



OPTIMIZATION OF PROCESS PARAMETERS FOR BIODIESEL PRODUCTION FROM SESAME(*SESAMUM INDICUM*) OIL USING DOE TECHNIQUES

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Abstract: The paper focuses on biodiesel production from sesame oil using transesterification process. Taguchi experimental design is used for the production of sesame oil methyl ester by using process parameters optimization. Taguchi model of the L₁₆ (4 x 4) orthogonal array, signal/noise (S/N) ratio and analysis of variances are employed to determine the optimal process parameters which was effectively used for enhanced yield evaluation. A statistical experimental design was conducted using the Taguchi method and the results obtained were analyzed using statistical software package to predict the optimal yields and parameters for the process. The predictions were analyzed and the most suitable parameters for biodiesel production were selected. The impact of effective four factors including molar ratio, catalyst concentration, temperature and reaction time were elucidated upon the biodiesel production, and the best operating conditions were obtained for the highest production performance using the Taguchi method and MINITAB-17 Software. By maintaining a molar ratio of 6:1 (methanol to oil), 2 wt % of catalyst concentration (KOH), 30 minutes reaction time and 60°C reaction temperature process variables for regression analysis, a predicted yield of 91.56 % can be obtained. Experiments were carried out on the identical process parameters and accomplished a yield of 91.38%. Through this, it is clear that the experimentation and the regression analysis by Taguchi are in acceptable concurrence with an error of 0.8% which can be acclaimed as an experimental error. It was then concluded that catalyst concentration and reaction temperature are significant parameters influencing yield of sesame biodiesel, and the Taguchi optimization approach help predict maximum yield with minimal experiments. The biodiesel SOME produced with the optimized process parameters meets the global standards for biodiesel ASTM D6751 and EN 14214 and hence it could be considered as a suitable substitute for fissile diesel fuel in unmodified diesel engine applications.

Index Terms - Analysis of variances, Transesterification technique, Sesame oil, Biodiesel creation, Optimization, Taguchi technique.

I. INTRODUCTION

Strict diesel emissions standards and the decline in fossil fuels have led researchers to accept alternatives to petroleum products. The increasing use of energy in the transportation and industrial sectors has led to raise in the utilize of fossil fuels, which has necessitated the growth of renewable energy resources. At present, in the local, transportation and industrial fields, the earth's growth activities are increasingly active, leading to the needle-like growth of the widespread use of petroleum-based fuels. According to a report by the International Energy Agency, energy demand in 2025 is expected to be 40-50% higher than current energy consumption. Recently, research has been conducted on biofuels such as biodiesel, alcohol, and hydrogen, which are considered as alternatives to fossil fuels [1,2]. Currently, biodiesel is the most widely accepted alternative to diesel fuel engines because of its technical and environmental advantages. Biodiesel created from animal fats, vegetable oils, or water-based edible oils (WCO) are now being investigated by several scientists for their use. Biodiesel is more acceptable as compared to petroleum diesel owing to its

biodegradability, high cetane number, low carbon monoxide, particulate matter, unburned hydrocarbons, and sulfur content, High viscosity, oxidation, and poor atomization of fuel [3].

Due to the supply of biodiesel increases, interest in non-edible oils such as jatropha, castor, jojoba, tung oil, and sesame oil increases. Amid these oils, sesame oil is a thriving legume tree associated with humid and subtropical climatic conditions with latent for high oil production. It adapts to various soil conditions from strong droughts such as stones and strong sand to wetlands.

Sesame oil is classified as edible oil. However, it is cheaper and can be used as food, so it can also be used for the production of biodiesel. It comes from sesame seeds and belongs to the herbaceous Pedaliaceae family [4]. The cost-effective impact of this crop is that it has been widely planted in numerous regions of the globe (5 million acres), primarily distributed in humid regions (basic producing countries: India, China, Myanmar) and modified to semi-arid regions. [12]. Global productivity of sesame is 3.3 MT / year. [16]

The main purpose of this research work is to optimize key factors in the transesterification method of sesame biodiesel. Since *Sesamum indicum* oil has not so far more investigated for biodiesel creation, it is taken into account to optimize key process factors such as molar ratio, catalyst concentration, reaction temperature, and reaction time etc. In addition, the properties of sesame oil and SOME were measured and compared to the ASTM D7652 biodiesel standard. Using DOE (Taguchi technique), with a set of orthogonal array, the optimal permutation of experimental factors was methodically anticipated from the consequences of 16 experimental trails.

2. Material and Methods

2.1 Materials

The refined *Sesamum indicum* oil was procured from domestic oil manufactures at Pune was used without further processing [3]. Analytical grade chemicals such as methanol 99% and KOH catalyst were purchased from the D Haridas & Company chemist shop from Pune. The experimental facilities have been provided by Rajshree Shau College of Engineering Pune.

2.2 Transesterification of Sesame Oil

Transesterification is the reaction of triglyceride (fat / oil) with an alcohol in the existence of acid, alkaline or lipase as a catalyst to form monoalkyl ester which is biodiesel and glycerol. Sesame oil is filtered with filter paper and used for biodiesel production by the transesterification method. (Fig. 1)

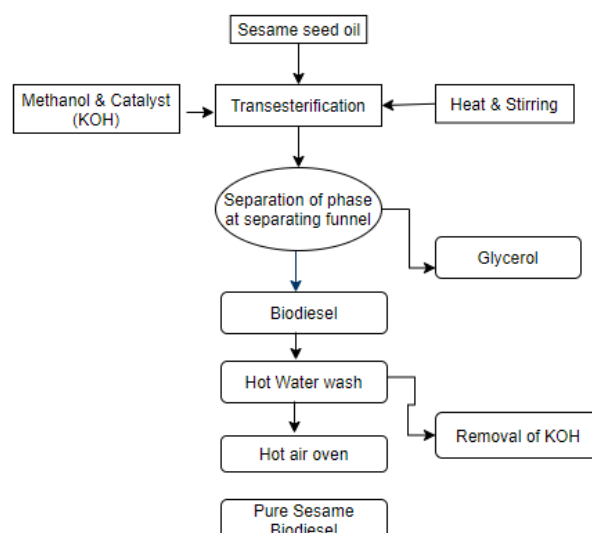


Fig. 1 Flowchart of biodiesel production process

- (1) 150 gm of Sesame oil was taken in a heat resistant glass container 500mL and preheated up to 100–110 °C to remove the moisture from the oil and then allowed to cool to 55°C.
- (2) Now methyl alcohol (CH₃OH) for methanol-to-oil molar ratios of 4.5:1, 5:1 and 6:1 and 7:1 catalyst potassium hydroxide (KOH) at 0.5 wt% (weight percent), 1.0, 1.5 and 2.0 w% of the oil were mixed in concert.
- (3) This identical mixture of methyl alcohol and catalyst KOH was mixed with 250 mL sesame oil.
- (4) The conical flask containing the mixture of oil, alcohol and catalyst (KOH) was heated at a constant temperature of 50–65 °C and stirred at the same time simultaneously inside a water bath shaker at about 300-700 rpm for 60 min, 30 min, 45 min, 60 min and 75 min respectively.
- (5) After finishing point of the reaction time, the products were poured into a separatory funnel and kept 4–5 hr for separation of phases. In the separatory funnel, the products divided into two layers. Owing to higher specific weight, glycerol established at the bottom and the upper layer was biodiesel (Fig. 2c). The glycerol was discarded.
- (6) For refinement of biodiesel a bubble washing method was used. The consequence of different oil to methanol molar ratio and catalyst concentration was observed.

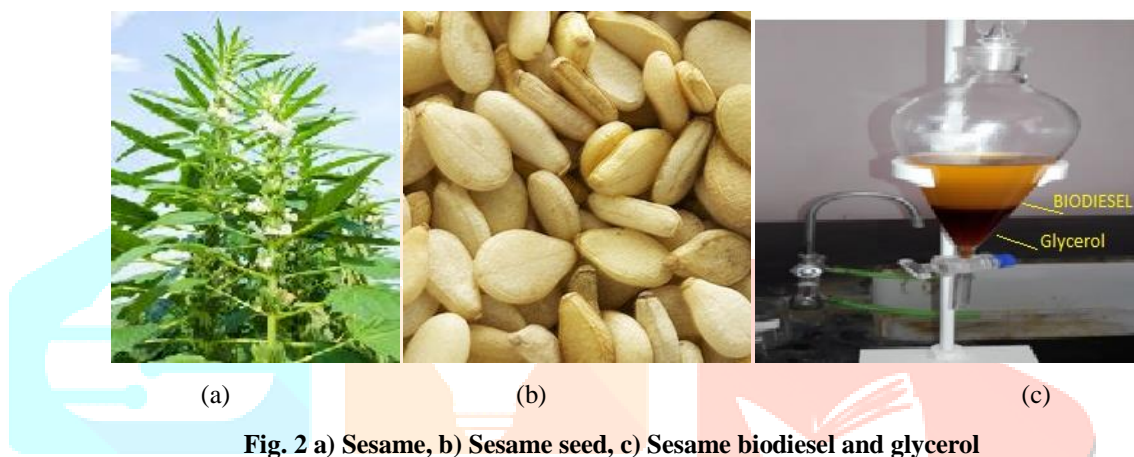


Fig. 2 a) Sesame, b) Sesame seed, c) Sesame biodiesel and glycerol

2.3 Characterization

The obtained sesame biodiesel were tested for different properties by international standard methods [2]. Table 1 shows the several properties, apparatus used and their standards for sesame biodiesel and diesel.

Table 1: Characteristics of Sesame biodiesel in relate with diesel

| S. No. | Property | Diesel | Sesame Oil | Biodiesel (SOME) |
|--------|-----------------------------------|--------|------------|------------------|
| 1 | Specific gravity | 0.83 | 0.88 | 0.85 |
| 2 | Kinematic Viscosity at 40°C (cSt) | 3.72 | 52.5 | 4.72 |
| 3 | Fire point (°C) | 64.00 | 360 | 176 |
| 4 | Calorific value (kJ/kg) | 42500 | 39480 | 38480 |
| 5 | Flash point (°C) | 62 | 345 | 142 |
| 6 | Cetane No | 48 | 36 | 42 |
| 7 | Oxygen Content | - | 11.60% | 11.60% |

3. DESIGN AND ANALYSIS OF EXPERIMENT

3.1 Design of experiment (DOE)

Design of Experiments (DOE) is one of the important and powerful statistical techniques to study the effect of multiple variables simultaneously and involves a series of steps which must follow a certain sequence of experiment to yield an improved understanding of process performance as reported by Taguchi G (1990). By implementing these techniques can reduce the experimental cost with minimum number of experiments. This technique uses orthogonal array for designing the experiments and it can predict the important influencing parameters for biodiesel yield. In the present analysis L₁₆ orthogonal array is chosen for optimization of sesame biodiesel.

By way of imposing these strategies can diminish the experimental value with minimum number of experiments. This approach makes use of orthogonal array for designing the experiments and it may predict the vital influencing factors for biodiesel yield. Within the present analysis L_{16} orthogonal array is chosen for optimization of sesame biodiesel. Eq. (3.1) represents the number of experiments that need to be performed and it is clear that the number of experiments performed depends on the level and the number of elements selected. From this equation it is found that there are 16 sets of experiments that need to be performed to calculate the response parameters [2]:

$$N=(L-1)F+1, \quad (\text{Eq. 3.1})$$

Where: L - chosen level, F – number of factors.

3.2 Optimization by Taguchi

The main objective of the existing analysis is to maximize the sesame biodiesel yield, for this motive taguchi optimization technique became adapted. First of all a sixteen set of experiments had been finished, and yield possibilities were calculated for each case via various the 4 distinct parameters of molar ratio, catalyst attention, reaction time and temperature. The received experimental consequences were furnished in Minitab software and analyzed for larger the best (Eq. (3.4)). Table 2 shows the various parameters those changes in three levels. Table 3 shows the sesame biodiesel yield and SN ratio level (SNRL) for 16 set of experiments:

$$\text{Nominal the best} - SNR_i = 10 \log \left(\frac{\bar{y}_i^2}{s_i^2} \right), \quad (\text{Eq. 3.2})$$

$$\text{Smaller the better} - SNR_i = -10 \log \left(\sum_{j=1}^n \frac{y_j^2}{n} \right), \quad (\text{Eq. 3})$$

$$\text{Larger the better} - SNR_i = -10 \log \frac{1}{n} \left(\sum_{j=1}^n \frac{1}{y_j^2} \right), \quad (\text{Eq. 3.4})$$

Where,

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

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

Table 2: Parameters at three various levels

| S. No. | Parameters | Level-1 | Level-2 | Level-3 | Level-4 |
|--------|-------------------------------------|---------|---------|---------|---------|
| 1 | Molar ratio (methanol to oil ratio) | 4.5 | 5 | 6 | 7 |
| 4 | Reaction temperature | 50 | 55 | 60 | 65 |
| 3 | Reaction time | 30 | 45 | 60 | 75 |
| 2 | Catalyst concentration | 0.5 | 1 | 1.5 | 2 |

Table 3: Orthogonal array for DOE with various parameters

| S. No. | Molar ratio | Catalyst conc. (wt %) | Reaction time (min) | Reaction temp. (°C) | Yield (wt %) | SNRL |
|--------|-------------|-----------------------|---------------------|---------------------|--------------|---------|
| 1 | 4.5:1 | 0.5 | 50 | 30 | 83.88 | 38.4732 |
| 2 | 4.5:1 | 1 | 55 | 45 | 84.45 | 38.5320 |
| 3 | 4.5:1 | 1.5 | 60 | 60 | 86.12 | 38.7021 |
| 4 | 4.5:1 | 2 | 65 | 75 | 89.95 | 39.0800 |
| 5 | 5:1 | 0.5 | 55 | 60 | 80.54 | 38.1202 |
| 6 | 5:1 | 1 | 50 | 75 | 82.11 | 38.2879 |
| 7 | 5:1 | 1.5 | 65 | 30 | 88.34 | 38.9231 |
| 8 | 5:1 | 2 | 60 | 45 | 86.65 | 38.7554 |
| 9 | 6:1 | 0.5 | 60 | 75 | 86.64 | 38.7544 |
| 10 | 6:1 | 1 | 65 | 60 | 83.62 | 38.4462 |
| 11 | 6:1 | 1.5 | 50 | 45 | 84.26 | 38.5124 |
| 12 | 6:1 | 2 | 55 | 30 | 91.38 | 39.2170 |
| 13 | 7:1 | 0.5 | 65 | 45 | 83.36 | 38.4192 |
| 14 | 7:1 | 1 | 60 | 30 | 87.1 | 38.8004 |
| 15 | 7:1 | 1.5 | 55 | 75 | 89.18 | 39.0053 |
| 16 | 7:1 | 2 | 50 | 60 | 90.22 | 39.1061 |

Table 3 shows various levels of catalyst concentration, molar ratio, reaction time, and reaction temperature. Where SNRL represents the algebraic mean of a certain parameter at that level. The delta represents the difference between the maximum and minimum values of a certain parameter and was assigned a rank based on the high and low values of the delta. The catalyst concentrations ranked 1 and is determined as a parameter affecting the yield of sesame biodiesel.

Table 4: Response table for SN ratios (Larger is better)

| Level | Molar Ratio | Cat.Conc. | React Temp °C | React Time |
|-------|-------------|-----------|---------------|------------|
| 1 | 38.70 | 38.44 | 38.59 | 38.85 |
| 2 | 38.52 | 38.52 | 38.72 | 38.55 |
| 3 | 38.73 | 38.79 | 38.75 | 38.59 |
| 4 | 38.83 | 39.04 | 38.72 | 38.78 |
| Delta | 0.31 | 0.60 | 0.16 | 0.30 |
| Rank | 2 | 1 | 4 | 3 |

3.3 Analysis of variance

The SN Ratio represents the ratio of mean value of biodiesel yield to standard deviation. SNR helps in predicting the optimum level of parameters with respect to optimum conditions to maximize the biodiesel yield [1]. The appropriate parameters that affect the biodiesel yield at that certain situation cannot be predicted using SNR. Therefore, statistical analysis of variation is used for sesame biodiesel yield (ANOVA). In this analysis ANOVA is used for estimation for sesame biodiesel and its various parameters that affect sesame biodiesel yield.

Table 5 reveals the several process variables for F and P values. The % contribution was also intended by the contribution factor formulae as shown in the Eq. (5). Higher F-values are important parameters in the preparation of sesame biodiesel and its yield. The contribution factor will also show important process parameters, but this F value will be confirmed again. From Table 5, the maximum F value (9.48) and corresponding low P value (0.049) are important.

$$\% \text{ Contribution factor} = \frac{SS_f}{SS_T} \times 100 \dots \dots \dots \text{(Eq. 3.5)}$$

Where,

$$SS_f = \sum_{j=1}^3 n[(SNR_L)_{fj} - SNR_T]^2$$

$$SS_T = \sum_{i=1}^9 [SNR_i - SNR_T]^2$$

SS_f = sum of the squares for the f^{th} parameter, SS_t = the total sum of the squares of all parameters, N = No. of experiments at level J of factor f.

Table 5: Analysis of Variance for various process parameters

| S. No. | Source | DF | Adj SS | Adj MS | F value | P value |
|--------|----------------|----|---------|--------|---------|---------|
| 1 | Molar | 3 | 19.437 | 6.479 | 2.09 | 0.280 |
| 2 | Catalyst Conc. | 3 | 88.244 | 29.415 | 9.48 | 0.049 |
| 3 | Temp | 3 | 5.492 | 1.831 | 0.59 | 0.662 |
| 4 | Time | 3 | 24.816 | 8.272 | 2.67 | 0.221 |
| 5 | Residual Error | 3 | 9.311 | 3.104 | | |
| 6 | Total | 15 | 147.299 | | | |

3.4 SN Ratio (SNR)

Fig. 3, 4 shows the changes of molar ratio, catalyst concentration, reaction time and reaction temperature in terms of SN ratio. The maximum value in each factor represents the optimum level for sesame biodiesel yield. Therefore, at molar ratio of 7:1, catalyst concentration of 2.0 grams, reaction time of 30 minutes and temperature of 60°C gives the maximum yield for sesame biodiesel.

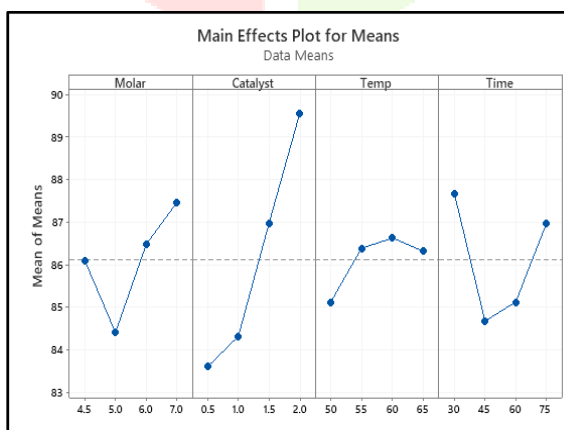


Fig.3. Main Effects Plot for Data means

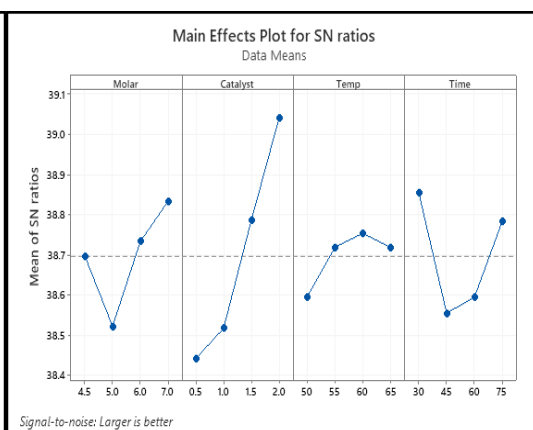


Fig.4. Main Effects Plot for SN ratios

3.5 Regression Equation

Regression equation is algebraic model that establish certain interaction between input and output parameters. Results are obtained with a relatively small amount of error. It gives the effect with a comparatively minute quantity of error. The optimum yield of sesame biodiesel is intended by using the regression equation. From the SN ratio graphs the optimum value of each parameter for the maximum yield are obtained [1]. By putting these optimum values in the regression equation, the optimum yield value is 90.56 %.

$$\% \text{ Yield} = 72.47 + 0.834 \text{ Molar} + 4.098 \text{ Catalyst} + 0.0768 \text{ Temp} - 0.0111 \text{ Time} = 90.56$$

The optimum values from SN ratio plots of molar ratio (6:1), catalyst concentration Eq. (4), reaction time (60 min) and reaction temperature (60 °C) were considered for experimentation. Following these circumstances, the experimentation is executed and for better accuracy the experimentation is repetitive for three trials, the average of three experimental yield is 91.59 % as revealed in the Table 8.

Table 6. Experimental response of yield percentage

| S. No. | Trails | Yield (%) |
|--------|---------|-----------|
| 1 | 1 | 91.15 |
| 2 | 2 | 92.26 |
| 3 | 3 | 91.38 |
| 4 | Average | 91.59 |

CONCLUSION

The DOE (Taguchi) technique has been conceded out for optimizing the transesterification methods for production of biodiesel from sesame oil. The various input variables such as molar ratio, catalyst concentration, reaction temperature, and reaction time have been optimized by using SN ratio depend upon this study, it can be concluded that as follows;

- 1] For maximize the sesame biodiesel yield with min. number of experiments a Taguchi optimization DOE method was proposed. In this, four process variables such as molar ratio, Catalyst concentration, reaction time and temperature were analyzed.
- 2] The process variables were optimized via statistical technique of L_{16} orthogonal array and ANOVA. The optimum values of process variables were: 6:1 molar ratio, 2 grams of catalyst concentration, 30 minutes of reaction time and 60°C of reaction temperature.
- 3] The maximum yield from regression analysis is 90.59 % and by experimentation it is 91.38 % and an error of 0.8 % which is commended to experimental error and it may be established.
- 4] Here R square value is 93.68%, this shows that the model obtained is fitted to real data.
- 5] Various physicochemical properties of sesame biodiesel were measured by using standards. And these properties are nearly similar to standard diesel fuel.
- 6] Therefore, Taguchi optimization analysis with proper process variables are successfully used to maximize the sesame biodiesel yield

An optimum parameter combination for maximum ester conversion rate is obtained by using the analysis of S/N ratio. Hence it is accomplished that sesame biodiesel could be considered as potential substitute fuels for Diesel engines application.

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