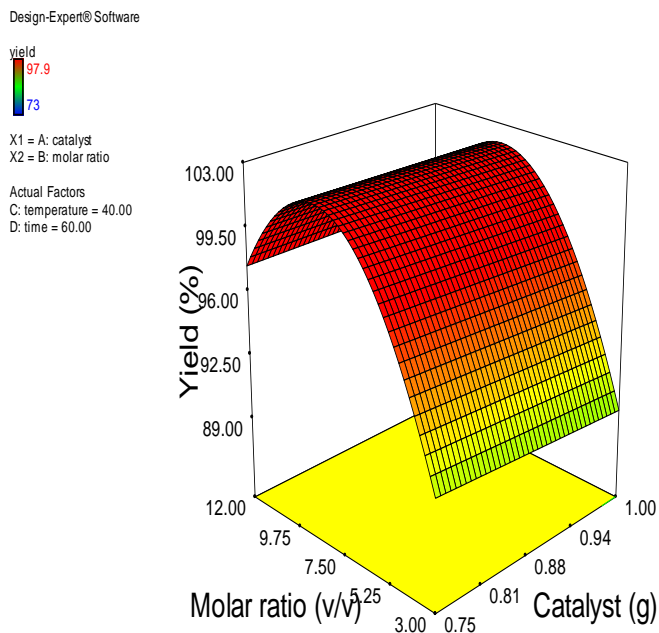
As shown in Fig. 5, increased biodiesel yield was observed with increasing catalyst amount and methanol- oil molar ratio. Increasing both factors up to certain optimum values result in high biodiesel yield. Further increase in the factors beyond the optimum region resulted in retardation in biodiesel production due to backward reaction and formation of soap. At catalyst amount higher than 9.66g, the biodiesel yield began to decrease at all of methanol- oil molar ratios. Similarly, at methanol- oil molar ratio higher than 8.70: 1, biodiesel yield begins to decrease. The contour plot in Fig. 5b shows that midpoint of catalyst amount and methanol-oil molar ratio result in optimum biodiesel yield (7.88g of catalyst and 7.50:1 molar ratio respectively). In the interaction between catalyst amount and reaction temperature, the contour plot in Fig. 6b illustrate that increasing reaction temperatures result in increased biodiesel yields. Also increase in catalyst amount increased yield of biodiesel till a midpoint after which the yields decreased. As seen from the contour curve, reaction temperatures must be kept high enough for the reaction rate to be rapid but low enough to avoid the evaporation of methanol. A temperature of 52.50°C can be deduced as the optimum from the plots.

Table 4. ANOVA for response surface quadratic model for yellow oleander seed biodiesel optimization

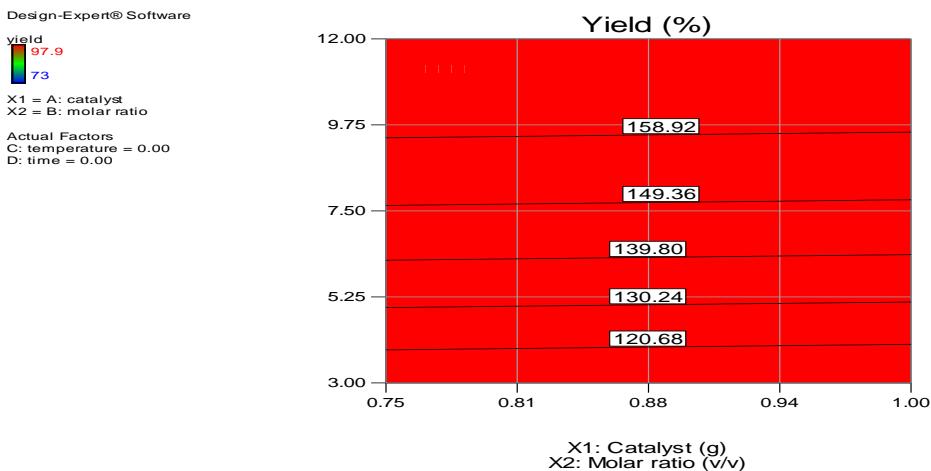
Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Model	893	14	63.8	7.65	0.00017	significant
<i>A-catalyst</i>	92.8	1	92.8	11.1	0.00451	
<i>B-molar ratio</i>	95.3	1	95.3	11.4	0.00411	
<i>C-temperature</i>	1.75	1	1.75	0.21	0.654	
<i>D-time</i>	0.099	1	0.099	0.0119	0.915	
<i>AB</i>	199	1	199	23.9	0.000195	
<i>AC</i>	10.1	1	10.1	1.21	0.289	
<i>AD</i>	6.19	1	6.19	0.743	0.402	
<i>BC</i>	24.5	1	24.5	2.94	0.107	
<i>BD</i>	73.7	1	73.7	8.85	0.00945	
<i>CD</i>	28.8	1	28.8	3.46	0.0827	
<i>A2</i>	3.43	1	3.43	0.411	0.531	
<i>B2</i>	109	1	109	13.1	0.00256	
<i>C2</i>	0.507	1	0.507	0.0608	0.809	
<i>D2</i>	6.93	1	6.93	0.832	0.376	
Residual	125	15	8.33			
<i>Lack of Fit</i>	78.6	10	7.86	0.846	0.617	not significant
<i>Pure Error</i>	46.5	5	9.29			
Cor Total	1.02E+03	29				
Std. Dev.	2.89		R-Squared	0.877		
Mean	89.8		Adj R-Squared	0.763		
C.V. %	3.21		Pred R-Squared	0.49		
PRESS	519	Adeq Precision		12.4		

Long reaction times and excess catalyst amounts result in decreased biodiesel yields. Short reaction times and small catalyst amounts also result in low biodiesel yield. The optimum points on the contour curves presented in Fig. 4.16b and 4.17b are at the midpoints of catalyst and process time. A similar trend is seen in the effect of interaction between methanol- oil molar ratio and reaction temperature. A molar ratio of 6.98:1 and temperature of 53.56°C produced an optimum yield of 90.34% for yellow oleander. Fig. 9b is the contour plot for the interactive effect processing time and methanol- oil molar ratio on biodiesel production.

Fig. 10 is contour plot of interactive effects of reaction temperature and process time on biodiesel yield. Higher reaction temperatures lead to increased biodiesel yield. However, temperature above 65°C will cause methanol to vaporize leading to low biodiesel yield. Therefore, temperature below 65°C with midpoint time of 105 minutes is essential for high yield of biodiesel.



a)



b)

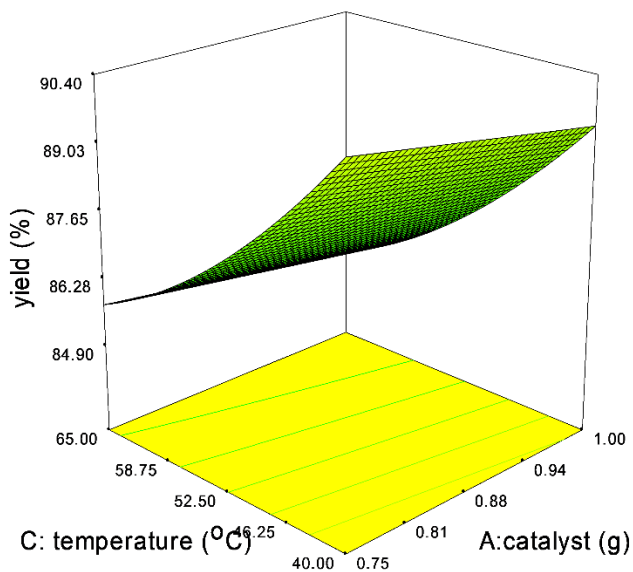
Fig. 5. Effect of interaction catalyst amount and methanol- oil molar ratio on yellow oleander biodiesel yield: a) 3-dimensional surface plot; b) contour plot.

Design-Expert® Software



X1 = A: catalyst
X2 = C: temperature

Actual Factors
B: molar ratio = 3.00
D: time = 60.00



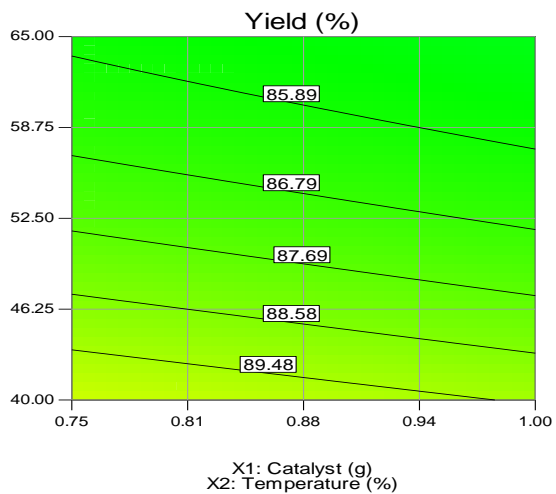
a)

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X1 = A: catalyst
X2 = C: temperature

Actual Factors
B: molar ratio = 3.00
D: time = 60.00



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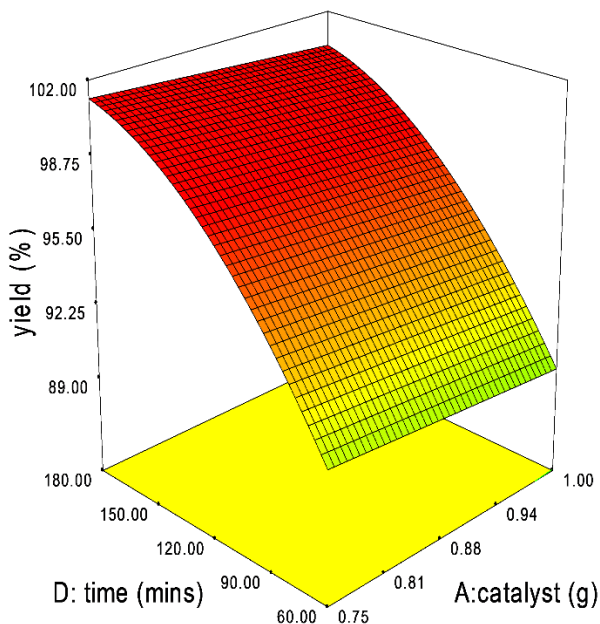
Fig. 6. The interaction effect of catalyst amount and temperature on yellow oleander biodiesel yield: a) 3- Dimensional surface plot; b) Contour plot

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X1 = A: catalyst
X2 = D: time

Actual Factors
B: molar ratio = 3.00
C: temperature = 40.00



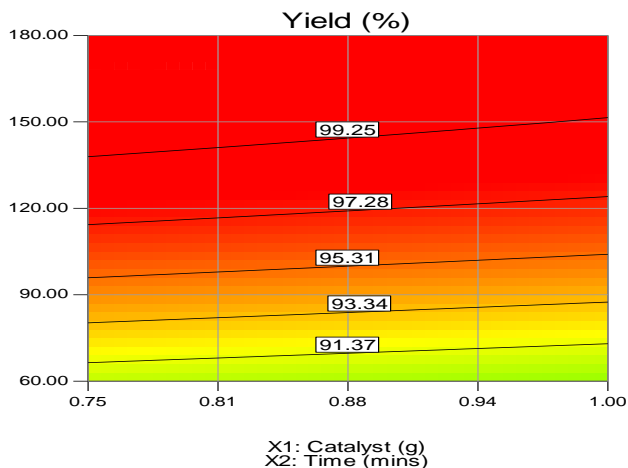
a)

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X1 = A: catalyst
X2 = D: time

Actual Factors
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C: temperature = 40.00



b)

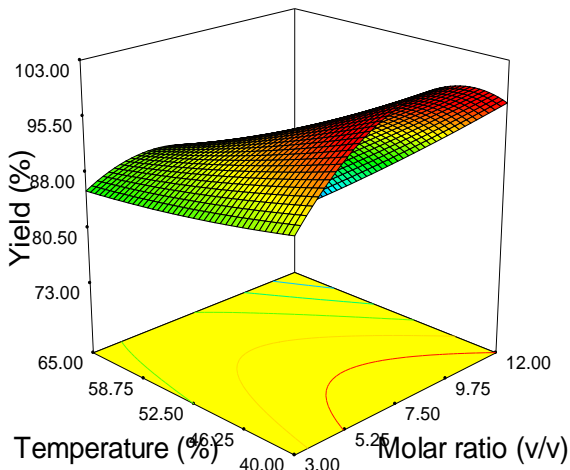
Fig. 7. The interaction effect of catalyst amount and processing time on yellow oleander biodiesel yield: ((a) 3- Dimensional surface plot (b) Contour plot

Design-Expert® Software



X1 = B: molar ratio
X2 = C: temperature

Actual Factors
A: catalyst = 0.75
D: time = 60.00



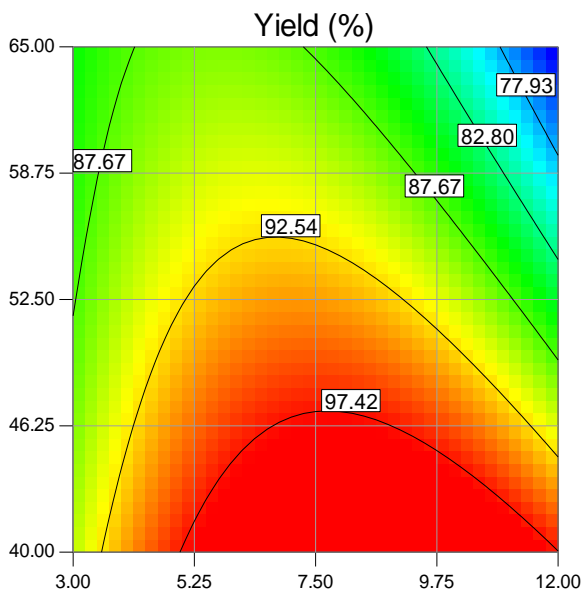
a)

Design-Expert® Software



X1 = B: molar ratio
X2 = C: temperature

Actual Factors
A: catalyst = 0.75
D: time = 60.00



X1: Molar ratio (v/v)
X2: Temperature (%)

b)

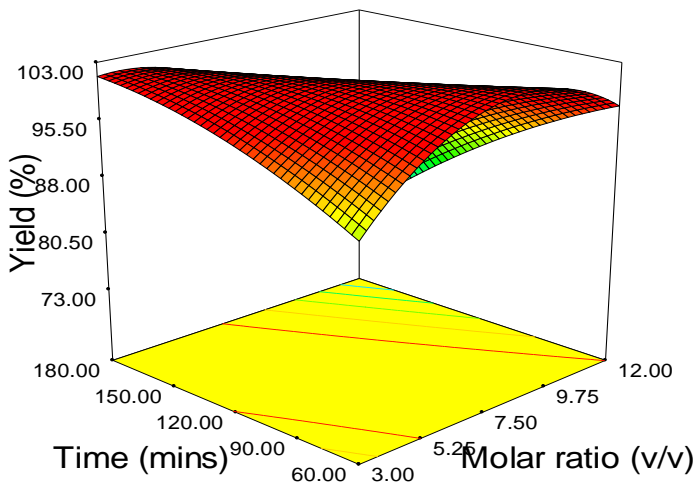
Fig. 8. The effect of interaction between methanol- oil molar ratio and temperature on yellow oleander biodiesel yield: a) 3- Dimensional surface plot; b) Contour plot

Design-Expert® Software



X1 = B: molar ratio
X2 = D: time

Actual Factors
A: catalyst = 0.75
C: temperature = 40.00



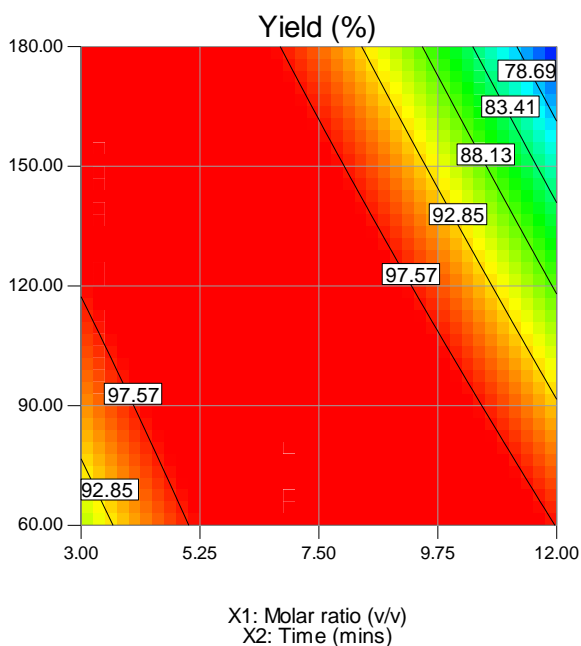
a)

Design-Expert® Software



X1 = B: molar ratio
X2 = D: time

Actual Factors
A: catalyst = 0.75
C: temperature = 40.00



b)

Fig. 9. The interaction effect of methanol-oil molar ratio and processing time on yellow oleander biodiesel yield: a) 3- Dimensional surface plot; b) Contour plot

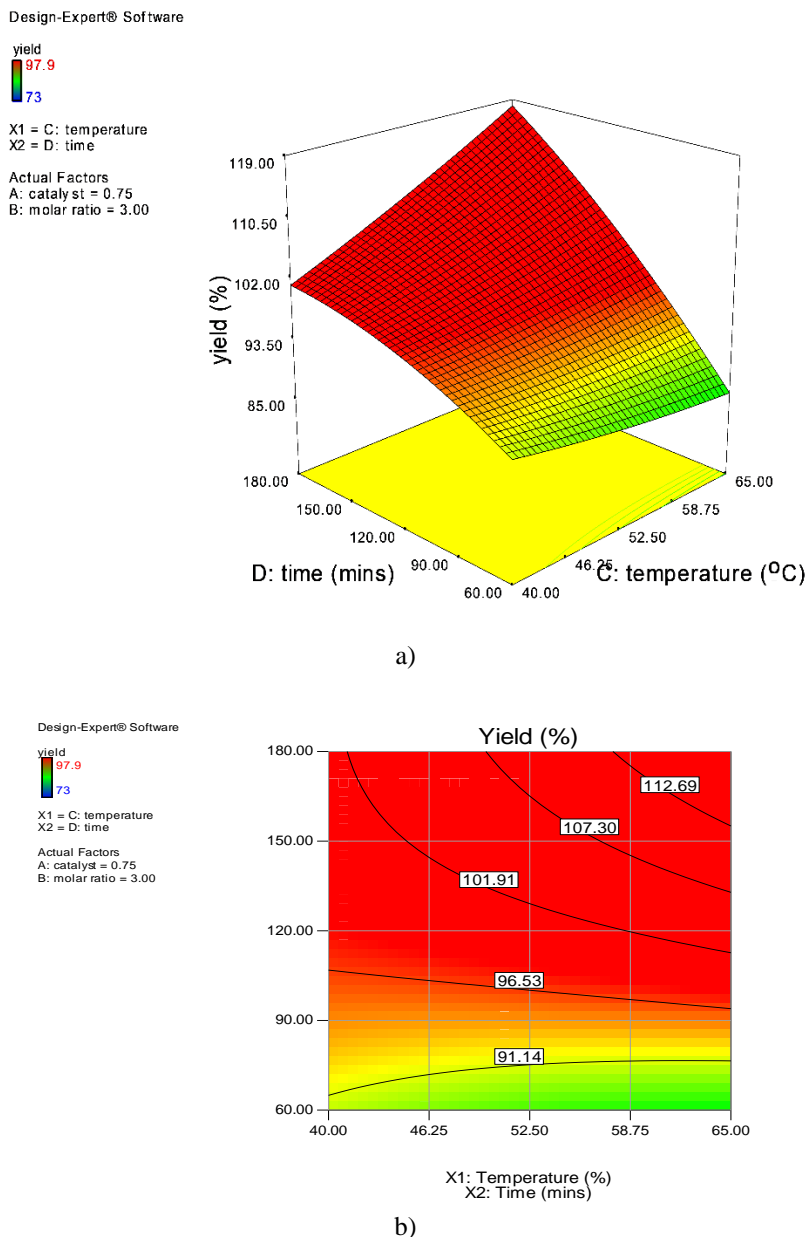


Fig. 10. The interaction effect of reaction temperature and processing time on yellow oleander biodiesel yield: a) 3- Dimensional surface plot; b) Contour plot

Validation of the Model

The maximized production parameters were used in validating the predicted response. A biodiesel yield of 90.85% was obtained from the real experiments with as low as only 0.62% error. The result is in close agreement with model prediction, indicating that the model is adequate for yellow oleander biodiesel production. Predicted optimum parameters from the model equation for biodiesel production are amount of catalyst, 11.44g; methanol- oil molar ratio, 5.90:1; reaction temperature of 46.61°C and process time of 90.52 minutes. The predicted biodiesel yield under

these optimum conditions is 91.42% while actual yellow oleander biodiesel yield 90.85%. Predicted and actual yield under optimum production parameters conditions is in Table 5. The design expert plots of predicted biodiesel yield against actual yield in Fig. 11 clearly show how close the experimental values of biodiesel yields are to the predicted values.

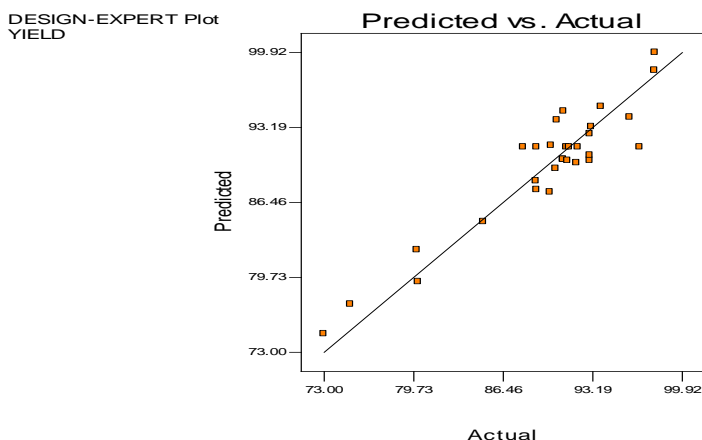


Fig. 11. Design expert plot of predicted yield against actual (experimental) yield for yellow oleander biodiesel

Table 5. Predicted and actual yields under optimum production parameters

Optimum condition				Biodiesel Yield(%)	
Catalyst oil gram	Methanol molar ratio	Temperature °C	Processing time Minutes	Predicted	Experimental
11.44	5.90:1	46.61	90.52	91.42	90.85

Conclusion

The application of CCD RSM in seeking optimal conditions for biodiesel production has been investigated. From the results obtained the following conclusions are drawn:

- the optimal process conditions for Yellow oleander biodiesel production were determined to be: Catalyst amount, 11.44g, Methanol- oil ratio, 5.90:1, Temperature, 46.61 °C, Reaction time, 90.52 mins;
- a maximum yield of 90.85 was achieved under the optimal conditions;
- the result showed that catalyst concentration and methanol- oil molar ratio had significant effects on biodiesel yield;
- the interaction between methanol- oil molar ratio and time and temperature and time had significant effects on biodiesel yield;
- the predicted values from the RSM model were in good agreement with the experimental values, indicating the accuracy of the model;
- the optimization of process parameters using CCD RSM led to a significant improvement of biodiesel yield reducing reaction time and amount of catalyst required.

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