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Optimization of Biodiesel Production Parameters Using Bauhinia variegate oil

G.N. Dayananda, K.V.Yathish, D.G. Kantharaj, B.R.Omkaresh, E.C. Manjunath Abstract

Response surface methodology (RSM) was adopted in which the optimal conditions were determined for the biodiesel production from Bauhinia variegate oil (BVO) employing central composite design (CCD). A total of 30 experiments were conducted to study the effect of methanol to oil molar ratio, reaction time, reaction temperature and catalyst loading on the biodiesel yield. A yield of 94.1% of Bauhinia variegate methyl ester (BVME/biodiesel) was obtained at optimum conditions: 6.86:1 molar ratio, 65.46 min reaction time, 1.08 wt.% catalyst concentration (KOH), 60.38 °C temperature and constant agitation speed (650 rpm). The fuel properties of the biodiesel were determined employing the ASTM test method.

Key words: *Bauhinia variegate oil, Bauhinia variegate biodiesel, Response surface methodology*

I. Introduction

Alternative fuel gain more importance in the today's world due to the depletion of fossil fuels [1]. Among the alternative fuels, biodiesel is more important which is alternative to the diesel fuel [2]. Generally biodiesel is produced from non-edible oil seeds and animal fat by the chemical process called transesterification [3]. For transesterification reaction, methanol is used as alcohol due low cost compare to other alcohols and potassium hydroxide is used as catalyst. Non-edible oils are preferred for biodiesel production to avoid the imbalance of food and fuel while using edible oil [4,5].

Non-edible oil seeds such as *Terminaliabelerica, Azadirachtaindica,Ricinuscimmunis, pongamiapinnata, Madhucaindica, Jatrophacurcus, Aeglemarmelos, Thevetiaperuviana*etc. are used for biodiesel production [6,7,8].

Meanwhile, other potential non-edible oil resource i.e., *Bauhinia variegate* is identified for biodiesel production. B. Variegate is a medicinal tree belongs to the family Leguminosae (Caesalpinioideae), which is also called mountain ebony [9]. It is planted on garden, park androadsides as ornamental plant intropical and subtropical regions. All the parts of the *B. variegate* tree have medicinal values. *B. variegate* tree yield non-edible oil seeds which have a Lectins (glycol protein) shows good antitumor activity [10]. Yatish et al. used B. variegate oil for biodiesel production (sodium phosphate catalyst used) and achieve 95.1% of yield with optimum reaction conditions [11].

From the literature, there are no reports available for the production biodiesel from B. variegate using potassium hydroxide catalyst. Present study, we use response surface methodology for optimization biodiesel production parameters using potassium hydroxide catalyst. The fuel properties of the biodiesel also evaluated.

II. Materials and methods

Raw materials

B. variegate seeds were collected from Bannerugatta forest, Karnataka State in India. Methanol and potassium hydroxide were purchased fromSigma-AldrichCo., and were used as such. B. variegate oil was extracted by using screw type mechanical expeller and 23% of oil was obtained.

Design of experiments

Response surface methodology (RSM) standard tool called central composite design (CCD) was used to determine the effects of BVME yield in the transesterification reaction. A five level four factor CCD consists of 30 experiments ($=2^x + 2x + N_0 = 16+8+6= 30$, where x =4, no. of independent variables and N_0 is the number of center points) including 16 factorial points ($=2^x$), 8 axial points (=2x) and 8 replicates ($=N_0$) at the centre point to estimate the pure error. The independent variables were molar ratio (*P1*), catalyst loading (*P2*) and reaction time (*P3*).

The high and low values are considered as +1 and -1 levels respectively as shown in Table 1.

Experimental Range and Values for RSM

Independent Variables	Range and Level				
	-α	**-1**	**0**	**1**	**A**
P_1 = Molar ratio	3:1	4.5:1	6:1	7.5:1	9:1
P_2 = Reaction time (min)	30	45	60	75	90
P_3 = Catalyst concentration (wt. %)	0.6	0.8	1.0	1.2	1.4
P_4 = Temperature (^oC)	41	48	55	62	69

Statistical analysis (ANOVA)

The response biodiesel yield (Y) was used to develop on empirical regression model for five level four transesterification reaction variables using second order polynomial Eq. (1).

$$Y = \beta 0 + \beta 1P_1 + \beta 2P_2 + \beta 3P_3 + \beta 4P_4 + \beta 11P_1^2 + \beta 22P_2^2 + \beta 33P_3^2 + \beta 44P_4^2 + \beta 12P_1P_2 + \beta 23P_2P_3 + \beta 13P_1P_3 + \beta 34P_3P_4 + \beta 14P_1P_4 + \beta 24P_2P_4 \quad (1)$$

where Y is the predicted response, *β0*the intercept, *β1, β2*and *β3* are linear coefficients, *β11, β22* and *β33* are quadratic coefficients, *β12, β23* and *β13*, are interactive coefficients and *P1*, *P2*,*P3*and*P4*are independent variables. Design-Expert version 8.0.7.1(Stat Ease, Inc., Minneapolis, USA) and was used for regression analysis to evaluate the analysis of variance (ANOVA) of the experimental data.

Biodiesel production

The free fatty acid of the oil was found to be 1.1%. Hence, single stage alkali transesterification was conducted. 200 g of*B. variegate* oil wastaken in a 500 ml round bottom three neck flask equipped with reflux condenser and magnetic stirrer. Firstly, the oil was preheated to 60 ^oC then the desired amount of catalyst and methanol mixture was added to the oil. The reaction was carried out at fixed time and temperature with stirring speed of 650 rpm. After the reaction complete allowed the reaction mixture to 8 hours for phase separation. The upper phase (biodiesel) and lower phase (glycerine) was separated and biodiesel was washed with pH 7 water, then heated at 120 ^oC. The clear biodiesel was obtained. The yield of the biodiesel was calculated according to Eq.(2).

$$\%\,\text{yield of BVME} = \frac{\text{Mass of BVME produced (g)}}{\text{Mass of BVO taken for reaction (g)}} \times 100 \quad (2)$$

III. Result and discussion

Regression model analysis for BVME yield

The transesterification parameters were analyzed by employing CCD and the results shown in the Table 2. To analyze the parameters a total of 30 experiments were conducted. Design expert software suggested the quadratic model to analyze the parameters. The BVME yield obtained from 54% to 94.8%. The BVME yield was predicted by second-order polynomial equation in terms of coded factors as:

$$Y = +94.35 + 2.70P_1 - 0.90P_2 + 6.80P_3 + 3.19P_4 + 0.89P_1P_2 + 0.31P_1P_3 - 0.29P_1P_4 + 1.43P_2P_3 - 0.64P_2P_4 - 0.68P_3P_4 - 4.01P_1^2 - 1.00P_2^2 - 6.60P_3^2 - 1.83P_4^2 \quad (3)$$

Where Y is the biodiesel yield, P_1, P_2, P_3 and P_4 represents molar ratio, reaction time, catalyst concentration and temperature respectively.

Table 2 Experimental Process obtained for B. variegate oil

Run	Molar ratio	Reaction time (min)	Catalyst concentration (w%)	Temperature (^0C)	BVME yield (%)	
					Obtained	Predicted
1	6.00	60.00	1.40	55.00	82.4	83.3
2	7.50	75.00	0.80	48.00	72.9	74.2
3	4.50	45.00	0.80	62.00	80.1	80.0
4	7.50	45.00	1.20	48.00	86.1	85.8
5	6.00	60.00	1.00	69.00	93.5	93.1

6	4.50	75.00	0.80	48.00	65	64.7
7	6.00	90.00	1.00	55.00	88.1	88.0
8	7.50	75.00	1.20	62.00	93.5	92.6
9	6.00	60.00	1.00	55.00	94.3	94.0
10	4.50	45.00	0.80	48.00	70	69.8
11	3.00	60.00	1.00	55.00	73.1	74.0
12	9.00	60.00	1.00	55.00	84	86.1
13	6.00	60.00	1.00	55.00	94.3	94.0
14	7.50	45.00	1.20	62.00	92	92.4
15	4.50	75.00	1.20	48.00	82	84.0
16	7.50	75.00	1.20	48.00	90.1	90.7
17	6.00	60.00	0.60	55.00	54	56.0
18	6.00	60.00	1.00	55.00	93.7	94.0
19	6.00	60.00	1.00	55.00	94.8	94.0
20	4.50	45.00	1.20	62.00	87.4	86.9
21	7.50	45.00	0.80	62.00	82	84.0
22	6.00	60.00	1.00	55.00	94.2	94.0
23	4.50	75.00	0.80	62.00	72	74.7
24	4.50	45.00	1.20	48.00	81.4	82.3
25	7.50	45.00	0.80	48.00	73.1	75.0
26	6.00	60.00	1.00	55.00	94.8	94.0
27	6.00	60.00	1.00	41.00	81	82.1
28	7.50	75.00	0.80	62.00	78.1	81.8
29	6.00	30.00	1.00	55.00	93.1	92.4
30	4.50	75.00	1.20	62.00	87	85.4

The model was analyzed by analysis of variance (ANOVA) for obtaining the fitness of the model employing least square method as shown in the Table 3.

Table 3ANOVA for Response Surface Quadratic Model

Source	**Sum of Squares**	**Df**	**Mean Square**	**F value**	**p-value Prob>F**
Model	3077.09	14	219.79	534.12	<0.0001
P1-Methanol:oil	174.42	1	174.42	423.86	<0.0001
P2-Reaction time	19.16	1	19.16	46.81	<0.0001
P3-Catalyst Conc	1108.40	1	1108.40	2693.56	<0.0001
P4-Temperature	243.84	1	243.84	592.57	<0.0001
P1P2	12.78	1	12.78	31.06	<0.0001
P1P3	1.50	1	1.50	3.65	0.0755
P1P4	1.38	1	1.38	3.36	0.0869
P2P3	32.78	1	32.78	79.65	<0.0001
P2P4	6.63	1	6.63	16.11	0.0011
P3P4	7.43	1	7.43	18.05	0.0007

2 P1	440.92	1	440.92	1071.49	<0.0001
2 P2	27.26	1	27.26	66.24	<0.0001
2 P3	1193.66	1	1193.66	2900.75	< 0.0001
2 P4	92.30	1	92.30	224.29	<0.0001
Residual	6.17	15	0.41		
Lack of Fit	5.32	10	0.53	3.11	0.1112 (not significant)
Pure Error	0.85	5	0.17		
Cor total	3083.26	29			

The ANOVA showed that the R^2 value was 0.9980 (a value > 0.75 indicates fitness of the model). The values of adj R^2(0.9961) and pred R^2(0.9897) shows the model is good. The adequate precision value of the model is 88.138 and the value of C.V %0.77 confirms the model's flexibility and reliability. The model F value of 534.12 indicates that the model is significant. The p value of the model was <0.0001 (p <0.05) which is significant and the lack of fit model was found to be insignificant. Thus according to p value (value less than 0.05 indicates the significance level) obtained, model terms P1, P2, P3, P4, P1P2, P2P3, P2P4, P3P4, P^2, P^2, P^2 and P^2 were significant.

The Fig.1 shows a acceptable correlation between the predicted and experimental values of biodiesel production, with a high value of coefficient of determination (R^2= 0.9980).

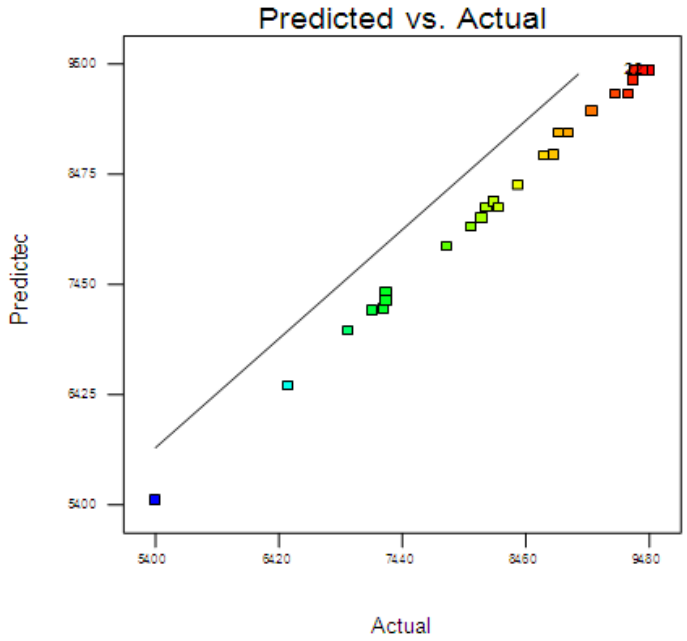

Fig.1 Actual yield vs. predicted yield

BVME yield

Fig. 2 represents the surface plots of conversion to BVME. Fig. 2 (A) represents the significant interation between temperature and catalyst concentration, the BVME yield increases significantly by increasing both temperature and catalyst concentration at 60 min reaction time and 6:1 molar ratio.Further increase the catalyst concentration beyond optimum value saponification or soap formation takes place which reduces the BVME yield. Fig. 2 (B) shows the nature of temperature and reaction time on the BVME yield, the temperature increases the BVME yield increases compare to reaction time. There is no much effect on BVME yield while increasing the reaction time at 6:1 molar ratio of methanol to oil and 1% catalyst concentration.Increase the reaction time and temperature beyond optimum value, reversible transesterification reaction occurs and reduces the BVME yield. Fig. 2(C)shows the mutual interaction between catalyst concentration and reaction time on the BVME yield in which there is an increase in BVME yield for increase in catalyst concentration compares to reaction time at 6:1 molar ratio and temperature of 55°C. The interaction

betweentemperature and molar ratio is shown in Fig. 2(D). The BVME yield was increases proportionally with increase in both variables at 60 min reaction time and 1% catalyst concentration.Fig. 2(E) shows an interaction betweencatalyst concentration and molar ratio, there is a steady increase in BVME yield when molar ratio and catalyst concentrationincreases at 60 min reaction time and temperature of 55°C. Finally, Fig. 2(F) shows interaction between reaction time and molar ratio, there is an increase in molar ratio it increases the BVME yield and at the same time there is no much increase in BVME yield while increase in the reaction time at 1% catalyst concentration and temperature of 55°C.

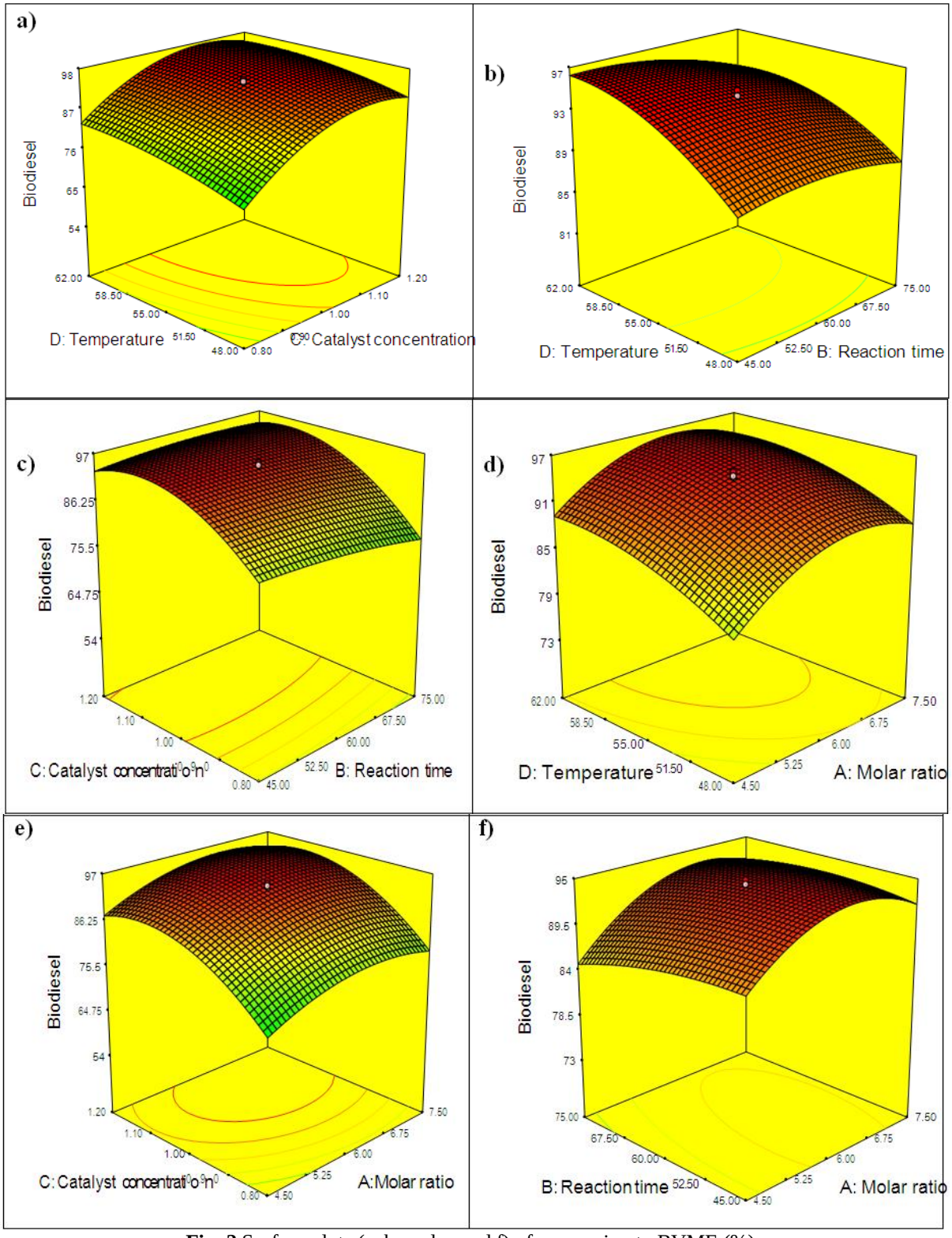

Fig. 2 Surface plots (a, b, c, d, e and f) of conversion to BVME (%)

Optimization of BVME yield

The model predicted the 97.05% of BVME yield at 6.86:1 molar ratio, 65.46 min reaction time, 1.08 wt.%catalyst concentration and 60.38 °C temperature with a desirability of 1.000 as indicated in Fig. 3. Experiments were carried out to validate the optimum conditions predicted by the model. The experimental result obtained was 94.1% biodiesel yield with 3.04% error. It was observed that error between predicted and actual yield was relatively small. Since it can be stated that the optimum conditions are 6.86:1 molar ratio, 65.46 min reaction time, 1.08 wt.%catalyst concentration and 60.38 °C temperature.

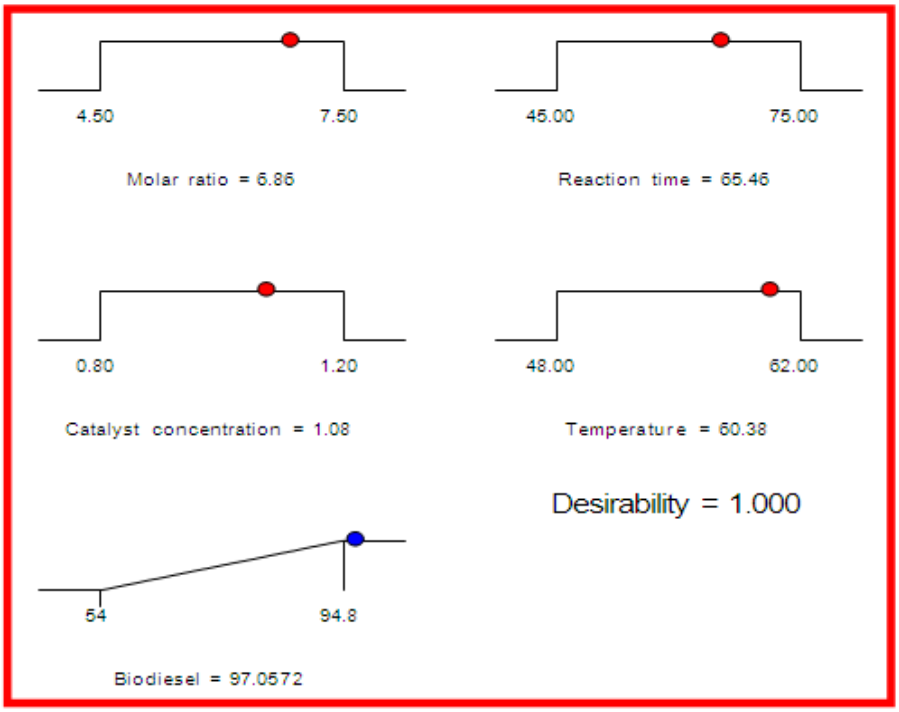

Fig. 3 Numerical optimization parameters for BVME yield.

IV. Fuel Properties of BVME

Table 4 shows the fuel properties of BVME. All the fuel properties such as calorific value, carbon residue, ash content, copper corrosion, viscosity, flash point and density were in the range of ASTM standards.

Table 4 Fuel properties of homogeneous catalyzed biodiesels

Properties	Units	BVME	Biodiesel standard ASTM 6751
Viscosity at 40 ^{o}C	cSt	4.5	1.9-6.0
Density	kg/m^{3}	880	870-900
Flash point	$^{\circ}$C	165	>130
Copperstrip corrosion, 50^{o}C, 3h	-----	1a	no. 3 max
Calorific value	kJ/Kg	39824	-----
Acid value	Mg KOH/g	0.6	0.8 max
Ash content	% mass	Nil	0.050
Carbon residue	% mass	Nil	0.020

BVME = Bauhinia variegate methyl ester

V. Conclusion

Bauhinia variegata seeds identified as potential resource for biodiesel production. The maximum Bauhinia variegate methyl ester or biodiesel yield 94.1% achieved at optimum conditions of6.86:1 molar ratio, 65.46 min reaction time, 1.08 wt.% catalyst concentration,

60.38 °C temperature and constant agitation speed of 650 rpm. The fuel properties of the biodiesel were determined employing the ASTM test method.

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