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Optimization of Effective Parameters of Pongamia Pinnata (Karanja) Biodiesel Using Taguchi Method

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ABSTRACT

Biodiesel is an alternative fuel for diesel engines that is produced by chemically reacting vegetable oil or animal fat with an alcohol such as ethanol or methanol in the presence of catalyst. The growing interest in biodiesel (fatty acid methyl ester or FAME) is because of the similarity of its properties when comparing with diesel fuel. Karanja (Pongamia pinnata), which is one of the source of biodiesel, widely spread all over tropical Asia, Australia, India and locally distributed throughout the state of Maharashtra(India) along the bank of rivers, very common near sea coast in tidal and beach forests in Konkan, along Deccan rivers. Mature seeds of Karanja have recently gained a great commercial relevance owing to their high oil content, which is used as an alternate source of fuel and energy. In this study, the optimization of experimental parameters, such as catalyst type, catalyst concentration, molar ratio of alcohol to oil and reaction temperature, on the transesterification for the production of Karanja methyl ester was performed. Alkali catalyzed method has been used for biodiesel production process by using catalysts such as KOH, NaOH, NaOCH3. The Taguchi method helped to understand the effect of control parameter and to optimize the experimental conditions from a limited number of experiments and contribution of each noise factor calculated by ANOVA. Finally the yield of Karanja methyl ester could be improved using control parameter which was obtained by Taguchi method.

Keywords: ANOVA, Basic catalyst, Biodiesel, FAME, Karanja (Pongamia pinnata), KOME (Karanja oil methyl ester), Taguchi method, Transesterification.

I. INTRODUCTION

Biodiesel is an alternative fuel made from renewable biological sources such as vegetable oils both edible and non edible oil and animal fat. It is derived from triglycerides by transesterification and from the fatty acids by esterification has attracted considerable attention during the past decade as a renewable, biodegradable, eco-friendly and non-toxic fuel. Several processes for biodiesel fuel production have been developed like emulsification, pyrolysis but transesterification is commonly used commercial process to produce clean and environment friendly fuel. Biodiesels such as rapeseed biodiesel, soy bean biodiesel, sunflower biodiesel, Jatropha biodiesel, Karanja biodiesel, rice bran oil, neem oil, sal seed oil, cotton seed oil, palm seed oil, rape seed oil, castor oil, rubber seed oil are some of the biodiesel currently considered as substitute for diesel. Biodiesel from karanja oil shows no corrosion on piston metal and piston liner whereas jatropha curcas has slight corrosive effect on piston liner.[1] Karanja (Pongamia pinnata) is a species of tree in the pea family, native in tropical and temperate Asia including parts of India, China, Japan, Malaysia, Australia and Pacific islands. The extracted oil cannot be used directly in diesel engines because of its high viscosity. High viscosity of pure vegetable oils would reduces the fuel atomization and increase fuel spray penetration, which is responsible for high engine deposits and thickening of lubricating oil. The use of chemically altered or transesterified vegetable oil called biodiesel does not require modification in engine or injection system or fuel lines and is directly possible in any diesel engine.[2]

There has been greater awareness on Biodiesel in India in the recent times due to shortage of fuel and increasing prices. Significant activities have picked up for its production especially with a view to reduce the huge cost involved in import of Petroleum products. In addition, the process of production of Biodiesel from non edible vegetable oil will boost the rural economy and it will provide non-polluting, bio degradable and safe environment.

S. Antony Raja et al[4] prepared a biodiesel from jatropha oil. They carried out transesterification reaction a batch reactor. For reaction 500ml of jatropha oil was heated upto 70°C in a round bottom flask to drawn off moisture and stirred vigorously. Methanol of 99.5% purity having density0.791 g/cm³ was used. 2.5 gram of catalyst NaOH was dissolved in Methanol in bi molar ratio,

in a separate vessel and was poured into round bottom flask while stirring the mixture continuously. The mixture was maintained at atmospheric pressure and 60°C for 60 minutes. After completion of reaction, the mixture was allowed to settle under gravity for 24 hours in a separating funnel. The products formed during transesterification were Jatropha oil methyl ester and Glycerin. The bottom layer consisted of Glycerin, excess alcohol, catalyst, impurities and traces of unreacted oil. The upper layer consisted of biodiesel, alcohol and some soap. The upper layer mixed and washed with hot distilled water to remove the unreacted alcohol; oil and catalyst were allowed to settle under gravity for 25 hours. They found that alkaline catalyzed transesterification shows good result for the production of biodiesel in large scale. Molar ratio of 1:6 of methanol to oil, 0.92% of NaOH catalyst, 60^{o} reaction temperature and 60 minute reaction time observed to be the best combinations of the parameters. Biodiesel characteristics like density, viscosity, flash point, cloud point and pour point are comparable to diesel. SunTaeKim et al[5] optimized the effective parameters of the Rapeseed Methyl ester production. In his study, the optimization of experimental parameters such as catalyst type, catalyst concentration, molar ration of alcohol to oil and reaction temperature on the transesterification for the production of rapeseed methyl ester has been studied. The Taguchi approach, was adopted as the as the experimental design methodology, which was adequate to understand the effects of the control parameters and to optimize the experimental conditions from a limited number of experiments. Finally they obtained the yield of rapeseed methyl ester that was improved to 96.7% with the by optimal conditions of the control parameters which were obtained by Taguchi method. TintTintKywe et al[6]prepared biodiesel from Jatropha oil in pilot plant with capacity of 30 gallons per day. They determined free fatty acid value of Jatropha oil among the chemical properties and the optimal conditions for transesterification reaction using KOH and NaOH. Yield of biodiesel from jatropha oil at optimal sodium hydroxide catalyst concentration 1%, reaction temperature 65°C, reaction time one hour and molar ratio of methanol to oil 6:1 was 92% from lab scale. Yield of biodiesel from jatropha oil at optimal potassium hydroxide catalyst concentration 1%, reaction temperature – room temperature, reaction time 5 hours and molar ratio of ethanol to oil 8:1 was 90% from the lab scale. The fuel properties of biodiesel, namely cetane index, flash point, pour point, kinematic viscosity, specific gravity, color, copper strip corrosion, acid value, water and sediment and distillation at 90% recovery, were found to be within the limits of American Society for Testing and Materials (ASTM) specifications for biodiesel and diesel fuel. R.K.Singh et al[2] prepared karanja oil methyl ester by transesterification using alkali catalyst which gave high level of methyl ester in short duration. For reaction they used 2000ml three necked round bottom flask as a reactor. One of the two side necks was equipped with a condenser and the other was used as a thermowell for temperature measurement. Water bath was used for heating. Karanja oil was converted to biodiesel by esterification followed by transesterification process. The methyl ester produced by these methods compared with diesel fuel to ascertain their suitability. Bobde. S.N et al[3] extracted Karanja oil from three different methods namely by using mechanical expeller method, solvent extraction method and by cold percolation method. They found that the maximum yields were obtained by the Solvent extraction method (31%) as compared to mechanical expeller (24%) and cold percolation method (27%).

II. ABOUT KARANJA

Pongamia pinnata (Karanja) is a medium –sized glabrous tree popularly known as Karanja in Hindi, Indian beech in English and Pongam in Tamil. It is adaptable tree for tropical and sub-tropical regions which requires excellent drainage and a sunny location naturally distributed in tropical and temperate Asian countries like India, Japan, Thailand, Malaysia and north-eastern Australia and in some Pacific islands.

Pongamia is a hardy tree of 12-15 meter height, branches spread into hemispherical crown of dense green leaves. It grows all over India from the coastline to the hilly slopes. It can be grow in different types of flood free soil and matured tree withstands water lodging. Its propagation is by direct seedlings or by planting nursery raised seedlings. Moreover, propagation is also done by branch cuttings and root suckers. Pongamia seeds can immediately be sown after removing from matured pods and start germination after 7 days of sowing and majority seeds germinate well.

Seeds of Pongamia have about 30-35% oil and up to 27-28% oil can be expelled by crusher and most of the physical and chemical properties of the oil are almost similar to those of the diesel. Oil is also used as a lubricant, water paint binder, pesticide and in soap making and tanning industries. It is also a drought resistant plant. Oil cakes are good organic fertilizer and bears nitrogen 4%, phosphorous 1% and potassium 1%, which is better than vermicompost. Root nodules formation due to Rhizobium Strains in nursery and in fields is

common by which nitrogen is replenished in soil. Dense network of lateral roots of Pongamia control soil erosion. Leaves also act as good manure. [3]

III. EXPERIMENTAL SETUP AND MATERIALS

A 500ml beaker was used as reactor. The beaker was placed on magnetic stirrer whose temperature could be controlled within $\pm 2^{o}$C. A separating funnel was used for settling and separating purpose. The karanja crude oil purchased from local market. All reagents for transesterification reaction i.e., catalysts and methanol, pH strips used from Chemistry department of KITS, Ramtek.

IV. TRANSESTERIFICATION PROCESS

Transesterification process is the reaction of triglyceride (fat/oil) with an alcohol in the presence of acidic, alkaline or lipase as a catalyst to form monoalkyl ester that is biodiesel and glycerol. The presence of strong acid or base accelerates the reaction. The main purpose of transesterification is to reduce the high viscosity of oil which is suitable for CI engine. In this study, Karanja methyl ester (KOME) is obtained by reacting karanja oil with methanol in the presence of base catalyst. The karanja oil is first filtered to remove solid impurities then it is preheated at100^{o}C for half an hour to remove moisture. A two stage process is used for transesterification of karanja oil. The first stage is esterification to reduce free fatty acid content in karanja oil with methanol (99% pure) and acid catalyst (98% pure) heated for one hour at 60-65^{o}C in magnetic stirrer. After esterification, the esterified oil washed using water. The washing is carried out in a separating funnel. The hot water having temperature as that of esterified oil added in a separating funnel. Impurities like dust, carbon content, sulfur content is washed away with water. After washing, the esterified oil was fed to the transesterification process. The basic catalyst was dissolved in methanol and added into esterified karanja oil while heating. This mixture is heated for 60 minutes. Once the reaction is complete, it is allowed for settling for 10-12 hours in a separating funnel. The products formed during transesterification were karanja oil methyl ester and glycerin. The bottom layer consists of glycerin, excess alcohol, catalyst impurities and traces of unreacted oil. The upper layer consists of clean amber colored karanja oil methyl ester. After settling, the glycerol layer is removed. The separated biodiesel is taken for characterization. [1], [2],[3],[4],[5],[6]

V. DESIGN OF EXPERIMENT FOR THE OPTIMIZATION OF TRANS-ESTERIFICATION OF KARANJA OIL

Design of experiment consists of a set of experiments which is the setting of several products or process parameters to be studied that are changed from one experiment to another. Design of experiments is also called matrix experiment, parameters are also called factors and parameter settings are also called levels. Conducting matrix experiment using orthogonal array is an important technique. It gives more reliable estimates of factor effects with fewer number of

TABLE 1. Design experiments with four parameters at three levels, for the production of Karanja oil methyl esters.

Parameters	Levels		
	1	2	3
A Molar Ratio (Oil/Methanol)	1:10	1:7	1:5
B Catalyst type	NaOH	KOH	NaOCH3
C Catalyst concentration (wt %)	1	1.5	2
D Reaction temperature (^{o}C)	60	70	80

experiments when compared with the traditional methods such as one factor at a time experiments. The design of experiment via Taguchi method uses a set of orthogonal array for performing of the fewest experiments. Taguchi method involves the determination of large number of experimental situation, described as orthogonal array, to reduce errors and enhance the efficiency and reproducibility of experiments. The columns of an orthogonal array are pair wise orthogonal that is for every pair of column, all combination of factor levels occur at an equal numbers of times. The columns of an orthogonal array represent factors to be studied and the rows represent individual experiments. This study is associated with four factors with each at three levels. The orthogonal array used to find the effects of four parameters namely the molar ratio of oil to methanol, catalyst type, catalyst concentration and reaction temperature on the production of karanja oil methyl ester. The four selected parameters at three levels i.e., L-9 (34), experimentally studied are shown in table1. Table 2 shows the orthogonal array used to design experiments with four parameters at three levels. [5], [9], [10]

In this study, Minitab15, which is software for automatic design, was used to analyze the results and optimize the experimental conditions for setting the control variables.

TABLE 2. Orthogonal array used to design experiments with four parameters at three levels, L-9(3^4)

Experiment No.	Column number and parameters assigned			
	1 Molar ratio (A)	2 Catalyst type (B)	3 Catalyst concentration (wt %) (C)	4 Reaction temperature (^oC) (D)
1	1	1	1	1
2	1	2	2	2
3	1	3	3	3
4	2	1	2	3
5	2	2	3	1
6	2	3	1	2
7	3	1	3	2
8	3	2	1	3
9	3	3	2	1

VI. DETERMINATION OF OPTIMAL EXPERIMENTAL CONDITION BY THE DESIGN OF EXPERIMENT.

The yields of karanja oil methyl ester, prepared under nine sets of experimental conditions are shown in table. All experiments were performed with three repetitions, under the same experimental conditions (e.g., molar ratio of methanol to oil, catalyst type, catalyst concentration and reaction temperature).

TABLE 3. Yield of Karanja methyl ester and S/N ratios for the nine sets of experiments.

Exp. No	Yield of karanja methyl ester				S/N ratio (η)
	Sample 1	Sample 2	Sample 3	Mean	
1	71.4	85.4	80.7	79.16	37.89
2	59.2	62.8	64.5	62.16	35.85
3	50.4	63.8	62.3	58.83	35.24
4	61.1	59.1	60.7	60.30	35.60
5	72.8	67.2	64.8	68.26	35.93
6	62.60	58.20	59.70	60.16	35.57
7	68.00	70.10	69.60	69.23	36.80
8	64.00	61.30	59.50	61.60	35.77
9	57.40	60.40	59.20	59.00	35.41
			Mean	60.00	36.00

In Taguchi method, the signal to noise ratio is used to measure the quality characteristics deviating from the desired value. S/N ratio developed by Genichi Taguchi, is a predictor of quality loss after making certain simple adjustments to the product's function. It isolates the sensitivity of the product's function to noise factors. The signal to noise ratios (S/N) are log functions of desired output, serve as the objective functions for optimization, help in data analysis and the prediction of the optimum results.

Depending upon the objective of the robust parameter design experiment, Taguchi defined three different statistics called *signal to noise ratios.* These ratios were defined as the means to measure the variation of the response with respect to the noise factors.
There are three forms of signal to noise (S/N) ratio that are common interest for optimization of static problems.
Smaller-the-better expressed as

$$\eta = -10\text{Log [mean of sum of squares of measured data]} \quad (1)$$

This is usually the chosen S/N ratio for all the undesirable characteristics like defects for which the ideal value is zero. When an ideal value is finite and its maximum or minimum value is then the difference between the measured data and the ideal value is expected to be as small as possible. Thus, the generic form of S/N ratio becomes,

$$\eta = -10\text{Log [mean of sum of squares of \{measured-ideal\}]} \quad (2)$$

Larger-the-better expressed as

$$\eta = -10\text{Log [mean of sum of squares of reciprocal of measured data]} \quad (3)$$

Nominal-the-better expressed as

$$\eta = -10\text{ Log [square of mean/variance]} \quad (4)$$

This study is associated with four parameters with each at three levels. Above table indicates that the best suitable orthogonal array is L9. Table 2 shows the design matrix for L9. After conducting all the nine experiments and measuring the percentage yields so that there are nine observations in total for each experiment.

According to the analysis for the case of larger the better the mean squared deviations (MSD) of each experiment were evaluated using the following equation

$$\text{MSD} = \frac{1}{n} \sum_{i=1}^{n} \left(\frac{1}{\eta_i}\right)^2 \quad (5)$$

Where n is the number of repetitions of each experiment and η_i the yield of karanja methyl ester. Then the S/N ratio was evaluated using the equation.

$$\text{S/N ratio} = -10\text{Log (MSD)} \quad (6)$$

The effect of parameter level is defined as the deviation it causes from the overall mean. Hence as a first step, calculating the overall mean value of S/N ratio for the experimental region defined by the factor levels in Table 3.

$$\text{Mean (m)} = \frac{1}{n}\sum_{i=1}^{n}(\eta_i) = \frac{1}{9}(\eta_1+\eta_2+\ldots..\eta_9) \quad (7)$$

The signal to noise ratios nine set of experiments shown in Table 3. The mean yield of karanja methyl ester and the S/N ratio were 60.00% and 36.000 respectively. Experiment no. 1 gave the highest mean yield of karanja methyl ester and had the largest S/N ratio.

The mean signal to noise ratio, which was calculated from the effect of the parameters and the interaction at assigned levels, was the average of all the S/N ratios of a setoff control parameters at a given level. For example, the effect of parameter at level A1 (at Experiment 1, 2 and 3) is calculated as

The effect of parameter at level A1 (mA1) = (1/3)x (η1+η2+η3) (8)

Similarly

The effect of parameter at level A2 (mA2) = (1/3)x (η4+η5+η6) (9)

The effect of parameter at level A2(mA3) = (1/3)x (η4+η5+η6) (10)

Using the S/N ratio data available in table the average of each level of the four factors is calculated and listed in Table 4 and mean effect plot of the control parameter shown in Fig. 1.

TABLE 4. Average S/N for different parameter levels

Parameters	Levels		
	1	2	3
A Molar ratio	36.32*	35.70	35.99
B Catalyst type	36.76*	35.85	35.40
C Catalyst concentration	36.29*	35.62	35.99
D Reaction temperature	36.41*	36.07	35.53

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Our goal in this experiment is to maximize the percentage yield of karanja methyl ester. Hence the optimum level for a factor is the level that gives the highest value of η in the experimental region. From table it is observed that the optimum settings of molar ratio, catalyst, catalyst concentration and reaction temperature are A1, B1, C1 and D1. Hence we can conclude that the settings A1B1C1D1 can give the highest η or the highest percentage yield.

Different parameters affect the yield of methyl ester to a different degree. The relative magnitude of the parameter effects are listed in table. A better feel for the relative effect of the different factors is obtained by the decomposition of variance, which is commonly called as analysis of variance (ANOVA). This is obtained by

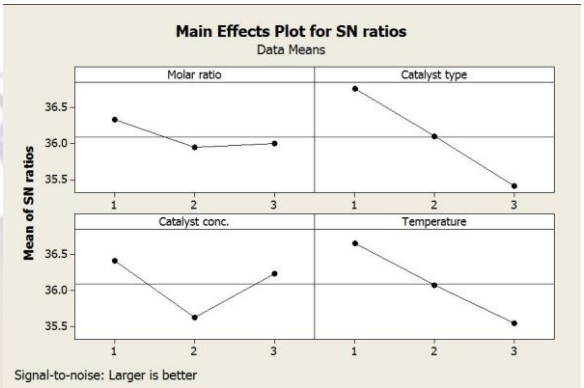

Fig 1. Main effects plot of the control parameters

Sum of square due to parameter A=
[(number of experiments at level A1)x (mA1-m)]+
[(number of experiments at level A2)x (mA2-m)]
+[(number of experiments at level A3)x (mA3-m)]. (11)

Similarly the sum of squares due to parameter B, C and D can be computed as 2.8803, 0.6858 and 1.1817 respectively. Now all these sum of squares are tabulated in Table 5. This is called as the ANOVA table.

In the present study, the degrees of freedom for the error will be zero. Hence an approximate estimate of the error sum of squares is obtained by pooling the sum of squares corresponding to the factors having the lowest mean square. The parameters A and C are used to estimate the error sum of squares. Together they account for four degrees of freedom and their sum of squares is 1.2633.

Referring to the sum of squares in Table 5., the parameter B makes the largest contribution to the total sum of squares [(2.8803/5.3233x100)]. The parameter D makes the next largest contribution (22.02%) to the total sum of squares, whereas the factors A and C together make 10.84% and 12.88% respectively. The larger the contribution of a particular parameter to the total sum of squares, the larger the ability is of that factor to influence S/N ratio. Moreover, the larger F-value, the larger will be the factor effect in comparison to the error mean square.

TABLE 5. ANOVA table for S/N ratio.

Symbol	DOF	Parameters	Sum of squares	Mean square	F	Contribution (%)
A.	2	Molar ratio	0.5775	0.2885	0.90	10.84
B.	2	Catalyst type	2.8803	1.4401	4.56	54.10
C.	2	Catalyst concentration	0.6858	0.3429	1.08	12.88
D.	2	Reaction temperature	1.1817	0.5908	1.87	22.02
Error	0		0			
Total	8		5.3233			
Error	(4)		1.2633	0.3158		

VII. RESULTS AND DISCUSSIONS

The calculated S/N ratio corresponding to nine set of experiments given in Table no. 3. The average S/N ratios of parameter at each level for KOME are shown in Table no. 4. Also the main effect plot for S/N ratio is shown in Fig. 1. The average S/N ratio for maximum percentage yield of KOME is obtained at level 1 (Molar ratio of oil to methanol 1:10), level 1 (catalyst NaOH), level 1 (Catalyst conc. 1%by wt) and level 1 (reaction temperature 60^{o}). i.e., the optimum parameter setting for high percentage yield of KOME is A1B1C1D1. The results of ANOVA for S/N ratios are given in Table no. 5. The contribution of factors in decreasing order for high percentage yield is catalyst type, reaction temperature, catalyst concentration and molar ration. The optimum parameters are obtained from the same level hence validation is not required in this study. The percentage of yield of KOME has been significantly improved using Taguchi Method.

VIII. CONCLUSION

In this study, the optimum parameter for high percentage yield was selected by varying parameters through Taguchi method. With an orthogonal array (L-9) a total set of nine experiments having three parameters each at three levels indicated that the Taguchi method was a efficient method of determining the optimum parameters for high percentage yield of KOME. ANOVA helped to estimate the contribution of each noise factor.

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