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Process parameters optimization for synthesis of methyl ester from sunflower oil using Taguchi technique

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In this work, transesterification of sunflower oil for obtaining biodiesel was studied. Taguchi's methodology (L_9 orthogonal array) was selected to optimize the most significant variables (methanol, catalyst concentration and stirrer speed) in transesterification process. Experiments have conducted based on development of L_9 orthogonal array by using Taguchi technique. Analysis of Variance (ANOVA) and the regression equations were used to find the optimum yield of sunflower methyl ester under the influence of methanol, catalyst & stirrer speed. The study resulted in a maximum yield of sun flower methyl ester as 96% with the optimal conditions of methanol 110 ml with 0.5% by wt. of sodium hydroxide (NaOH) stirred at 1200 rpm. The yield was analyzed on the basis of "larger is better". Finally, confirmation tests were carried out to verify the experimental results.

Keywords : Sunflower oil, Transesterification, Taguchi technique, Analysis of variance

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1. Introduction:

Biodiesel obtained from energy crops produces favourable effects on the environment, such as decrease in acid rain and in the greenhouse effect caused by the combustion. Due to these factors and to its biodegradability, the production of biodiesel is considered as an advantage over fossil fuels. In addition to this, it also shows a decrease in the emission of harmful gases and unburned hydrocarbons during the combustion [1]. Sunflower, the fourth source of oil seeds worldwide [2] is one of the most widely used vegetable oils because of its nutritional and industrial attributes. The biodiesel can be produced by either direct use or blending of vegetable oil with solvents and physio-chemical methods, such as micro-emulsification, pyrolysis and transesterification [3]. Transesterification is the process of reacting a triglyceride such as one of the vegetable oils with alcohol in the presence of a catalyst to produce fatty acid esters and glycerol [4]. The parameters such as quantity of methanol, catalyst type and concentration, reaction temperature and stirrer speed can prejudice the transesterification reaction. So, these experimental parameters can be considered to be in a constant temperature of 65-70°C to improve the yields of biodiesel and reaction rate. Taguchi has proposed a technique concerning design of experiment to investigate how the different parameters affect the mean and variance of a process.

The design proposed by Taguchi technique employs orthogonal arrays to organize the process control parameters with a minimum amount of experimentation by saving time and resources. The present work aims at optimizing the control parameters of the transesterification process such as methanol quantity, catalyst concentration and stirrer speed for the production of sunflower methyl ester (SFME). Following the experimental design technique of Taguchi with a set of orthogonal arrays, the optimal combination of experimental parameters were systematically estimated from the results of nine experimental runs [5], which had the equivalent effects of 27 experimental runs.

2. Materials and Methods:

A. Materials:

The sunflower seeds which contain the huge proportion of oil found in a fruit [6] were crushed using a commercial grinder and fed to an extractor fitted with a round bottomed flask. The extraction was purified by conventional filtration. The solvent was removed at 45^0C under vacuum using a rotary evaporator [7]. The base catalyst used for the process is sodium hydroxide (NaOH) because it shows largest activity [8] and the methanol was considered as the alcohol.

B. Methods and preparation of SFME:

Transesterification of sunflower oil is nothing but replacing the glycerol of triglycerides with a short chain alcohol in the presence of a catalyst which can be shown in the following reaction.

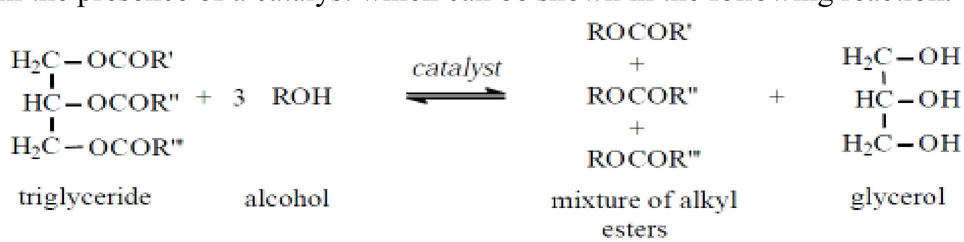

Figure (1): Transesterification reaction

The process was carried out by dissolving the NaOH catalyst in methanol, under low temperature conditions and atmospheric pressure. The process was established based on previous works [9] and preliminary experiments in the laboratory. The crude sunflower oil of 250 ml was preheated to 65°C for 30 min to increase reaction capacity with alcohol. Then methanol of 100 ml was taken in beaker and sodium hydroxide of 0.92 g (0.4% by wt) as a base catalyst was added and stirred vigorously for 30 min in order to dissolve the catalyst into the alcohol. The sunflower oil is transferred into the biodiesel reactor and then catalyst/alcohol mixture is pumped into it. The final mixture was stirred vigorously for 1 hour poured into separating funnel and kept for 15-20 hours when glycerol got settled at bottom of separating funnel leaving ester at the top. Ester was separated from glycerol and it was washed with distilled water to remove excess alcohol, sodium hydroxide and soap (glycerol). Finally, product obtained was heated to 60°C for 20 min to remove the moisture content completely from SFME.

3. Design of experiment:

To assess the two or more parameters which affect the transesterification process was found out by experimental design methodology adopting the Taguchi approach, with the orthogonal array design. The L_9 (3^3), orthogonal array [10] was employed which requires three process parameters and three levels. The molar ratio (oil/methanol) for the quantity of methanol 90 ml as 2.78: 1, 100 ml as 2.5:1 and 110 ml as 2.27:1. The process parameters and their levels are presented in Table (1).

Table (1): Process parameters and levels

Level	Methanol (ml)	Catalyst concentration (gm)	Stirrer speed (rpm)
1	90	0.69	1000
2	100	0.92	1100
3	110	1.15	1200

An orthogonal array consists of a set of tables, which can be used effectively to accomplish optimal experimental designs by considering a number of experimental situations. In this work, the "larger is better" quality characteristics were considered for producing the maximum yield. The L_9 (3^3) orthogonal array chosen in the study is given in Table (2).

Table (2): Orthogonal array $L_9(3^3)$

L_9 (3^3)	A	B	C
1	1	1	1
2	1	2	2
3	1	3	3
4	2	1	2
5	2	2	3
6	2	3	1
7	3	1	3
8	3	2	1
9	3	3	2

The process parameters selected for this experiment were methanol indicated by column A, catalyst concentration and stirrer speed indicated by column B & C respectively in the orthogonal array table. The experiment consists of 9 tests (each row in the L_9 orthogonal array) and the columns were assigned with parameters. The experiments were conducted as per the orthogonal array with level of parameters given in each array row. The experimental output to be studied is yield of test samples and the observations are further transformed into signal to noise (S/N) ratio. The response to be studied with the objective as larger is better, in Eq.(1) is calculated as logarithmic transformation of loss function as shown

$$S/N = -10\log(\sum (1/Y_i^2)/n) \tag{1}$$

Where "n" is the number of observations, "Yi" is the measured value of yield. It is suggested that quality characteristics are optimised when the S/N response is as larger is better.

4. Results and Discussion:

Experimental values of yield and the calculated values of S/N ratio for a given response are listed in Table (3). The S/N ratios for the nine sets of experiments are shown Table (3).

Table (3): Orthogonal array and results

Ex. No.	Methanol (ml)	Catalyst concentration (gm)	Stirrer speed (rpm)	Yield (ml)	S/N ratio (dB)
1	90	0.69	1000	180.00	45.11
2	90	0.92	1100	198.00	45.93
3	90	1.15	1200	201.00	46.06
4	100	0.69	1100	205.50	46.26
5	100	0.92	1200	213.00	46.57
6	100	1.15	1000	206.75	46.31
7	110	0.69	1200	230.50	47.25
8	110	0.92	1000	216.00	46.69
9	110	1.15	1100	238.25	47.54

Experiment no.9 gave the highest yield of SFME and had the largest S/N ratio. The relationship between the yield of sunflower methyl ester and the S/N ratio observed in other experiments, show the similar trend.

A. Results of Statistical Analysis of Experiments:

The experimental results and calculated values were obtained and then the results were analyzed with the aid of commercial software MINITAB 16 ®. This was specially utilized for the design of experiment and statistical analysis of the experiment appliances. The effect of controlled process parameters such as the methanol, catalyst concentration and stirrer speed has been analyzed and the rank of involved factors like yield which supports S/N response is given in Table (4).

Table (4): Response for S/N ratio of SMFE yield

Level	Methanol (ml)	Catalyst concentration (gm)	Stirrer speed (rpm)
1	45.7	46.21	46.03
2	46.38	46.4	46.58
3	47.16	46.64	46.63
Delta	1.46	0.43	0.59
Rank	1	3	2

It is evident from the tables that, among these process parameters, methanol is a dominant factor on the production of yield. The effects of those controlled parameters are graphically represented in Figure (2).

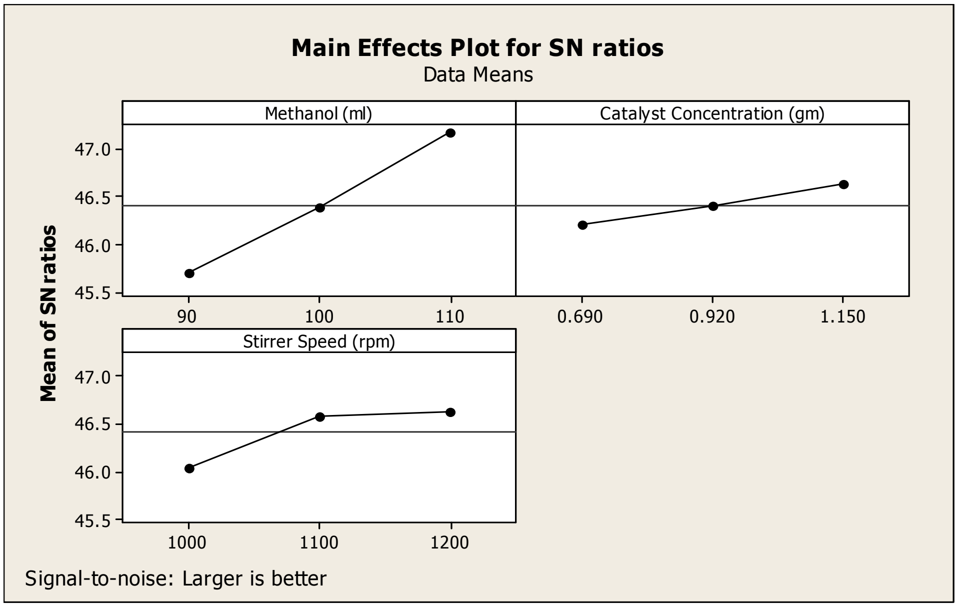

Figure (2): Main Effects Plot for S/N ratios

It clearly indicates that the third level of methanol, third level of catalyst concentration and third level of stirrer speed are the optimum points, but these optimum conditions are not available in L_9 orthogonal array. Hence, the optimum conditions were tested separately using transesterification process and it was found that the maximum yield of 240 ml SFME was produced with the methanol of 110ml, NaOH catalyst as 1.15gm and 1200 rpm of stirrer speed.

B. Analysis of Variance for yield:

The result of the analysis of variance (ANOVA) on the yield production of SFME is shown in Table (6). This analysis is carried out at a level of 5% significance that is up to a confidence level of 95%. The last column of the table indicates the percentage of contribution (Pr) of each factor on the total variation indicating the degree of its influence on the results. Table (6) shows the results of process parameters influence in SFME production by using ANOVA.

Table (6): ANOVA results for yield

Source	DF	Seq SS	Adj SS	Adj MS	F	P	Pr%
Methanol (ml)	2	1873.6	1873.6	936.8	66.09	0.015	76.28
Catalyst concentration (gm)	2	153.6	153.6	76.8	5.42	0.16	5.18
Stirrer speed (rpm)	2	363.5	363.5	181.8	12.82	0.07	13.86
Error	2	28.3					4.68
Total	8	2419.01					100

Hence, the amount of methanol is an important control process parameter to be taken into account in the production of SFME.

C. Multiple Linear Regression Models:

The multiple linear regression equation can be developed by using statistical software MINITAB 16®. This developed model gives the relationship between independent/predictor variable and a response variable by fitting a linear equation to the measured data. Eq. (2) represents the regression equation developed for yield production as given below,

$$\text{Yield (ml)} = -62.9 + 1.76\ \text{Methanol (ml)} + 21.7\ \text{Catalyst concentration (gm)} + 0.0696\ \text{Stirrer speed (rpm)} \quad (2)$$

R-Sq = 92.4%

D. Confirmation Experiment

The last step is the confirmation test. Table (7) indicates the values used for conducting the yield production and Table (8) shows the results of confirmation experiment and its comparison with regression model which helps to identify the optimum parameter values from the experimental analysis. The mathematical model was developed with the help of regression Eq. (2) and the results were analyzed along with the values obtained experimentally. From the analysis, the actual yield is found to be varying from the calculated one using regression equation and the error percentage less than 7.6 %. As these values are closely resemble the actual data with minimum error, it can be stated that design of experiments by Taguchi method was successful for calculating yield from the regression equation.

Table (7): Confirmation experiment for yield

Level	Methanol (ml)	Catalyst concentration (gm)	Stirrer speed (rpm)
1	93	0.7	1050
2	98	0.8	1120
3	107	0.9	1180

Table (8): Confirmation experiment Vs Regression model

Ex. No	Exp. yield in (ml)	Reg. model equation for yield	Error (%)
1	195	189.05	3.05
2	208	204.89	1.49
3	231	227.08	1.69

5. Conclusions:

The Taguchi method, which uses a set of orthogonal arrays for performing the fewest experiments, was employed to design experimental trials involved in production of sunflower methyl ester. In addition, an ANOVA test was performed to systematically analyze the relative importance of each experimental parameter on the production of sunflower methyl ester. From the results of the study, the following conclusions are drawn

- According to the Taguchi method, the influencing parameters are found in the order of methanol 110 ml, catalyst concentration 1.15 gm (0.5% wt) and stirrer speed 1200 rpm which produces the maximum yield of 96%.
- Similarly, ANOVA test results show that methanol has the highest statistical influence in SFME yield production by 76.28% and the contribution of other parameters are 13.86% by stirrer speed and 5.18% by catalyst concentration.
- The yield production is dominated by different parameters in the order of methanol, stirrer speed and catalyst concentration. ANOVA test results show that yield production increases significantly as methanol quantity increases.
- From confirmation tests, the errors connected with yield ranges less than 7.6% which prove that the design of experiments by Taguchi method was successful for calculating yield from the regression equation.

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