Transesterification Studies on Non-Edible and Edible Oil for the Production of Methyl Ester (Biodiesel) – Optimization Using Response Surface Methodology

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Abstract: Present work reports an optimized protocol for the production of biodiesel through alkaline catalyzed transesterification of edible and non edible oils, three principal variables, molar ratio of methanol to oil (6:1, 12:1), catalyst weight % (0.5%-2%), reaction temperature (40°C-65°C) and reaction time (30min-120 min) effecting the yield of alkaline catalyzed production of methyl ester (biodiesel) from neem oil and palm oil were investigated. The rate of transesterification in a batch reactor increased with temperature up to 60°C. The biodiesel with the best yield and quality was produced in case of neem oil methyl ester (NOME), at 9:1 mole ratio and 1wt% of catalyst (KOH) the yield was 92%. For palm oil methyl ester (POME), the best yield was produced in the mole ratio of 10:1, 1.25 wt% of catalyst and the yield was 90%. From the cost estimation it was concluded that the POME was costlier than NOME. Design Expert 12 Software Package used for Optimization of NOME by the Response Surface Methodology.

Keywords: Transesterification, Neem Oil, Palm Oil, Response Surface Methodology (RSM), Cost estimation.

1. INTRODUCTION

Energy has played an important role in economic development of most countries in the world as shown by an evidently high and positive correlation between energy consumption and living standards. At present, most of the world’s primary commercial energy supply is divided into natural and exhaustible petroleum resources.

The vegetable oils and its derivatives have been used as alternative diesel fuels. From the times of petroleum crisis in 1970 the demand and prices has been increased day by day more interests were seen towards substitution of fossil fuels with biodiesel. Also biodiesel production has been in great interests because of concern seen towards world growing environment problems in the last few decades. Now a day’s biodiesel is emerging as an alternative fuel for petroleum diesel. The methods used for production of biodiesel conventionally by pyrolysis, micro emulsification, dilution, transesterification etc. [1, 2]. The use
of non-edible and edible oil methyl esters, as alternative fuels for diesel engines due to the rapid decline in crude oil reserves. The advantages of edible and non-edible oils as diesel fuel are liquid nature portability, heat content (80% of diesel fuel), ready availability and renewability [3, 4].

Biodiesel is produced by a chemical process called transesterification which reduces the high viscosity of vegetable oil to make it suitable to be used in diesel engines (Santori et al, 2012). Transesterification involves the reaction between oil/fat and alcohol in presence of a catalyst. Reaction temperature, catalyst type, molar ratio of alcohol to oil and reaction time were observed to influence the transesterification process to a greater extent [5, 6].

The objective of this paper was to investigate the process variables for biodiesel production from Neem and Palm oil in presence of potassium hydroxide catalyst and to optimize the selected parameters using response surface methodology by Design Expert 12.0 software package.

2. MATERIALS AND METHODS

2.1 Experimental Procedure

2.1.1 Precondition of oil: Preconditioning of oil involved the removal of the moisture and neutralization of free fatty acids (FFA). Usually the present feed (Neem and Palm oil) consist of, less than 5% of FFA, to be neutralized to the formation of soap. For determination of FFA present in the oil taken, titrated the oil sample with 0.1N KOH solution with Phenolphthalein as indicator. Volume in ml of 0.1N KOH required to neutralize 1 gram of oil is called acid value and calculate the amount of KOH required to neutralize the free fatty acids in the oil by using acid value. Thus by adding calculated amount of KOH, free fatty acids are removed in the form of soap. The moisture in oil will be removed by heating the oil sample up to 105-120 °C and maintaining it there for twenty minutes so that all moisture gets evaporated.

2.2 Transesterification reaction:
Transesterification reaction carried out in two stages.

2.2.1 First stage (Acid catalyzed stage):
The free fatty acids can be reduced to esters by two processes viz. hydrolysis, methanol with acid catalyst. The treated oil was heated to 35 °C to melt the solid fats presented in the oil. Methanol of 99% pure was added (0.1 liters/liter of oil) to the heated oil and stirred for five to ten minutes. One milliliter of 95% pure sulfuric acid (H₂SO₄) was added for each liter of oil using a graduated eye dropper. The compound stirred for one hour maintaining the temperature at 35°C. Heating is stopped and the mixture is stirred for another hour. The mixture was allowed to settle for eight hours in a decanter to remove glycerin and chemical water.

2.2.2 Second stage (alkali catalyzed stage):
In the second stage, the product of the reaction in acid stage was used for the base transesterification. The estimated amount of catalyst (KOH) and methanol was added. The mixture was heated and maintained temperature at 55°C and stirred at low speed of 500 to 800rpm for 30minutes. The heating was stopped and the products were cooled and transferred to decanter after completion of reaction and allowed to settle for 8-10hrs.

2.2.3 Purification of products:
At this stage two major products obtained that are glycerin and methyl ester. Each has a substantial amount of the excess methanol that was used in the reaction. The glycerin phase was much denser than methyl ester phase and settled down while methyl ester floated up. These two can be gravity separated and
the glycerin simply drawn off. Bubble wash method was used to remove excess methanol from methyl ester by adding initially one milliliter of phosphoric acid (H₃PO₄) to the washing water. One third water in volume to the oil was bubble washed for two hours. The mixture was allowed to settle in a decanter for one hour and the water was removed. The biodiesel was heated to 100°C to dispense with the traces of water and preserved.

2.3 FFA analysis of Methyl Ester using GC-MS:
Oils are natural products consisting of ester mixtures derived from glycerol (triglyceride), whose chains of fatty acid contain about 14 to 20 carbon atoms with different degrees of unsaturation [8]. The transesterification reaction consists in the conversion of the triglyceride molecules, by means of the action of short chain alcohol, i.e., methanol, ethanol into the corresponding fatty acid esters. According to the source of oil seed, variations in the chemical composition of the oils are expressed by variations in the molar ratio among different fatty acids in the structure. The prepared methyl ester i.e., used neem oil methyl ester and palm oil methyl ester was analyzed by Agilent 6890 series GC-MS system to determine the composition of fatty acids. The methyl ester is mainly formed by transesterification of saturated and monounsaturated fatty acids while the remaining polyunsaturated and some bulk saturated fatty acid are responsible for high viscosity. The higher level of unsaturated fatty acid reduces fuel quality, because of its easy oxidation.

2.4 Effect of process parameters on the production of NOME& POME:
The important process variables that effects transesterification reaction were oil temperature (40-65°C), molar ratio(6:1-12:1),catlyst concentration(0.5-2.0 wt%) and reaction time (30-120min).

2.5 Optimization of NOME:
A factorial design was applied to find out the influence of the operational conditions of the transesterification process, such as methanol to oil molar ratio, KOH content, and reaction time, on mass yield of NOME. The results were analyzed using the response surface method. Process Variables for Optimization using Box-Behnken Design were given in table 1. Methanol to oil molar ratio, catalyst weight% and temperature were selected as independent variables to optimize yield.

| Table 1. Different Process Variables for Optimization using Box-Behnken Design |
|----------------------------------|--------|--------|--------|--------|
| **Name**                        | **Units** | **Type** | **Std. Dev** | **Low**  | **High** |
| Methanol to oil molar ratio     | -       | Factor  | 0          | 8       | 10       |
| Catalyst Weight%                | Wt%     | Factor  | 0          | 0.5     | 1.5      |
| Temperature                     | °C      | Factor  | 0          | 50      | 70       |
| Yield                           | %       | Response | 0.5039    | 86.3    | 92.44    |

3. RESULTS AND DISCUSSION

3.1 FFA Analysis of biodiesel using GC-MS:
The fatty acid composition of the oils seems to have an important role in the performance of the biodiesel in diesel engines. Based on the fatty acid composition and many other parameters, the EU biodiesel specifications will be mandatory to limit the oxidative stability as crucial parameter for injection pump performances.

Vegetable oils are natural products consisting of ester mixtures derived from glycerol (triglyceride), whose chains of fatty acid contain about 14 to 20 carbon atoms with different degrees of un-saturation. The transesterification reaction converts the triglyceride molecules into corresponding fatty acid esters by short
chain alcohol, i.e., methanol, ethanol into the corresponding fatty acid esters. According to the source of oil seed, variations in the chemical composition of the vegetable oil are expressed by variations in the molar ratio among different fatty acids in the structure. The prepared Methyl Esters i.e., Neem methyl ester was analyzed by Agilent 6890 series GC-MS and Palm methyl ester was analyzed by Agilent GC-5973 N GC-MS system to determine the composition of fatty acids. The methyl ester is mainly formed by transesterification of saturated and monounsaturated fatty acids while the remaining polyunsaturated and some bulk saturated fatty acid are responsible for high viscosity. The higher level of unsaturated fatty acid reduces fuel quality, because of its easy oxidation. The GC-MS report of both the methyl esters is shown in figure 1 & 2.

3.2 Influence of Processing Parameters on Biodiesel Yield
3.2.1 Effect of Methanol to Oil Ratio on Methyl Ester Yield: Neem oil methyl ester (NOME) yield was calculated by varying molar ratios of methanol to oil (6:1, 7:1, 8:1, 9:1, 10:1, 11:1 and 12:1), kept constant parameters 1.0 wt. % KOH, 60°C temperature, and 90 min reaction time by experiments. The transesterification reaction depends on the molar concentration of methanol to oil ratio and at 9:1 molar ratio NOME yield was maximum 92%. Palm oil methyl ester (NOME) yield was calculated by varying molar ratios of methanol to oil (6:1, 7:1, 8:1, 9:1, 10:1, 11:1 and 12:1), kept constant parameters 1.0 wt. % KOH, 60°C temperature, and 90 min reaction time by experiments. The transesterification reaction depends on the molar concentration of methanol to oil ratio and at 10:1 molar ratio POME yield was
maximum 89%. So, the optimum methanol to oil ratio for NOME and POME were 92% and 89%. The experimental data was shown graphically in figure 3.

![Figure 3](image_url)

**Figure 3.** Methyl ester yield versus molar ratio

### 3.2.2 Effect of Catalyst (KOH) Weight% on Methyl Ester Yield:

Neem oil methyl ester (NOME) yield was calculated by varying weight% of KOH catalyst (0.5, 0.75, 1.00, 1.25, 1.5, 1.75 and 2.0), kept constant parameters 9:1 molar ratio, 60°C temperature, and 90 min reaction time by experiments. The transesterification reaction depends on catalyst weight % and at 1.25 weight% NOME yield was maximum 92%. NOME yield was 87% at 2.0 weight% decreased due to negative effect of catalyst and soap production increased. Palm oil methyl ester (POME) yield was calculated by varying weight% of KOH catalyst (0.5, 0.75, 1.00, 1.25, 1.5, 1.75 and 2.0), kept constant parameters 9:1 molar ratio, 60°C temperature, and 90 min reaction time by experiments. The transesterification reaction depends on catalyst weight % and at 1.50 weight% POME yield was maximum 87%. POME yield was 85% at 2.0 weight% decreased due to negative effect of catalyst and increases soap production. The experimental data was shown graphically in figure 4.

![Figure 4](image_url)

**Figure 4.** Methyl Ester Yield with Catalyst (KOH) Weight%

### 3.2.3 Effect on Temperature on Methyl Ester Yield:

The important advantage of higher reaction temperature was decrease reaction time. The yield was decreased at higher reaction temperature due to vaporization of methanol. The reaction temperature altered in the experiments 40, 45, 50, 55, 60, 65°C to optimize Methyl ester yield by keeping constant alcohol to oil molar ratio of 9:1, KOH concentration of 1.25wt.% and 90 minutes reaction time for Neem oil. The reaction temperature altered in the experiments 40, 45, 50, 55, 60, 65°C to optimize Methyl ester yield by keeping constant alcohol to oil molar ratio of 10:1, KOH concentration of 1.50wt.% and 90 minutes
reaction time for Palm oil. According to the results Neem oil methyl ester yield increased gradually from 76% at 40°C to 92% at 60°C later decreased due methanol vaporization. Similarly the results Palm oil methyl ester yield increased gradually from 71% at 40°C to 91% at 60°C later decreased due methanol vaporization. So, the optimum reaction temperature for NOME and POME is 60°C. The experimental data was shown graphically in figure 5.

3.2.4 Effect of Reaction Time on Methyl Ester Yield:
These experiments were conducted for the varying reaction time 30, 45, 60, 75, 90, 105 and 120 minutes, keeping 60°C of reaction temperature, 1.25 wt. % of KOH catalyst and 9:1 molar ratio constant for Neem oil.
Experiments were conducted for Palm oil for varying reaction time 30, 45, 60, 75, 90, 105 and 120 minutes, keeping 60°C of reaction temperature, 1.50 wt. % of KOH catalyst and 10:1 molar ratio constant. The experiment values show that longer mixing gives higher yield than using shorter time. So, 90 min of reaction time gave a good result than other reaction times used here. In other words, the methyl ester yields increases with increasing the reaction time. However, based on the results, it shows that the methyl ester yields were lower when reaction time of 120 min was used. This undesirable result may be due to the higher soap formation when longer reaction time was used. Thus, the rate of soap formation was also increased. The yield of Neem and Palm methyl esters were shown in figure 6.

Figure 5. Methyl Ester Yield with Temperature

Figure 6. NOME & POME Yield versus reaction time
3.3 Cost Analysis:
The cost analysis of obtained NOME & POME production compared to the price of conventional diesel fuel as shown in the table 2. For the cost analysis some assumptions have been made to calculate the cost of transesterification and raw material cost (i.e. cost of NOME & POME) to determine the cost for the 1litre biodiesel in laboratory scale. The cost evaluation of selected methyl esters of high free fatty acid content oils, by-product like glycerol is to be considered in the analysis. The raw feed stock is the major component contributing to the cost of biodiesel production. The manufacturing costs included direct costs for oil, filtering, transesterification.

Table 2 Cost analysis of NOME & POME

| Processing input       | Price of output for NOME | Price of output for POME |
|------------------------|--------------------------|--------------------------|
| Seed oil               | Rs 15 per litre of NOME  | Rs 18 per litre of POME  |
| Cost of filtering      | Rs 5 per litre of NOME   | Rs 5 per litre of POME   |
| Cost of Transesterification | Rs 45 per litre of NOME | Rs 45 per litre of POME |
| Total Cost             | Rs 65 per litre of NOME  | Rs 68 per litre of POME  |
| Sell of By-products (Glycerol) | Rs 14 ( Refer note #) | Rs 11 ( Refer note #) |
| Net cost               | Rs 51 per litre of NOME  | Rs 57 per litre of POME  |

#The glycerol obtained during the process of transesterification of 1 liter of NOME & POME is 0.36 ml and 0.28 Therefore, the recovery observed by selling 0.36ml of glycerol is 0.36×40 i.e. Rs.14 and for POME is 0.28×40 is Rs 11.

From the results it was concluded that the POME was costlier than NOME. Further optimization studies were carried out for NOME.

3.4 Optimization of the Selected Parameters Using Box-Behnken Design:
Response surface methodology (RSM) was used to relate the three process variables, Methanol to oil molar ratio, catalyst concentration and temperature with the response (biodiesel yield) to determine the optimal combination of process variables to maximize the biodiesel yield. A three-factor BBM (Box-Behnken Model) based on 17 experimental runs at various numerical values of methanol to oil ratio, catalyst concentration and temperature were conducted to investigate their main interaction contribution on the % of yield keeping RPM and time as constant. The following coded and un-coded levels of independent factors for 17 experiments corresponding to BBM along their responses mentioned below.

The total number of experiments for the BBD was to find out by

\[ N = 2k*(k-1) + C_p \]  
(Eq.1)

Where \( N \) = number of trials; \( k \) = number of process variables; and \( C_p \) = centre points. Absolutely, 17 exploratory runs (4 axial, 8 factorial and 5-center points) were composed in view of the BBD matrix at three unique levels (-1,0,1) of autonomous parameters.

Experimental conditions and observed responses values of Box-Behnken Model with 12 factorial runs, 5-central points, and accordingly regression and quadratic equation was constructed by means of Box-Behnken Model to correlate relationship among approximation and prediction of responses.

The matrix for % Yield with actual and predicted values for each arrangement of response parameters is given in table 3. The centre point was repeated five times to assess the pure error. The quadratic model
portraying the reaction work with relapse coefficients for nitrates expulsion from engineered arrangement is given in the accompanying condition.

Table 3 Comparison of Actual and Predicted values of Neem oil Methyl ester Yield

| Std | Run | A: Molar ratio | B: Catalyst wt% | C: Temperature°C | Actual Yield | Predicted Yield |
|-----|-----|----------------|-----------------|------------------|--------------|-----------------|
| 13  | 1   | 9              | 1.25            | 60               | 92           | 92              |
| 15  | 2   | 9              | 1.25            | 60               | 92           | 92              |
| 1   | 3   | 8              | 1.25            | 60               | 87           | 87.25           |
| 7   | 4   | 8              | 1.25            | 65               | 88           | 88              |
| 3   | 5   | 8              | 1.5             | 60               | 88           | 87.75           |
| 17  | 6   | 9              | 1.25            | 60               | 92           | 92              |
| 14  | 7   | 9              | 1.25            | 60               | 92           | 92              |
| 12  | 8   | 9              | 1.50            | 65               | 90           | 90.25           |
| 2   | 9   | 10             | 1.00            | 60               | 86           | 86.25           |
| 5   | 10  | 8              | 1.25            | 55               | 87           | 87.00           |
| 10  | 11  | 9              | 1.50            | 55               | 90           | 90.25           |
| 8   | 12  | 10             | 1.25            | 65               | 86           | 86.00           |
| 16  | 13  | 9              | 1.25            | 60               | 92           | 92.00           |
| 4   | 14  | 10             | 1.50            | 60               | 87           | 86.75           |
| 9   | 15  | 9              | 1.00            | 55               | 90           | 89.75           |
| 11  | 16  | 9              | 1.00            | 65               | 90           | 89.70           |

Final Equation in Terms of Coded Factors:

Yield = +92.00 -0.5000A +0.2500B +0.0000C +0.0000AB -0.5000AC
+0.0000C -4.00A² -1.00 B² -1.000 C²

(Eq.2)

The equation in terms of coded factors can be used to make predictions about the response for given levels of each factor. By default, the high levels of the factors are coded as +1 and the low levels are coded as -1. The coded equation is useful to identify the relative impact of the factors by comparing the factor coefficients.

Final Equation in Terms of Actual Factors:

Yield = -451.75000 +77.50000 +40.00000 Catalyst weight% +5.70000 Temperature-7.85569E-14 Molar Ratio*Catalyst Weight% +0.10000 Molar Ratio*Temperature -2.53539E-15 Catalyst weight%*Temperature -4.00000Molar Ratio² -16.00000Catalyst weight%² -0.040000Temperature²

(Eq.3)

The equation in terms of actual factors can be used to make predictions about the response for given levels of each factor. Here, the levels should be specified in the original units for each factor. This equation should not be used to determine the relative impact of each factor because the coefficients are scaled to accommodate the units of each factor and the intercept is not at the center of the design space.

3.4.1 Analysis of Variance (ANOVA):

The ANOVA-developed for % yield and results are displayed in Table 4. Quality of the Quadratic Model for the yield using Box-Behnken Design was shown in table 5. The Model F-value of 347.56 implies that the model is significant.
There is only a 0.01% chance that a "Model F-Value" this large could occur due to noise. Values of "Prob. > F" less than 0.0500 indicate model terms are significant. In this case A, B, C, AB, AC, BC, A²B², C² are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve model. “The lack of fit value” of 16.10 implies Lack of fit is significant. There is only a 1.07% chance that a “Lack of fit F-value” this occur due to noise.

**Table 4.** ANOVA Table for Model to predict % yield using Box-Behnken Design ANOVA for Response Surface Quadratic Model Analysis of variance table (Partial sum squares-Type III)

| Source      | Sum of squares | df | Mean squares | F-value | p-value | Significant |
|-------------|----------------|----|--------------|---------|---------|-------------|
| Model       | 83.97          | 9  | 9.33         | 130.62  | <0.001  |             |
| A-Molar ratio | 2.00          | 1  | 2.00         | 28.00   | 0.0011  |             |
| B-Catalyst weight | 0.5000  | 1  | 0.500        | 7.00    | 0.0331  |             |
| C-Temperature | 0.0000        | 1  | 0.0000       | 0.0000  | 1.0000  |             |
| AB          | 0.0000         | 1  | 0.0000       | 0.0000  | 1.0000  |             |
| AC          | 1.0000         | 1  | 1.0000       | 14.00   | 0.0072  |             |
| BC          | 0.0000         | 1  | 0.0000       | 0.0000  | 1.0000  |             |
| A²          | 67.37          | 1  | 67.37        | 943.16  | <0.0001 |             |
| B²          | 4.21           | 1  | 4.21         | 58.95   | 0.001   |             |
| C²          | 4.21           | 1  | 4.21         | 58.95   | 0.001   |             |
| Residual    | 0.5000         | 7  | 0.0714       |         |         |             |
| Lack of Fit | 0.5000         | 3  | 0.1667       |         |         |             |
| Pure error  | 0.0000         | 4  | 0.0000       |         |         |             |

**Table 5.** Quality of the Quadratic Model for the yield using Box-Behnken Design

| Std. Dev. | R² | R² | Adeq. Precision |
|-----------|----|----|-----------------|
| Mean      | 89.18 | 0.9941 | 29.2711 |
| C.V.%     | 0.2997 | 0.9865 | 0.9053 |

The “Predicted R-Squared” of 0.9053 is reasonable agreement with the “Adjusted R-Squared” of 0.9865."Adequate Precision" measures the signal to noise ratio. A ratio greater than 4 is desirable. The ratio obtained 14.27 indicates an adequate. This model can be used to navigate design space. The following figure 7 drawn between predicted and experimental values of yield using Design expert. Almost all points were coincided with each other with very small deviation. The surface contour plots of NOME, between the methanol to oil molar ratio, catalyst concentration and temperature % of yield shown in figures 8, 9 and 10.
Figure 7. Predicted vs Actual Yield

Figure 8. Surface contour plots for the effect of molar ratio and catalyst weight% on yield%.

Figure 9. Surface contour plot for the effect of methanol to oil molar ratio and temperature on yield%.
Figure 10. Surface contour plot for the effect of methanol to catalyst weight and temperature on yield%.

4. CONCLUSION:

This study revealed that Neem oil methyl ester and Palm oil methyl ester produced successfully by alkali-catalyzed transesterification. According to the above experiments, the best operational conditions Molar ratio alcohol to oil 9:1 and 10:1, Catalyst concentration 1.25% w/w and 1.50%w/w, Reaction temperature, 60 °C and 55°C. From the cost analysis it was concluded that the POME was costlier than NOME. Hence further optimization studies were carried out for NOME. Optimization of selected parameters was done by using Response Surface Methodology in Design Expert 12.0 software by Box- Behnken method. The actual yields obtained are fitted with predicted yields. Production of biodiesel from non-edible oils for diesel substitute is particularly important because of the decreasing trend of economical extracted oil reserves and the environmental problems caused due to the use of fossil fuel. Non-edible oils can be an important source for methyl ester production in India as they largely available. Use of non-edible oil helps to improve the biodiesel economics.

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