

Optimization of biodiesel production from Sesamum indicum oil by Taguchi's Technique

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Abstract

In this work, Taguchi and regression analysis method have been applied to estimate the statistical factors of transesterification process and its effect on biodiesel production yield. Three important factors molar ratio, catalyst concentration and reaction temperature have been considered for transesterification. L9 (3x3) orthogonal array was developed and experiments performed correspondence to the orthogonal array. The Taguchi analysis results reveal that the molar ratio is the prime factor affecting biodiesel yield production. Quadratic regression analysis equation has been developed by using ANOVA general model. ANOVA also evaluate the contribution of each factor in biodiesel production. R-Sq value 0.9582 show that our analysis model is 95.82% valid. The final optimize values 8:1, 0.34%, 55°C for molar ratio, catalyst concentration and reaction temperature respectively. The optimized biodiesel production yield corresponding to optimized process parameters is 97.27. The fuel properties of the optimized biodiesel are satisfied the ASTM D7652 criteria. Hence, SOME (Sesame Oil Methyl Ester)

could be recommended as a suitable alternative for fossil diesel fuel in diesel engine applications.

Keywords- Sesame oil; Transesterification; Optimization; Taguchi method; Sesame Oil Methyl Ester.

1. INTRODUCTION

Biodiesel is a non-conventional renewable energy source. It is derived from biological sources like as vegetable based oil, fats of animal and waste cooking oil [1, 3]. Nowadays waste catering oil is identified as the most significant substitute of fossil fuel. Biodiesel has been got more attention because of the advantage over fossil fuel such as higher Cetane number, lower emission, and lower aromatic content [2, 6]. It is also capable of solving the energy crises. The raw material cost of biodiesel is dearer than that of final biodiesel product. Efforts have been made to decrease the prices of biodiesel by choosing low-priced raw materials, enhancing reaction conversion or simplifying the production method to overcome these demerits [3, 2]. A vast accepted growth in this area is the utilization of waste or used oil to decrease the prices of raw material of biodiesel [1, 8]. Inedible oil such as Karajan, Mahua, Jatropha, linseed, rubber seed and soapnut may be substantial sources of biodiesel production [4]. There are several methods of biodiesel production. In which, Transesterification is the most common cost effective method for biodiesel production [1, 4, 8]. Biodiesel is produced by chemically reacting lipids (e.g., animal fat, vegetable oil). It is the process of exchanging the organic alkoxy group of an ester with another organic group of alcohol [7]. These reactions are catalyzed by mixing of an acid (e.g., H_2SO_4) or base catalyst (e.g., KOH, NaOH etc.). The reaction can also be accomplished in the presence of enzymes (biocatalysts) particularly lipases.

Sesame oil comes in the edible category. But it's low cost and poor used as food product make it further uses product as a biodiesel production. It is derived from sesame seeds and belongs to the herbaceous Pedaliaceae family [4]. The economic significance of this crop is widely cultivated in several parts of the world (5 million acres), mainly in tropical areas (basic producers: India, China, Myanmar) and has been adapted to semiarid regions [4]. The global production rate of sesame is 3.3 MT/year [4].

The main objective of this study is to optimize the key factors of the transesterification process of *Sesamum indicum* oil methyl ester. As *Sesamum indicum* oil has not yet been more studied for the biodiesel production, it is considered important to optimize the key process parameters like molar ratio of methanol to oil, catalyst concentration, temperature of reaction, and time of reaction. Furthermore, the properties of *Sesamum indicum* oil and its methyl ester were determined and compared with ASTM D7652 biodiesel standards.

2. Material and methods

2.1 Materials

The Sesamum indicum oil was purchased from Moksha Lifestyle oil supplier at Delhi. Analytical grade chemicals such as methanol 99% and KOH catalyst were purchased from the scientific chemist shop from Patiala. The experimental facilities have been provided by Thapar University, Patiala.

2.2 Transesterification process

Initially, the 250-mL Borosilicate reaction glass was charged with 150gm of Sesamum indicum oil and heated to the set temperature using the simple heater. The desired amount of alcohol (e.g. Methanol, Ethanol etc.) was calculated accordance to its molar ratio (mol/ mol). A fixed quantity of a catalyst (e.g., KOH, NaOH etc.) was dissolved in preheated alcohol using a mixer. When catalyst absolutely dissolved in alcohol solution, the mixture was poured into the preheated Sesamum indicum oil. Parafilm-sealed glass beaker was used to stop the leakage. Then the final solution was kept in water bath shaker machine and set the reaction temperature and RPM of shaking. When the reaction time was completed (i.e. 60 min in the experiment). Then the glass beaker was removed from the water bath shaker. Now keep the products of the reaction into a separating funnel where separation takes place. After separation, two separate layers appeared. Top layer and bottom layer are biodiesel and glycerol respectively. After collection of biodiesel separately, biodiesel was washed two- three times with hot (approximately 45°C) deionized water to remove the impurities from biodiesel as an unreacted catalyst.

2.3 Design of experiments (DOE)

3-factor 3-level experiments are designed by Taguchi method in the form of L9 orthogonal array. Taguchi recommended the loss function to estimate the deviation between the desire and experimental values of performance parameters. The loss function has further been changed into SNR values. Single to noise ratios is the log function of estimated result. It is used to determine the extent of deviation in quality function from the expected value. Three types of SNRs can be used in Taguchi Method that depends on your objective. First one for Larger-the-better (LTB) to maximize the outcome, second is Smaller-the-better (STB) to minimize the outcome and last one is Nominal-the-better (NTB) to normalize the outcome. The SNRs model is given below.

$$\text{Nominal the best- SNR}_i = 10 \log \left(\frac{\bar{y}_i^2}{S_i^2} \right) \quad \text{Eq. (1)}$$

$$\text{Smaller the best- SNR}_i = -10 \log \left(\frac{\sum_{j=1}^n \frac{y_j^2}{n}}{\bar{y}_i^2} \right) \quad \text{Eq. (2)}$$

$$\text{Larger the best- SNR}_i = -10 \log \left(\frac{\sum_{j=1}^n \frac{1}{y_j^2}}{\frac{1}{\bar{y}_i^2}} \right) \quad \text{Eq. (3)}$$

Where

$$y_i = \frac{1}{n} \left(\sum_{j=1}^n y_{ij} \right) \quad \text{(Response mean)}$$

$$S_i^2 = \frac{1}{n-1} \left(\sum_{j=1}^n y_{ij} - \bar{y}_i \right) \quad \text{(Variance)}$$

i = experiment number, j = trial number and n = number of trials.

Table 1.

Biodiesel production process parameter and their level

Independent Variable	Symbol	Level		
		-1	0	1
Alcohol/oil molar ratio (mol/mol)	A	4:01	6:01	8:01
Catalyst concentration (% w/w)	B	0.34	0.50	0.67
Reaction temperature (°C)	C	50	55	60

2.4 Process Control parameters and their levels

Various process parameters affecting the yield of biodiesel production such as catalyst concentration, reaction temperature, molar ratio, types of alcohol, time for reaction, agitation or stirring speed and moisture content. Among these parameters, only three more influencing parameter and their three levels have been taken in this study as shown in Table 1. The effect of these selected parameters on biodiesel yield were found by conducting L9 experiment as per L9 orthogonal array shown in Table 2.

Table 2.
The results of experiments and SNRs value.

Experiment no.	Process control factors			Biodiesel Yield (%)	S/N ratio
	A Molar ratio (mol/mol)	B Catalyst concentration (w/w)	C Reaction temperature (°C)		
1	-1	-1	-1	93.67	39.4320
2	-1	0	0	92.64	39.3360
3	-1	1	1	90.35	39.1186
4	0	-1	0	95.29	39.5809
5	0	0	1	93.95	39.4579
6	0	1	-1	94.05	39.4672
7	1	-1	1	97.15	39.7489
8	1	0	-1	95.82	39.6291
9	1	1	0	96.59	39.6986

3. RESULTS AND DISCUSSION

3.1 Static design analysis of signal to noise ratio (SNR)

The calculated SNR value with respect to experiments are given in Table 2. The effect of process control parameters on average SNR for biodiesel yield are shown in Figure 1. The experimental outcomes show that the Experiment No 7 has the highest value of Biodiesel production yield 97.15% and experiment No 3 has the lowest biodiesel production yield 90.35%. Though the set of process parameter corresponding to experiment No. 7 has the maximum yield, this would not be the optimum stabilized set of parameter. SNRL (The level mean signal-to-noise ratio), has been calculated (shown in Table 3), which is the average of all value of SNRs of an individual control parameter at a specific level. Experimental investigation for parameter A at level -1 have done and SNRL has been found to be 39.3 using the mean of SNRs values corresponding to -1 level 39.4320, 39.3360, 39.1186 is taken from experiment nos. 1, 2 and 3. Similarly, all the values are calculated corresponding to all other parameters with their respective level. Δ SNR is the difference of maximum SNRL to minimum SNRL. Based on the value of delta SNR, rank was given. It shows the most influenced or most effective parameter for biodiesel yield. So, according to Δ SNR rank has been assigned. Rank 1 is given to molar ratio for highest delta, Rank 2 to catalyst concentration and last third rank is given to the reaction temperature.

The optimal values of the independent variables selected for the transesterification of sesame oil were obtained by solving the regression equation (Eq. (2)) using the MINITAB software. The optimal conditions for this process were statistically predicted as A = 8:1, B = 0.34 wt. %, and C = 55°C. The predicted sesame biodiesel

yield under the above set of conditions was $Y = 97.27\%$. In order to verify the prediction of the model, the average biodiesel yield obtained was 98.63% , which is well within the predicted value of the model equation.

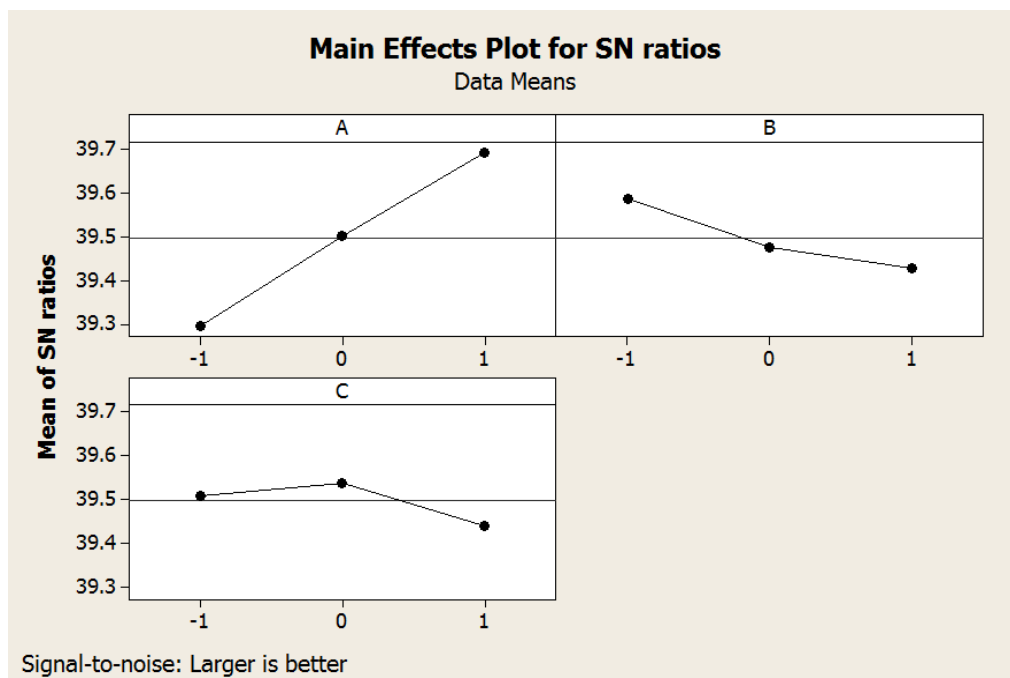


Figure 1. Effect of process control parameters on average SNR for biodiesel yield.

3.2 Consequence of control parameters on yield

The consequence of control parameters on the yield of biodiesel production is shown in Figures 3-5. It is observed that the molar ratio (from Figure 4) contributes significant effect on biodiesel yield. In the beginning (molar ratio 4:1), alcohol is insufficient to react with all alkoxy group of ester so conversion of alkoxy ester into methyl ester is incomplete. When increasing molar ratio then at the optimum condition the complete conversion take place. It is observed from surface plot graph Figure 2. That in the beginning biodiesel production yield increases with reaction temperature and after reaching maximum value it goes down. This effect is shown because methanol is converted into vapor (boiling temperature of methanol is 65°C) at the high temperature. The increasing catalyst concentration effect (from Figure 3) decrease the biodiesel yield. The extra catalyst might cause of soap formation, emulsion, follow-on in the formation of gel with the reaction.

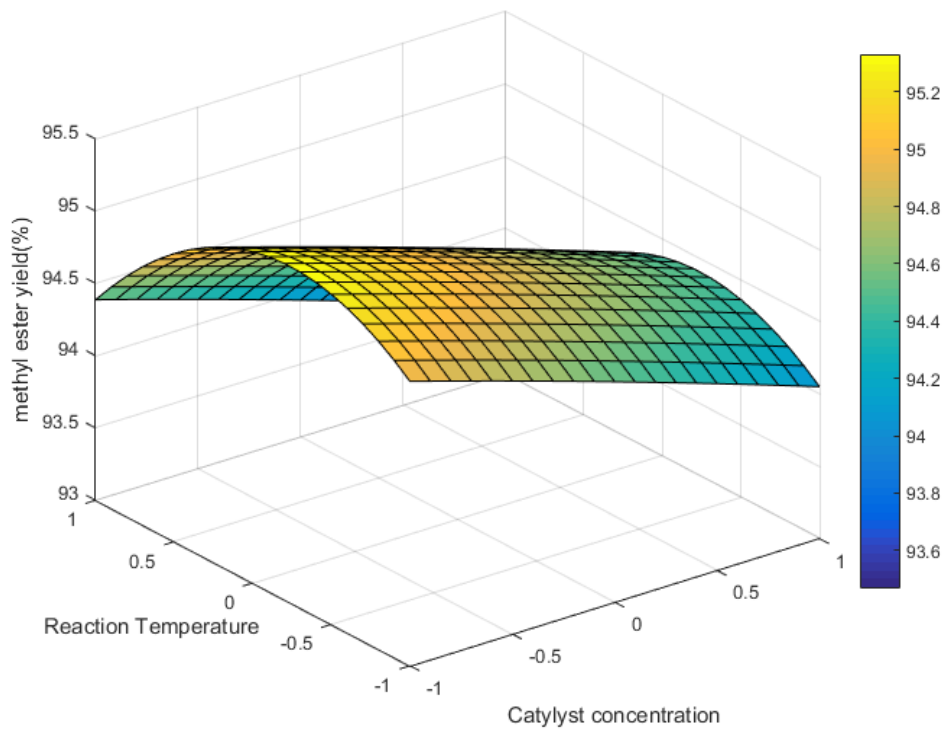


Figure 2. The effect of catalyst concentration and reaction Temperature on methyl ester yield at constant molar ratio 6:1.

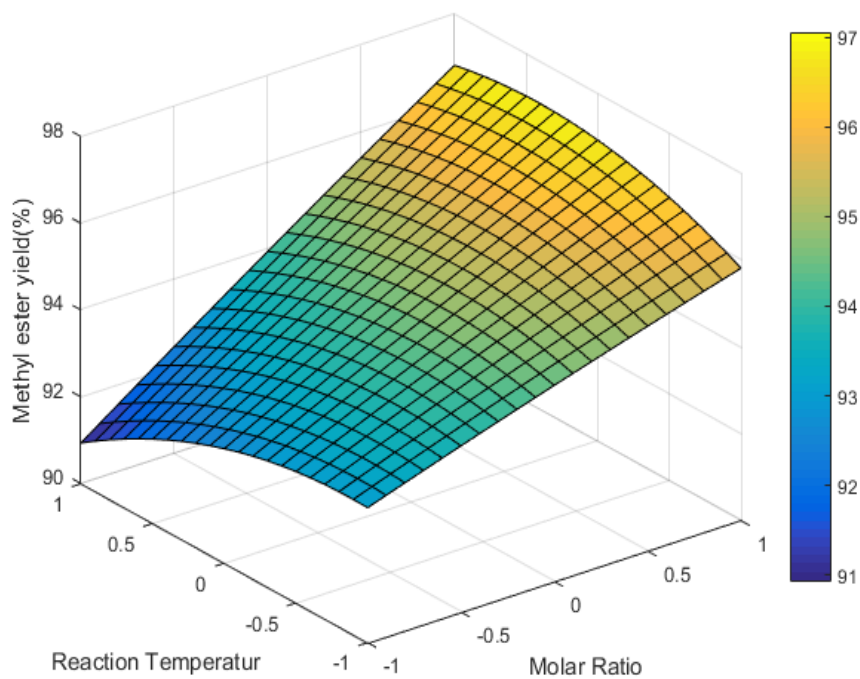


Figure 3. The effect of Molar ratio and reaction temperature on methyl ester yield at constant catalyst concentration 0.5%.

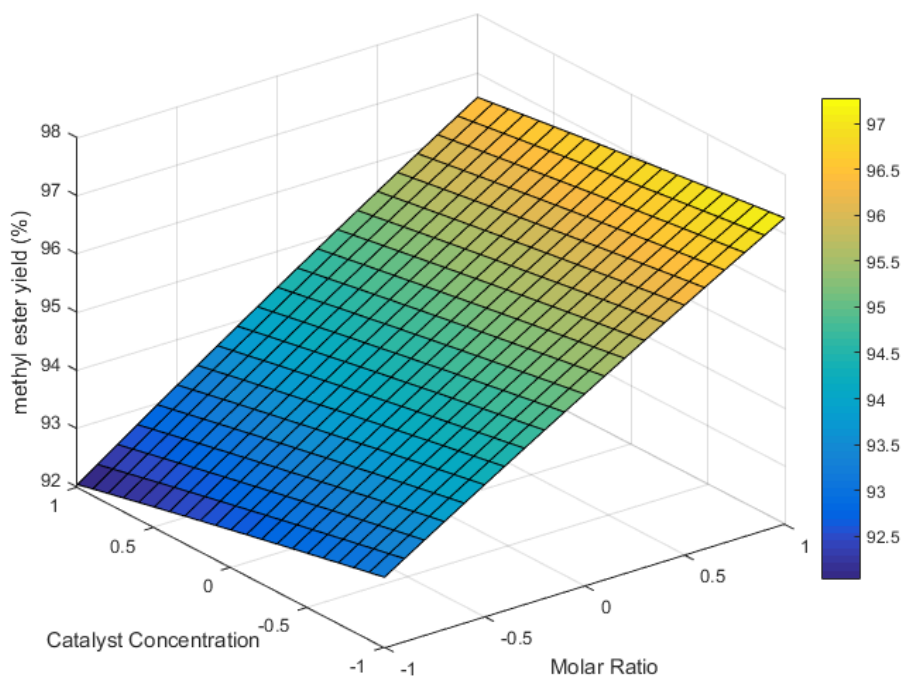


Figure 4. The effect of Molar ratio and catalyst concentration on methyl ester yield at constant reaction temperature 55°C.

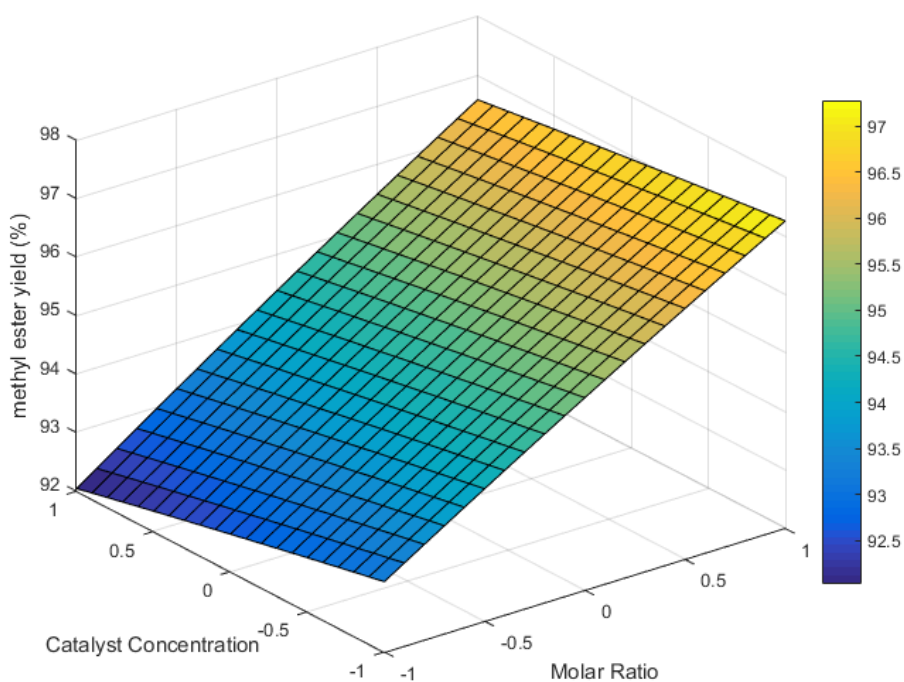


Figure 5. The effect of Molar ratio and catalyst concentration on methyl ester yield at constant reaction temperature 55°C.

3.3 Analysis of variance (ANOVA)

ANOVA is used to compute the response magnitude (in percentage) for each given parameter in the L9 orthogonal array. In this experimental study, ANOVA computes the relation with each parameter of biodiesel production as given in Eq. (4). The most significant parameter is identified and its contribution also identified by using ANOVA. Molar ratio is most significant parameter and its contribution is 78.09% for biodiesel production. Similarly for catalyst concentration and reaction temperature 13.33%, 4.6% respectively.

$$Y_{YIELD} = 94.85 + 2.150A - 0.4578B - 0.2900C - 0.06111A^2 - 0.01556B^2 - 0.6144C^2 + 0.1200AB + 0.7956AC \tag{Eq. (4)}$$

Where A, B and C is the level corresponding to their factor as shown in Table.1

Table 3.
Average S/N for different parameter levels

Level	Control factor		
	Biodiesel yield (% yield)		
	A	B	C
-1	39.3	39.59	39.51
0	39.5	39.47	39.54
1	39.69	39.43	39.44
Delta	0.4	0.16	0.1

Table 4.
Analysis of Variance for R, using Adjusted SS for Tests

Source	DF	Seq SS	Adj SS	Adj MS	F	P
A	2	27.7422	27.7422	13.8711	18.68	0.051
B	2	4.6579	4.6579	2.3289	3.14	0.242
C	2	1.6393	1.6393	0.8196	1.1	0.475
Error	2	1.4849	1.4849	0.7424		
Total	8	35.5242				

$$S = 0.861646 \quad R-Sq = 0.9582 \quad R-Sq(adj) = 830.28\%$$

3.4 Fuel properties of biodiesel

The fuel characteristics of the biodiesel obtained from *Sesamum indicum* oil shown in Table 5. These properties were evaluated according to ASTM D6751 standard

methods. For commercial fuel, the finished biodiesel must be examined using analytical equipment to make sure that it meets the international standards. Kinematic viscosity of SOME at 40°C satisfied ASTM standard. Flash point of SOME was very high. It is good for safety purpose to store the fuel at higher temperature compared to diesel fuel.

Table 5.
Properties of sesame oil and sesame biodiesel.

Parameter	Sesame Oil	Sesame Biodiesel	ASTM D6751
Moisture Content %	0.08	0.017	<0.03
Kinematic Viscosity (mm ² /s) At 40°C	33.34	5.67	1.9 to 6
Density (kg/m ³) at 25°C	842	88.79	-
Higher Heating Value (MJ/kg)	38.25	40.35	-
Flash Point (°C)	285	218	>130
Pour Point (°C)	-4.9	-9.5	-
Cloud Point (°C)	-1.2	-2.9	-

3. CONCLUSION

Based on observation of ester recovery, it was found that Taguchi method (L9 parameter design) given the optimize process parameter of biodiesel production from sesame oil (8:1 molar ratio, 0.34 catalyst concentration and 55°C reaction temperature) and the corresponding yield 97.27. In transesterification process, the contribution factor is higher for molar ratio is 78.09% for ester recovery. Similarly, 13.33 % and 4.6% for catalyst concentration and reaction temperature respectively. A second-order response equation obtained for the biodiesel yield as a function of three variables. The fuel properties of biodiesel yield satisfy the ASTM standard. Hence it is concluded that sesame biodiesel could be considered as potential alternative fuels for Diesel engines application.

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