

OPTIMIZATION OF MAHUA OIL METHYL ESTER BY USING TAGUCHI EXPERIMENTAL DESIGN

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ABSTRACT

The most commonly used method for biodiesel preparation is via transesterification of vegetable oil using alkaline catalysts. The optimization of experimental parameters, such as catalyst type, catalyst concentration, oil to alcohol molar ratio and reaction time, on the transesterification for the production of Mahua oil methyl ester has been studied. The Taguchi approach (Taguchi method) was adopted as the experimental design methodology, which was adequate for interpreting the effects of the control parameters and to optimize the experimental conditions from a limited number of experiments. The optimal experimental conditions obtained from this study were oil to the alcohol molar ratio 1:15, sodium hydroxide as the catalyst, at a catalyst concentration of 0.4 wt %, and a reaction time of 5 min. According to Taguchi method, the catalyst type played the most important role in the yield of Mahua oil methyl ester.

KEYWORDS: Biodiesel, Taguchi method, Mahua oil, Transesterification reaction, Oscillatory Baffled Reactor

I. INTRODUCTION

Biodiesel, an alternative diesel fuel, is made from renewable biological sources such as vegetable oils and animal fats. It is biodegradable and nontoxic, has low emission profiles and so is environmentally beneficial[1].

Depending on climate and soil conditions, different nations are looking into different vegetable oils for diesel fuel substitute [2]. Biodiesel is produced from renewable biological sources such as vegetable oils and animal fats. Research on vegetable oils as diesel fuel was conducted at least 100 years ago but interest lagged because of cheap and plentiful supplies of petroleum fuels. Periodic increase in petroleum prices due to more demand, stringent emission norms, and feared shortages of petroleum fuels [3]. For this reason, Mahua Oil has significant potential as sources for biodiesel production which delivers an estimated annual production potential of 181 thousand metric tons in India [4].

The transesterification reaction is influenced by several experimental parameters, such as the molar ratio of alcohol to oil, catalysts type and concentration, as well as the reaction conditions. In order to improve yields of biodiesel and the reaction rate, the following experimental parameters have to be considered; the molar ratio of alcohol to oil is one of the most important parameters capable of affecting the yield of biodiesel. Excess alcohol is usually used to shift the equilibrium of the reaction in the direction of the product and to achieve a higher yield from the transesterification of vegetable oil. If the molar ratio is constant, a similar yield of biodiesel will be obtained, regardless of vegetable oil used. The molar ratio is correlated with the type and concentration of catalyst. The transesterification with a basic catalyst is generally much faster than that with an acidic catalyst, but the reaction catalyzed by an acid catalyst results in a very high yield of biodiesel [5, 6].

For the production of biodiesel, the optimum conditions of various parameters have determined by trial and error method. Therefore, to investigate the effects of all combinations of parameters included in experimental research much time and efforts would be needed.

The aim of this study was to optimize the control parameters, such as oil to alcohol molar ratio, catalyst type, catalyst concentration and reaction time, on the transesterification to produce Mahua Oil Methyl Ester. Using an experimental design technique by adopting the Taguchi approach (Taguchi method), with a set of orthogonal arrays, the optimal combination of experimental parameters was systematically estimated from the results of nine experimental runs.

II. MATERIALS AND METHODS

2.1 Materials

Madhuca longifolia, commonly known as mahua or Mahua, is an Indian tropical tree found largely in the central and north Indian plains and forests. The two major species of genus *Madhuca* found in India are *Madhuca Indica* (*latifolia*) and *Madhuca longifolia* (*longifolia*). The seed potential of this tree in India is 500,000 tons and oil potential is 180,000 tons. It is a fast-growing tree that grows to approximately 20 meters in height, possesses evergreen or semi-evergreen foliage, and belongs to the family Sapotaceae. It is found in India in the states of Chhattisgarh, Jharkhand, Uttar Pradesh, Bihar, Maharashtra, Madhya Pradesh, Kerala, Gujarat and Orissa. Oil content in *latifolia* is 46% and 52% in *longifolia*. In seeds oil content is 35% and protein at 16% [7].

2.2 Production of Mahua Oil Methyl Ester

The set of experiments was made using a Taguchi experimental design and randomly conducted to evaluate the four factors at three points each (Table 1). The four factors such as oil to alcohol molar ratio, catalyst type, catalyst concentration and reaction time. The catalyst concentration was compared on a weight basis. The reactions were carried out in Oscillatory Baffled Reactor at room temperature 25°C to 30°C [8]. The velocity of the oscillation was fixed at 138 r/min for all experiments. Mahua oil (100 ml) and the desired methanol and catalyst H₂SO₄ were transferred into the reactor for stage 1 at room temperature. Same runs carried out for stage 2 using different catalyst shown in Table 1. All 9 experiments were run in triplicate. After different time oscillation the reacted mixtures were transferred to separating funnels and the glycerol was separated after settling. The methanol was then removed from methyl ester layer by heating at 40°C for 30 min.

Table 1: Design experiments, with four parameters at three-level, for the production of mahua oil methyl esters

Parameters	Levels		
	1	2	3
A Molar ratio (oil/methanol)	1:9	1:12	1:15
B Catalyst type	KOH	NaOH	NaOCH ₃
C Catalyst conc. (wt %)	0.3	0.4	0.5
D Reaction time (min)	5	10	15

2.3 Design of Experiment for the Optimization of Transesterification of Mahua Oil

The design of experiment used a statistical technique to investigate the effects of various parameters included in experimental study and to determine their optimal combination. The design of the experiment via the Taguchi method uses a set of orthogonal arrays for performing of the few experiments. That is, the Taguchi method involves the determination of a large number of experimental situations, described as orthogonal arrays, to reduce errors and enhance the efficiency and reproducibility of the experiments. Orthogonal arrays are a set of tables of numbers, which can be used to efficiently accomplish optimal experimental designs by considering a number of experimental situations [6].

An experimental design methodology adopting the Taguchi approach was employed in this study, with the orthogonal array design used to screen the effects of four parameters, including the oil to alcohol molar ratio, catalyst type, catalyst concentration and reaction time, on the production of mahua oil methyl esters. The diversity of factors was studied by crossing the orthogonal array of the control parameters, as shown in Table 2 and the numbers indicate the levels of the parameters.

Table 2: Orthogonal array used to design experiments with four parameters at three-levels, L-9

Experiment no.	Parameters and their levels			
	Molar ratio (oil/methanol)	Catalyst type	Catalyst concentration (Wt %)	Reaction time (min)
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

In this study, QUALITEK-4, which is software for the Automatic Design and Analysis of Taguchi Experiments, was used to analyze the results and optimize the experiment conditions for setting the control variables.

III. RESULTS AND DISCUSSION

3.1 Determination of Optimal Experimental Condition by the Design of Experiment

The yields of mahua oil methyl ester prepared under nine sets of experimental conditions are shown in Table 3. From these results, experiment no. 5, which had a mean yield of Mahua oil methyl ester of 95.776%, appeared to have the set experiment conditions with optimal parameters. Experiment no. 1 showed the lowest yield of Mahua oil methyl ester, at 84.066%. However, it is likely that this would not be a preferred way of selecting the optimal conditions using the Taguchi method for the design of an experiment.

Table 3: Yields of mahua oil methyl ester and S/N ratios for the nine sets of experiments

Ex. No.	Yield of Fatty Acid Methyl Ester				S/N Ratio
	Sample 1	Sample 2	Sample 3	Mean	
1	94.19	94.47	97.66	94.44	39.591
2	93.94	94.15	96.88	94.99	39.551
3	74.5	94.6	91.5	86.866	38.629
4	93.25	94.01	97.8	95.02	39.556
5	93.94	94.19	99.2	95.776	39.611
6	90.2	83.8	78.2	84.066	38.448
7	93.88	94.09	96.36	94.776	39.532
8	92.11	93.77	99.65	95.51	39.588
9	90.3	91.5	84.4	88.733	38.945
			Average	92.353	39.277

Generally, a process to be optimized has several control factors which directly decide the target or desired value of the end product. The optimization then involves determining the best control factor levels so that the output is at the target value. A signal-to-noise (S/N) ratio is a performance measure, which calculates the effect of the noise factors on the quality characteristic. These S/N ratios are offered to offer a product design that simultaneously places the response to a target and a minimum variance. The S/N ratios are different in terms of their characteristics, of which there are generally three types, i.e. smaller-the-better, larger-the-better and nominal-the-best.

According to the analysis for the case of 'larger-the-better', the S/N ratio was assessed using the following equation [9].

$$\frac{S}{N} \text{ ratio} = -10 \log \left(\frac{1}{n} \sum_{i=0}^n \left(\frac{1}{y_i} \right)^2 \right)$$

Where n is the number of repetitions of each experiment and Y_i the yield of Mahua oil methyl ester. The S/N ratios for the nine sets of experiments are also shown in Table 3. The mean yield of Mahua oil methyl ester and the S/N ratio were 92.353% and 39.272, respectively. Experiment no. 5 gave the highest mean yield of Mahua oil methyl ester. The relationship between the yield of Mahua oil methyl ester and the S/N ratio was also similarly observed in other experiments.

The mean S/N ratio, which was calculated from the gist of the parameters and the interactions at assigned levels, was the average of all the S/N ratios of a set of control parameters at a given point. The distributions for the four influential parameters are summarized in Table 4. The contribution of an experimental parameter was calculated from the maximum difference in the values between the mean S/N ratios at each stage. The order of influence of the parameters in terms of the yield of rapeseed methyl ester was: B (catalyst type) > D (reaction time) > A (molar ratio) > C (catalyst concentration). In order to more systematically perform an analysis of the relative importance of each parameter, an analysis of variance (ANOVA) was used to optimize the results obtained using Taguchi method. This provided information on the relative influence of parameters and their interactions with respect to the various results. According to the ANOVA results, the most influential parameter in the production of Mahua oil methyl ester was the catalyst type shown in figure 1.

Table 4: Mean S/N ratio at a given level, the difference between two levels, and the distribution

Parameters	Level			Difference		
	1	2	3	L ₂₋₁	L ₃₋₁	L ₃₋₂
Molar ratio (oil/methanol)	39.257	39.205	39.355	- 0.52	0.097	0.149
Catalyst type	39.558	39.585	38.674	0.027	0.885	- 0.912
Catalyst concentration	39.209	39.348	39.259	0.138	0.049	-0.089
Reaction Time	39.384	39.177	39.256	-0.208	-0.129	0.079

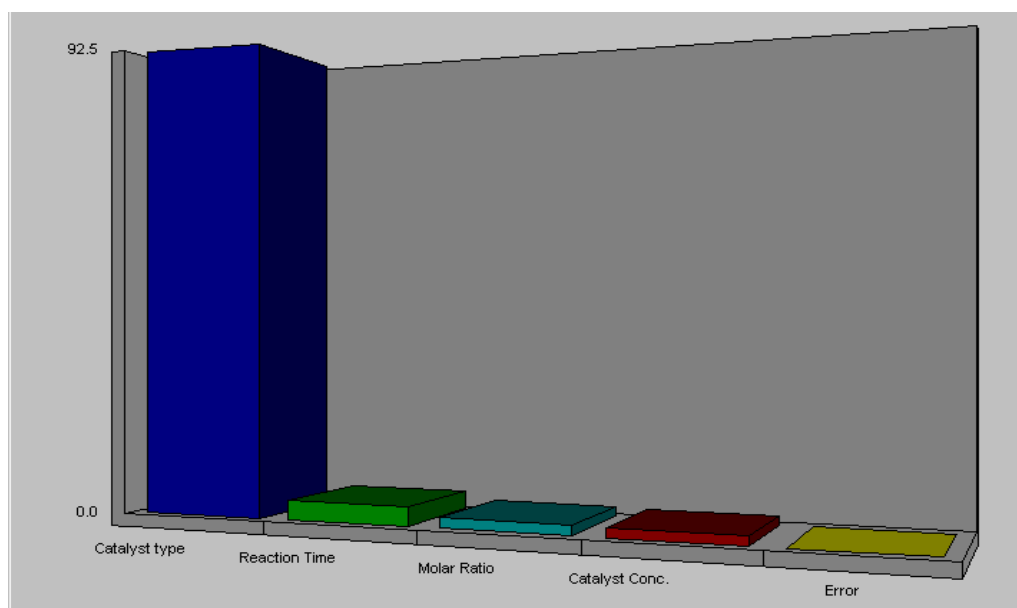


Figure 1: Significant Factor and Interaction Influence

3.2 Result of the Proposed Optimized Experimental Condition and Validation Studies

The optimum conditions to achieve effective performance for the production of Mahua oil methyl ester and the contributions of each parameter to the S/N ratio under the optimal conditions are shown in Table 5. The catalyst type was found to be the most important parameter in influencing the production of Mahua oil methyl ester and the parameter contribution under the optimized conditions

was found to be 0.313. The molar ratio had the least impact of all the parameters studied, with a parameter contribution of 0.082.

Table 5: Optimum conditions for settling the control parameters and their contributions

Parameters		Level	Level description	Contribution
A	Molar Ratio	3	1:15	0.082
B	Catalyst type	2	NaOH	0.313
C	Catalyst concentration	3	0.4 wt %	0.076
D	Reaction temperature	1	5 min	0.111
Total contribution from all factors				1.272
Current grand average of performance				37.679
Expected result under optimum conditions				38.951

IV. FUTURE WORK

The work can be further extended to innumerable situations in industrial processes for estimation of multiple parameters in production of Biodiesel. The ester of this oil can be used as environment friendly alternative fuel for diesel engine creating a greener environment in the future. Its optimization can be easily carried out with the use of optimization tool Taguchi Methodology.

V. CONCLUSIONS

The Taguchi method, which uses a set of orthogonal arrays for performing the fewest experiments, was employed to design experimental trials, with an ANOVA performed to more systematically analyze the relative importance of each experimental parameter in the production of Mahua oil methyl ester.

The Taguchi method provided a systematic and efficient mathematical approach to evaluate and optimize the process for the production of Mahua oil methyl ester, using only a few well-defined experimental sets for the optimization of the design parameters.

The all four parameters i.e. molar ratio, catalyst concentration, catalyst type and reaction time were found to be significant parameters affecting the production of Mahua oil methyl ester. The contribution of the catalyst type in the production process was larger than that of any other parameter. The yield of Mahua oil methyl ester obtained with the optimal experimental parameters was greater than that obtained from experiment no. 5, which gave the highest yield from the experimental trials, and the theoretically expected value.

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