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Optimization of Biodiesel Production from Pongamia Oil using Taguchi Method

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Abstract*:- Fossil fuels which contribute more to the energy requirement is depleting rapidly as the utilization is increasing day by day. Automobile sector is one of the major consumer of fossil fuels and also pollutes the environment through its emissions. The search for alternate energy source has been increasing; one of the better alternative is biodiesel. The biodiesel replaces the fossil fuel as its properties very close to fossil fuel (diesel). The process used for production of biodiesel is transesterification and the yield of biodiesel from this process varies based on some parameters. This work aims at optimization of transesterification process parameters for the production of biodiesel from Pongamia Oil. Two stages of transesterification are done to produce biodiesel from Pongamia Oil as it has high free fatty acid content. In first stage, the 8.5% free fatty acid is reduced to 1.33% by acid esterification. Then, in second stage biodiesel is produced using alkaline transesterification process. Molar ratio of Methanol to oil, Catalyst concentration, Reaction time and Reaction temperature were the four parameters considered for optimization. Taguchi experimental design is used for Pongamia Methyl Ester (PME) via process parameter optimization. The physiochemical properties and fatty acid methyl ester concentrations were experimentally analyzed. The experimental study revealed that 8:1 Molar ratio of Methanol to oil, 1.5 %(w/w) Catalyst concentration, 75 min Reaction time and 60^{0}C Reaction temperature are the optimal process parameters. The biodiesel PME produced with the optimized process parameters yielded 90.2% of biodiesel by the Taguchi method. Hence from the results obtained through Taguchi optimization and the experiment conducted were very close and the parameters can be used to produce high yield of biodiesel from PME.

Keywords: Pongamia Oil, transesterification, Taguchi method, Biodiesel, optimization.

1. INTRODUCTION

Biodiesel, an alternative fuel for diesel engines, is biodegradable, nontoxic and renewable. Generally, biodiesel is produced via the transesterification of vegetable oils or animal fats with short chain alcohols (e.g., methanol), which is carried out by acid or base catalysis, to form alkyl esters that have properties similar to those of fossil-derived diesel. Biodiesel has gained greater attention because of the advantages such as (i) being renewable and biodegradable, (ii) higher cetane number,(iii) lower emission of carbon monoxide, particulate matters and unburnt hydrocarbon and (iv) lower sulfur and aromatic content. However, still it is not fully replacing fossil diesel, because of disadvantages such as higher NOx emission, higher viscosity, lower oxidative and storage stability which need to be addressed. Through persistent and intensive research, some of the above problems have already been addressed. An antioxidant additive can be used to increase the long term storage stability. Oxygenated, antioxidant and cetane improving additives can be used to reduce the NOx emission [1].

One possible alternative to fossil fuel is the use of oils of plant origin like vegetable oils & tree borne oils seeds. Pongamia is a medium sized tree is found almost throughout India. Pongamia is widely distributed in tropical Asia and it is nonedible oil of Indian origin. It is found mainly in the Western Ghats in India, northern Australia, Fiji and in some regions of Eastern Asia. .The plant is also said to be highly tolerant to salinity and can be grown in various soil textures viz. stony, sandy and clayey. Pongamia can grow in humid as well as subtropical environments with annual rainfall ranging between 500 and 2500 mm. This is one of the reasons for wide availability of this plant species. The tree bears green pods which after some 10 months change to a tan colour. The pods are flat to elliptic, 5-7 cm long and contain 1 or 2 kidney shaped brownish red kernels. The yield of kernels per tree is reported between 8 and 24 kg. .The kernels are white and covered by a thin reddish skin. The composition of typical air dried kernels is: Moisture 19%, Oil 27.5%, and Protein 17.4%. The present production of Pongamia oil approximately is 200 million tons per annum. The time needed by the tree to mature ranges from 4 to 7 years and depending on the size of the tree the yield of kernels per tree is between 8 and 24 kg. India is a tropical country and offers most suitable climate for the growth of Pongamia tree. It is found in abundance in rural areas and forests of entire India, especially in eastern India and Western Ghats [5].

In this study, Pongamia Oil is used as an alternative feedstock for the production of biodiesel. Since, the Pongamia Oil has free fatty acid content, acid transesterification is done in order to bring down the fatty acid content. Next, the Pongamia Methyl Ester (PME) is produced using alkaline transesterification in the presence of KOH as catalyst. The process parameters to produce biodiesel were optimized by using Taguchi method. Molar ratio of Methanol to oil, Catalyst concentration, Reaction time and Reaction temperature were the four parameters considered for optimization. Instead of performing 81 experimental studies to optimize the process parameters in the Taguchi experimental design, 9 experiments were sufficient to achieve high yield of biodiesel.

2. MATERIALS AND METHODOLOGY

2.1. Materials and experimental design

Pongamia Oil was purchased from a retailer shop, 99.9% pure analytical grade methanol; sulphuric acid and potassium hydroxide in pellet form of above 85% purity were used for the biodiesel production. The experimental setup consists of half litre spherical glass beaker, a speed controlled mechanical stirrer, a temperature controlled heater and a digital thermometer. The arrangement of the transesterification reactor is shown in Fig.1

Fig.1: Transesterification reactor

Fig.2: FFA Test

2.2. Transesterification process

Transesterification process is of two methods i.e. one step and two step. In one step transesterification process, alkaline transesterification process is carried out. In two step transesterification process, first acid esterification is done and then alkaline esterification is carried out. One of the main properties of the raw oil to decide about the type of transesterification process such as one step or two step transesterification process is free fatty acid (FFA) content. If the FFA content of the oil is less than 2.5%, then one step transesterification process with a base catalyst should be used and if it exceeds 2.5%, two step transesterification process should be the choice. In this study as the FFA content of Pongamia Oil was 8.5%, two step transesterification method has been adopted.

The FFA content in oil is determined by titration test. In this test, a sample of 2-5g of oil is taken in a conical flask, and then 50ml of ethanol and 4-5 drops of phenolphthalein indicator is added in the flask. It is allowed to heat for about 4-5min and then cooled. Then, it is titrated against 0.1N of KOH solution. The KOH solution is added till permanent pink colour appears as shown in Fig.2. Then, FFA content is calculated. In Pongamia Oil, the FFA content is around 8.5%. Thus, two step transesterification process is carried out.

In acid transesterification process, methanol and sulphuric acid is added to Pongamia Oil in order to reduce the FFA. The molar ratio of 8:1 and acid concentration of 3.5% to the weight of oil is used in order to reduce the FFA from 24.5% to 1.33% by acid transesterification. In this process, for a sample of 500ml of Pongamia Oil, 190ml of methanol and 12.5ml of sulphuric acid is added. The stirrer speed is maintained at 500rpm constantly for proper mixing and it is stirred at a temperature of 58-60^{0}C for 1 hr. Then, it is allowed to settle for about 5-8 hrs. After settling, impurities were drained out and the mixture is taken.

Then, alkaline transesterification is carried out. In this process, oil is pre-heated and stirrer is maintained at 500rpm for constant mixing. The methoxide solution was prepared by dissolving KOH pellets in methanol. Once, the oil reaches certain temperature, methoxide solution is slowly poured in the reactor. Fig 3 shows the chemical kinetics of transesterification process. The major influencing parameters considered for optimization are molar ratio of methanol to oil, catalyst concentration, reaction time and reaction temperature and selected values for these parameters are shown in Table 1

Parameters		Levels		
		1	2	3
A	Methanol to oil (molar ratio)	6:1	8:1	10:1
B	Catalyst Concentration(wt%)	0.5	1	1.5
C	Reaction time(min)	60	75	90
D	Reaction Temperature(^{0}C)	50	55	60

Table 1: Chosen parameters and Levels

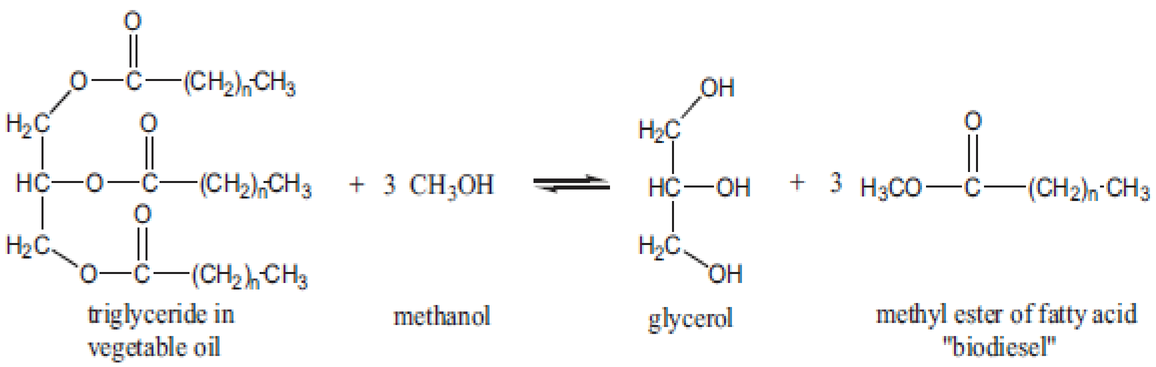

Fig.3.Transesterification reaction

Upon reaching certain reaction time, the mixture is poured into a settling flash and allowed to settle for about 24 hrs. After 24 hrs of settling, the heavy glycerol layer is settled at the bottom of the funnel and drained through a drainage valve. The remaining crude biodiesel is then gently washed with distilled water in order to remove the impurities and unreacted methanol and then it is heated so as to remove all the impurities and water. The percentage of yield of biodiesel has been calculated using the formula:

$$\text{Biodiesel yield } (\%) : Y = \frac{\text{grams of methyl ester produced}}{\text{grams of oil used inreaction}} \times 100$$

2.3. Design of Experiments (DOE) using orthogonal array

Dr. G. Taguchi developed a new method to examine the effect of different parameters of a process on the mean and variance of performance characteristic that determines the proper functioning of the process. This method for design of experiments makes use of orthogonal arrays for the optimization of different parameters influencing the process and the extent to which they can be varied. The very specialty of this method is not to investigate all the possible parameters combinations but only few pairs of combinations. This method paves way for collation of data for the determination of factors which most influence the quality of product with minimal number of experiments so as to reduce precious time and resources. This method is very effective with nominal number of parameters (3–50), few interactions between them and a very few contributing significantly [1].

From the Orthogonal Arrays (OA), the required number of experiments and their conditions can be finalized. The number of parameters and the variation levels of each parameter decide the OA selection. The least possible number of experiments N is decided from the number of levels L and number of design and chosen control parameters P using the relation

$$N = (L - 1) P + 1.$$

2.4. Selection of control parameters and their levels

Among the different parameters influencing the production yield of biodiesel such as reaction temperature, time for reaction, type of alcohol and its quantity, type of catalyst and its concentration, agitation or stirring speed, quality of the reactants and moisture content in the oil, only the four most influencing parameters and three levels (L = 3, P = 4 as shown in Table 1) have been considered in this study. The effects of the four chosen parameters at three different levels have been studied by conducting only nine experiments as per L9 OA shown in Table 2.

2.6. Signal to Noise Ratio (SNR)

Taguchi suggested to use the loss function to calculate the deviation between the experimental value and desired value of performance characteristics. The value of loss function has further been converted into a signal to noise ratio (SNR). SNRs are the log functions of the expected outcome which would be serving as objective of optimization problem. Then SNR is used to calculate the extent of deviation of quality function from the expected value.

There are three types of SNRs used in Taguchi method depending upon the objective of the problem. Larger-the-Better (LTB) for maximization problems, Smaller-the-Better (STB) for minimization problem and Nominal-the-Better (NTB) for normalization problems can be adopted. The SNR (dB) for NTB, STB and LTB models can be calculated as given below.

$$\text{Nominal the best} - \text{SNR}_i = 10 \log \left(\frac{\overline{y_i^2}}{s_i^2} \right)$$

$$\text{Smaller the better} - \text{SNR}_i = -10 \log \left(\sum_{j=1}^{n} \frac{y_j^2}{n} \right)$$

$$\text{Larger the better} - \text{SNR}_i = -10 \log \frac{1}{n} \left(\sum_{j=1}^{n} \frac{1}{y_j^2} \right)$$

SNR based experimental data evaluation has been carried out for the identification of optimal parameter combinations. As the objective is to attain maximum yield of biodiesel, out of the available three different SNR quality characteristics, based on the nature of variables, Larger-the-Better (LTB) has been adopted in the present study. Accordingly the optimal level of control or design parameter will be the level with the highest SNR. By using SNR analysis, it is possible to obtain optimum level of each parameter and optimum set of parameters producing the maximum biodiesel yield [1].

3. RESULTS AND DISCUSSIONS:

3.1. Properties of Pongamia Oil

Pongamia Oil has been used for biodiesel production without any refining process. Its physicochemical properties and fatty acid composition have been studied to find the suitability as feed stock for biodiesel production. Based on its properties and fatty acid content, suitable production process was selected. Table 2 shows the fatty acid composition of different types of saturated and unsaturated fatty acids of Pongamia Oil. The highest content of unsaturated fatty acid and saturated fatty acid was found to be oleic acid and palmitic acid. Table 3 depicts the physicochemical properties of Pongamia Oil. The properties such as density, kinematic viscosity, free fatty acid content (FFA), Acid value, Iodine value, molecular weight were studied.

Fatty acids	**Composition %**
Palmitic acid C16:0	11.8
Stearic acid C18:0	7.8
Oleic acid C18:1	51.8
Linoleic acid C18:2	16.5
Linolenic acid C18:3	2.9
Arachidic C20:0	1.5
Eicosenoic C20:1	1.4
Behenic C22:0	4.9
Lignoceric C24:0	1.4

Table 2: Fatty acid composition of Pongamia Oil

Parameters	Values
Density at 15^0C (g/cm3)	0.933
Kinematic viscosity at 40^0C (mm^2/s)	58.24
Free fatty acid (% FFA as oleic acid)	8.5
Acid value (mg KOH/g)	16.83
Molecular weight (g/mol)	892.7

Table 3: Physicochemical properties of Pongamia Oil

3.2. Determination of optimal experimental condition by Taguchi method

The experiment is based on the Taguchi orthogonal array (OA) L9 method. Four variables (Molar ratio of Methanol to oil, Catalyst concentration, Reaction time and Reaction temperature) are used at three levels to design the experiment in MINITAB 16. The OA based Taguchi method reduces the variance for the experiment by optimizing the process parameters. To identify the combined effect of Molar ratio of Methanol to oil, Catalyst concentration, Reaction time and Reaction temperature on yield, the S/N ratio of the Taguchi method (log functions of the desired output) is used for data analysis and the prediction of the optimum parameters.

The percentage yield of Pongamia Methyl Ester (PME) from Pongamia Oil under designed nine set of experiments; their SNR's are tabulated in Table. In this present work, the maximization of biodiesel yield is set as objective, hence the larger the better (LTB) SNR model has been used. Table 4 shows that experiment 4 has the highest yield of 88.64 and experiment 5 has the lowest yield of 84.28. Though, experiment 5 has the highest yield, it is not the optimum set of parameters. ANOVA is used to determine the effects of the process parameters on PME production. According to this analysis, the most effective parameters are discussed in terms of Molar ratio of Methanol to oil, Catalyst concentration, Reaction time and Reaction temperature.

Experiment	Methanol to oil Ratio	Catalyst Concentration	Reaction Time	Reaction Temperature	Yield%	SNRA
1	6:1	0.5	60	50	84.3	38.5166
2	6:1	1	75	55	86.9	38.7804
3	6:1	1.5	90	60	85.21	38.6098
4	8:1	0.5	75	60	88.64	38.9526
5	8:1	1	90	50	84.28	38.5145
6	8:1	1.5	60	55	87.65	38.855
7	10:1	0.5	90	55	84.35	38.5217
8	10:1	1	60	60	86.54	38.7443
9	10:1	1.5	75	50	85.27	38.6159

Table 4: Percentage of Yield and SNR for 9 experiments

In this method, the S/N ratio is used to measure the deviations of the quality characteristics from the desired value. ANOVA is used to determine the effects of the process parameters on PME production. According to this analysis, the most effective parameters are discussed in terms of Molar ratio of Methanol to oil, Catalyst concentration, Reaction time and Reaction temperature. The effects of different levels of individual parameters on the yield of Pongamia Methyl Ester (PME) are shown in Fig.4. A higher mean S/N ratio indicates a stronger effect of the control parameter at that level on the PME yield. The catalyst concentration exerts the strongest influence on the PME yield. The optimum conditions for the highest PME yield are A2B3C2D3. In order words, based on the S/N ratio, the optimal parameters are A (Methanol to oil ratio) at level 2 (8:1), B (catalyst concentration) at level 2 (1.5 wt. %), C (reaction time) at level 3(75 min) and D (reaction temperature) at level 3 (60^0C). Under these conditions, the PME yield in the confirmation experiment is 90.2%.

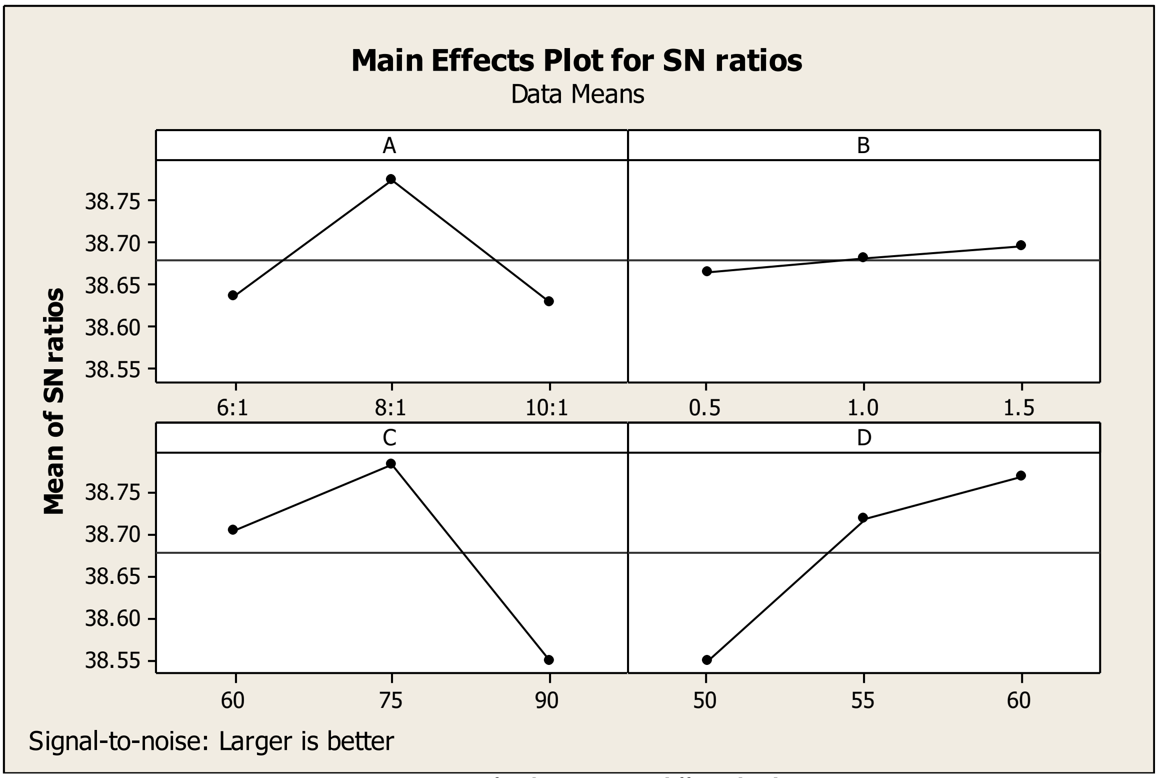

Fig 4: SNR of each parameter at different level

4. CONCLUSION

This paper investigates the optimization and effects of process parameters on the production of Pongamia Oil on the production of Pongamia Methyl Ester (PME).

- ❖ The FFA of Pongamia Oil is 8.5% and is reduced to 1.33% by acid esterification followed by alkaline esterification to produce PME.
- ❖ The series of experiments conducted based on the Taguchi analysis and the yield obtained ranges from 88.64% to 84.28% for the varying parameters.
- ❖ Though DOE the optimum conditions for the production of Pongamia Methyl Ester(PME) are 8:1 Molar ratio of Methanol to oil, 1.5%(w/w)Catalyst concentration, 75 min Reaction time and 60^0C Reaction temperature.
- ❖ The PME obtained by using the optimized parameter resulted in 90.2% yield which is close to the value obtained through Taguchi analysis.
- ❖ The concentration of catalyst and the molar ratio of methanol to oil were identified as the two most important process parameters for the production of Pongamia Methyl Ester (PME).

The tests indicate that it is possible to increase the PME yield significantly using the proposed statistical technique.

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