PARAMETRIC OPTIMIZATION OF KUSUM OIL BIODIESEL PRODUCTION USING TAGUCHI-PCA METHOD

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Abstract: Biodiesel (mono alkyl esters) is a cleaner-burning diesel fuel produced using natural, renewable sources, for example, vegetable oils. The utilization of biodiesel in an ordinary diesel engine outcomes in a large reduction of unburned hydrocarbons, carbon monoxide, and other matter harmful to nature. biodiesel works well with new technologies and catalysts, which reduces the soluble fraction of diesel particulate but not the solid carbon fraction, particulate traps, and exhaust gas recirculation leading to longer engine life. As the fossil fuels are depleting constantly, a need has arrived to produce biodiesel from various feedstocks.An attempt has been made to produce biodiesel from Kusum (Carthamus tinctorius L.) oil and the process parameters namely the methanol-to-oil molar ratio, catalyst concentration, reaction time and reaction temperature for biodiesel production were optimized. Taguchi robust design process was used by taking L9 orthogonal array and analysed the performance parameters that influence the production process. The results showed that the optimum yield of biodiesel was 93.8% with viscosity 5.60 cSt, with a methanol-to-oil molar ratio of 4:1, catalyst concentration of 1.5 wt%, reaction time of 90 min and reaction temperature of 600C. The catalyst concentration was found to be the most influencing parameter which contributed 51.1% and 50.8% of the total effect on the yield of biodiesel, Y1, and viscosity of biodiesel, Y2, respectively. Regression equation was arrived by using ANOVA technique. The multi parameter output such as Yield and viscosity at 400C were optimized using Principle Component Analysis.

***Index Terms* -** Biodeisel, process parameters, Taguchi PCA method, Kusum oil**.**

I. INTRODUCTION

Kusum (Carthamus tinctorius L.) is a very spread, herbaceous, thorn like yearly plant. It is monetarily developed for vegetable oil separated from the seeds. Plants are 30 to 150 cm (12 to 59 in) tall with globular bloom heads having yellow, orange, or red blossoms. Each branch will for the most part have from one to five blossom heads containing 15 to 20 seeds for each head. Kusum is local to parched conditions having regular rain. It grows a profound taproot which empowers it to flourish in such conditions.

Carthamus tinctorius L. is a plant. The flower and oil from the seeds are utilized as prescription. Kusum seed oil is utilized for forestalling coronary illness, including "hardening of the arteries" (atherosclerosis) and stroke. It is additionally used to treat fever, tumors, hacks, breathing issues, coagulating conditions, torment, coronary illness, chest torment, and horrendous wounds. A few people utilize it for instigating sweating; and as a diuretic, stimulant, antiperspirant, and expectorant to help extricate mucus. Ladies now and again utilize Kusum oil for missing or agonizing menstrual periods; they utilize Kusum blossom to cause a fetus removal. In nourishments, Kusum seed oil is utilized as a cooking oil. In manufacturing, Carthamus tinctorius L. flower is utilized to shading beautifying agents and color textures. Carthamus tinctorius L. seed oil is utilized as a paint solvent.

The seed oil substance of Carthamus tinctorius L. ranges from 30-45 % percent. Its oil is utilized by both food producers and by industry. High linoleic Kusum oil is utilized as a part of human nourishment, however in later a long time advertise request has definitely moved from the conventional high linoleic oils to high oleic oil. High linoleic oil is esteemed as a drying specialist in paints and varnishes on account of its nonyellowing trademark. High oleic Carthamus tinctorius L. oil is bring down in soaks and higher in monounsaturated than olive oil (Berglung et al. 2007).

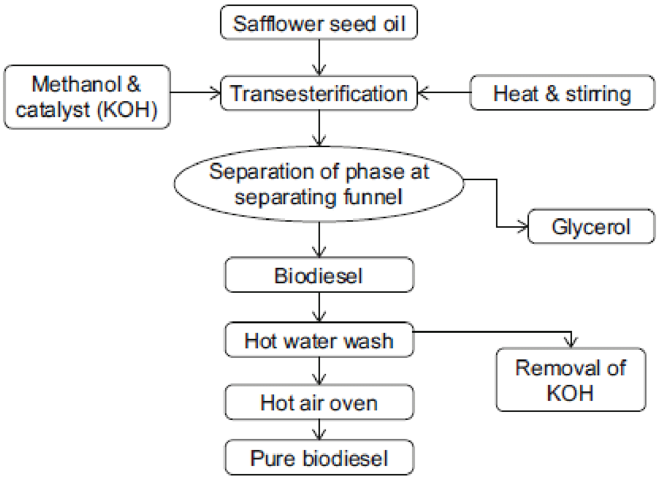
The transesterification procedure is a vital procedure in the creation of biodiesel in light of the fact that it diminishes the viscosity of vegetable oils and conveys it closer to that of conventional diesel oil, in this manner maintaining a strategic distance from the engine related issues and increasing the engine life. Alkali catalyzed transesterification reaction is the most embracing techniques for generation of biodiesel in light of the fact that it is easy to remove the particular esters. This procedure is material for oils that have low free fatty acid level (<4) and acid catalyst transesterification process is pertinent just for high free fatty acid level (>4) oils.

Enzymatic catalyzed transesterification can likewise be utilized for the generation of biodiesel from poor quality feedstock like utilized cooking oil, animal fat, and so forth. This procedure is done within the sight of enzyme, for example, lipase. This procedure has many points of interest over the ordinary transesterification process like age of zero byproduct, no difficulty in separation, and requiring moderate reaction conditions. However, yield, reaction times, and costs are still critical compared to alkaline catalyzed transesterification reaction [1, 2].

Noncatalyzed Biox transesterification process is another strategy accessible for the production of biodiesel. In transesterification reaction biodiesel yield is moderate in view of the low solvency of alcohol in oil. This problem is solved by using a cosolvent, tetrahydrofuran, to stabilize methanol [3, 4]. Noncatalyzed supercritical methanol technique is the latest strategy accessible for producing biodiesel. This technique requires just 4 minutes to finish the reaction yet requires high alcohal oil ratio, high capital cost, and high pressure (80 bar) and temperature (350°C– 400°C) [5– 7].

II. MATERIALS AND METHODS

2.1 Materials



Carthamus tinctorius L. seed oil was purchased from Terra Firma Projects Private Limited, Begumpet, Hyderabad, India. All reagents such as methanol (GR grade, moisture < 0.02%) and analytical-grade catalyst potassium hydroxide (KOH) were obtained from a local chemical store.

The Carthamus tinctorius L. oil was sifted utilizing filter paper and was used for biodiesel production by the transesterification process (Fig. 1).

2.2 Methods

(1)150 g of Kusum oil was taken in a 250-mL glass vessel and preheated up to 105– 110^0C to expel the moisture from the oil and afterward permitted to cool to 45– 50^0C.

(2) Now methyl Alcohal (CH_3OH) for methanol-to-oil molar proportions of 4:1, 6:1 and 8:1 and catalyst potassium hydroxide (KOH) at 0.5 wt% (weight percent), 1.0 and 1.5 w% of the oil were blended together.

Fig:1 Biodiesel preparation stages of Carthamus tinctorius L. oil

(3) This homogeneous blend of methyl liquor and catalyst KOH was blended with 150g Carthamus tinctorius L. seed oil oil.

(4) The conical flask containing the mixture of oil, alcohal and catalyst was heated at a consistent temperature of 50– 60^0C [8-10] and mixed at the same time at the same time inside a water bath shaker at around 700 rev/min for 60 min, 75 min and 90 min, individually.

Fig:2 a) Carthamus tinctorius L. seeds *b) Carthamus tinctorius L. flower* *c) Biodiesel separation from glycerine*

(5) After finishing of the reaction time, the products were poured into a separating funnel and kept 1– 2 h for separation of phases [11]. In the separating funnel , the products separated into two layers. Because of higher specific weight, glycerol settled at the bottom and the upper layer was biodiesel (Fig. 2c). The glycerol was disposed of.

(6) After separation, the biodiesel was washed with hot distilled water keeping in mind the end goal to expel remaining methyl alcohol, catalyst and impurities present.

(7) Finally, the biodiesel was set in a hot air oven furthermore, heated at 100^0C to remove excess water content.

Nine experiments run using the Taguchi method were selected according to orthogonal array L9. The experimental design matrix and effect of factors on the performance parameters are shown in Table 2.

2.3 SIGNAL-TO-NOISE RATIO:

Dr. Taguchi proposed a class of statistics called signal-to-noise ratios (S/N) which can be used to measure the effect of noise factors on the process performance. By maximizing the S/N ratios, the loss functions are minimized. These SIN ratios take into account both the amount of variability and closeness to the average response. In this paper, we will only consider three of them: smaller-is-better, larger-is-better and target-value-is-best.

Smaller-is-better (variance of response):

This S/N ratio assumes that the target for the response is zero and is appropriate when specifications indicate an upper tolerance limit only.

$$S/N_s = -10 \log \left(\frac{1}{n}\sum y_i^2\right)$$

The goal of an experiment for smaller-is-better situations is to minimize y_i^2 and y. That is we aim to maximize -10 log

Larger-is-better (mean of response):

This S/N ratio asswnes that the goal is to maximize the response and is appropriate when specifications indicate a lower tolerance limit only.

$$S/N_s = -10 \log \left(\frac{1}{n}\sum(1/y_i^2\right)$$

2.4 Principal Component Analysis

Principal Component Analysis is a dimension reduction tool that can be used in multi variable analysis problem. Principal Component Analysis aims at reducing a large set of variables to a small set that still contains most of the information contained in the large set. It is a method to identify patterns in a data in such a way as to highlight their similarities and differences. So the data can be compressed without losing any information. It is the most meaningful basis to re-express a noisy and grabbled data set. We often do not know what measurements best reflect the dynamics of our system in question. Sometimes we record more dimensions than we really need PCA alleviates this problem by mapping the original predictors into a set of principal components that is lesser in dimension than the number of the original variables. Such a transformation will usually be accompanied by a loss of information. The goal of PCA is, therefore, to preserve as much information contained in the data as possible. The optimal number of principal components (PCs) needed to achieve this task is not known a priori. The task is to find a set of principal components with Eigen values that have a significantly larger value than the remaining components.

Step 1: Get some data

Let X_i (j) represents the response.

Where i = (1, 2, …, m) where m is the number of experiments performed.

j = (1, 2, …, n) where n is the number of quality characteristics.

Step 2: Normalization of the quality characteristics

Equation (2) is used to normalize surface roughness as surface roughness is to be minimized.

Equation (3) is used to normalize material removal rate as MRR is to be maximized.

For Lower-the-better the equation used is $x^*_{i,k} = \min x_i(k)/ x_i(k)$

For Higher-the-better the equation used is $x^*_{i,k} = x_i(k)/ \max x_i(k)$, where, $x_i(k)$ represent the normalized data of the ith experiment and kth response.

Step 3: Calculate the covariance matrix

The normalized data is utilized to construct a variance covariance matrix R, which is illustrated as below:

$$\begin{bmatrix} R1,1 & \cdots & R1,n \\ \vdots & \ddots & \vdots \\ Rm,1 & \cdots & Rm,n \end{bmatrix}$$

Where Ri,j = Cov $x_i^*(j)$, $x_i^*(k)$ is the covariance of sequences $x_i^*(j)$ and $x_i^*(k)$; $\sigma x_i^*(j)$ and $\sigma x_i^*(k)$ are standard deviation of sequences $x_i^*(j)$ and $x_i^*(k)$.

Step 4: Calculate the Eigen vectors and Eigen value of the covariance matrix

Step 5: Form the feature vector:

Choose the Eigen vectors with the large Eigen values. To form feature vector, arrange the Eigen values from highest to lowest. Ignore the components of lowest significance. Final data set will have fewer dimensions. If we choose the first *p* Eigen vectors and final data will have *p* dimensions.

Step 6: Derive the new data set

III. RESULTS AND DISCUSSION

Table:1 Chosen parameters and their levels

Process parameters		Levels		
		-1	0	1
Methanol-to-oil molar ratio	A	4:1	6:1	8:1
Catalyst concentration, wt%	B	0.5	1.0	1.5
Reaction time, min	C	60	75	90
Reaction temperature	D	50	55	60

Table:2 Test data summary for the responses

Expt no.	Input parameters/levels				Responses	
	A	B	C	D	Yield (Y_1) of biodiesel	Viscosity (Y_2) at 40^0C in cSt
1	-1	-1	-1	-1	76.07	10.31
2	-1	0	0	0	94.21	8.79
3	-1	1	1	1	93.81	5.60

4	0	-1	0	1	86.12	12.01
5	0	0	1	-1	90.34	11.99
6	0	1	-1	0	95.54	7.14
7	1	-1	1	0	84.14	11.39
8	1	0	-1	1	88.66	8.98
9	1	1	0	-1	83.80	10.23

Table 1 shows the experimental data of chosen parameters for the preparation of biodiesel from Kardi oil. Four parameters Methanol-to-oil molar ratio, Catalyst concentration wt %, Reaction time min and Reaction temperature ^{0}C were chosen as input parameters. The Yield (Y1) of biodiesel and Viscosity (Y2) at 40^{0}C in cSt were taken as output parameters or responses in the experiments. The mean response refers to the average value of the performance characteristic for each parameter at different levels. The average value of Yield of biodiesel for each parameter at levels -1, 0 and 1 are calculated. The main effects of the various process parameters when they change from the lower to higher levels can be visualized from the Fig. 1 that shows the response graphs of Yield of biodiesel from Kusum Oil. It is clear from the Fig. 1 that the Yield of biodiesel from Kardi Oil is highest at A0 (6:1), B0 (1.0 % wt) , C1 (90 min), D0 (55^{0}C).

The results indicated that the increase of Methanol to Oil molar ratio tends to increase the Yield of biodiesel up to 6:1 and further it was decreased as the Methanol to Oil molar ratio increased to 8:1. As the Catalyst concentration, wt% was increased from 0.5 to 1.0, the Yield was increased and a slight decrease as it increased to 1.0. As the reaction time in minutes increased from 60 to 75 as well as up to 90, the yield has been increased continuously. Finally, the reaction temperature increased from 50^{0}C to 55^{0}C, the Yield increased more and with further increase in temperature up to 60^{0}C, the Yield was decreased. The catalyst concentration as well as the Reaction temperature influenced more for the yield of biodiesel which was also clear from the Table:3 as well as

Fig: 3.

Table 3: Response Table for Signal to Noise Ratios for the Yield of biodiesel
Signal to Noise ratio: Larger is better

Level	Methanol-to-Oil molar ratio	Catalyst concentration, %wt	Reaction time, min	Reaction temperature, °C
-1	38.85	38.28	38.73	38.40
0	39.14	39.18	38.88	39.20
1	38.64	39.17	39.02	39.03
Delta	0.50	0.91	0.29	0.79
Rank	3	1	4	2

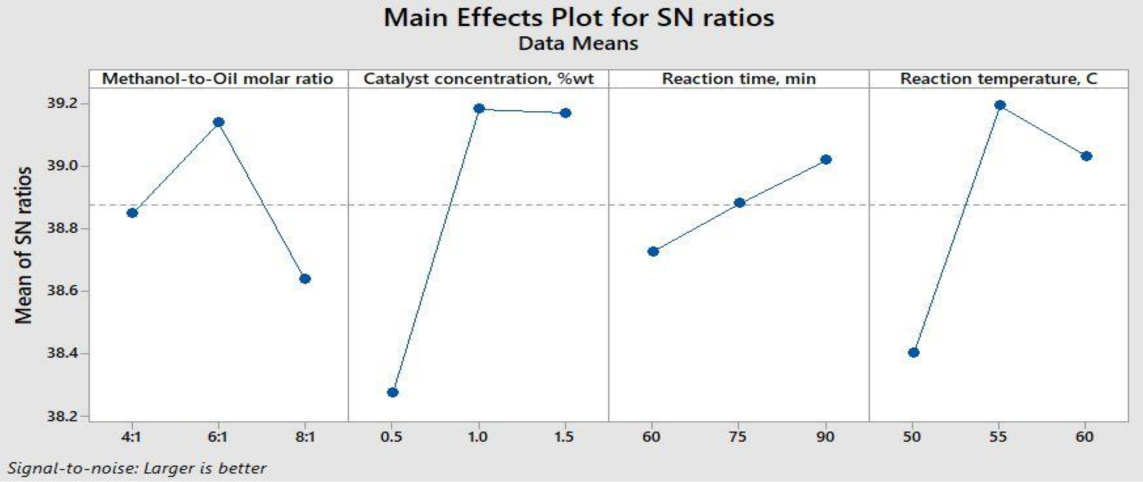


Fig:3 Main effects plot for S/N ratios for the Yield of biodiesel

From the table: 4 and Fig: 2 Methanol to oil ratio at level -1 (4:1), Catalyst concentration at level 1 (1.5 %), reaction time at level -1 (60 min) , reaction temperature at level 1(60^{0}C), the viscosity was minimum. Hence, when the Methanol to oil ratio is less, viscosity was lesser. Catalyst concentration maximum, viscosity was decreased, reaction time increased then viscosity was increased and then decreased. When reaction temperature was increased, the viscosity has been decreased to a great extent. This was evident from S/N ratios table as well as main effects plot. It is clear from the Fig: 4 that the viscosity of biodiesel made from Kusum Oil is lowest at A-1 (4:1), B1 (1.5 % wt) , C -1 (60 min), D1 (60^{0}C) and the order of ranking was Catalyst concentration %wt, Reaction temperature °C, Methanol-to-Oil molar ratio and Reaction time min. .

Table:4 Response Table for Signal to Noise Ratios
Signal to Noise ratio: Smaller is better

Level	Methanol-to-Oil molar ratio	Catalyst concentration, %wt	Reaction time, min	Reaction temperature, °C
-1	-18.04	-21.00	-18.80	-20.68
0	-20.08	-19.84	-20.22	-19.03
1	-20.13	-17.41	-19.22	-18.54
Delta	2.09	3.58	1.42	2.14

Rank	3	1	4	2

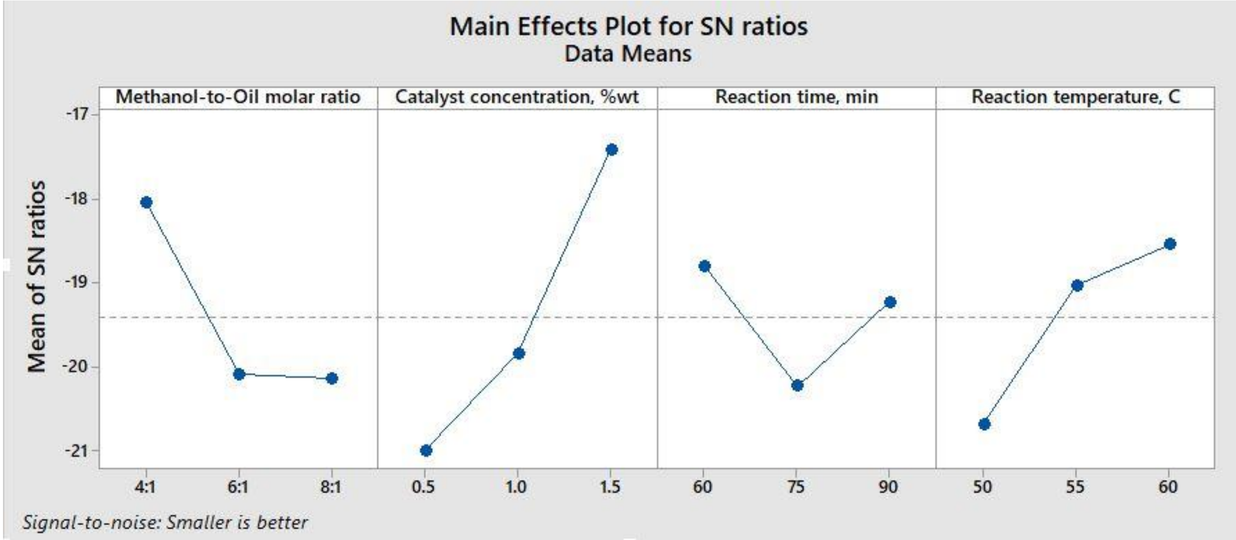


Fig:4 Main effects plot for S/N ratios for the Viscosity (Y_2) at 40^0C in cSt.

Table:5 Signal to Noise ratio for Yield (Y1) and Viscosity (Y2) at 40^0C in cSt of biodiesel

Yield (Y_1) of biodiesel	S/N ratio for Yield (Y1)	Viscosity (Y_2) at 40^0C in cSt	S/N ratio for Viscosity (Y2)
76.07	37.6243	10.31	-20.2652
94.21	39.4819	8.79	-18.8798
93.81	39.4450	5.60	-14.9638
86.12	38.7021	12.01	-21.5909
90.34	39.1176	11.99	-21.5764
95.54	39.6037	7.14	-17.0740
84.14	38.5001	11.39	-21.1305
88.66	38.9546	8.98	-19.0655
83.80	38.4649	10.23	-20.1975

Table: 5 shows the S/N ratio for yield (Y1) by considering larger is better and Viscosity (Y_2) at 40^0C in cSt by considering smaller is better.

Table 6: Normalized Data for Yield (Y1) and Viscosity (Y2) at 40^0C in cSt of biodiesel

Reference sequence Yield (Y_1) of biodiesel	Normalized data (Y1)	Reference sequence Viscosity (Y_2) at 40^0C in cSt	Normalized data (Y2)
76.07	0.79621	10.31	0.54316
94.21	0.98607	8.79	0.63708
93.81	0.98189	5.60	1.00000
86.12	0.90140	12.01	0.46627
90.34	0.94557	11.99	0.46705
95.54	1.00000	7.14	0.78431
84.14	0.88067	11.39	0.49165
88.66	0.92798	8.98	0.62360
83.80	0.87711	10.23	0.54740
76.07	0.79621	10.31	0.54316

For Lower-the-better the equation used is $x^*_{i,k} = \min x_i(k)/ x_i(k)$

For Higher-the-better the equation used is $x^*_{i,k} = x_i(k)/ \max x_i(k)$, where, $x_i(k)$ represent the normalized data of the ith experiment and kth response.

The Yield of biodiesel (Y1) was normalized using Higher-the-best equation and Viscosity (Y_2) at 40^0C in cSt was normalized using Lower-the-better equation.

Table: 7 Eigen analysis of the Correlation Matrix

Eigenvalue	**1.5827**	**0.4173**
Proportion	0.791	0.209
Cumulative	0.791	1.000

Table 8: Eigen vectors

Variable	**PC1**	**PC2**
Yield (Y1) of biodiesel, %	-0.707	-0.707
Viscosity(Y2) at 40 C in cSt	0.707	-0.707

Table 7 and 8 shows Eigen analysis of the Correlation Matrix and Eigen vectors.

Table: 9 Eigen analysis of the Covariance Matrix

Eigen value	**40.961**	**3.057**
Proportion	0.931	0.069
Cumulative	0.931	1.000

Table 10: Eigen vectors

Variable	**PC1**	**PC2**
Yield (Y1) of biodiesel, %	-0.976	-0.217
Viscosity(Y2) at 40 C in cSt	0.217	-0.976

Table 9 and 10 shows Eigen analysis of the Covariance Matrix and Eigen vectors.

To determine which factor has affected the product significantly and how much they contributed, statistical analysis of variance (ANOVA) of the response data can be used. The percentage of contribution of the factors was evaluated with the following equations (Kumar et al. 2015).

$$\text{Contribution factor (\%)} = (S_f/S_T)\times 100$$

where S_f is the sum of squares of fth factor and S_T is the total sum of squares of all parameters.

3.1 Analysis of variance (ANOVA)

The calculated sum of square (S_f) and percentage of contribution factors are shown in Table 11. From the contributions (Table 11), it can be concluded that the factor X_2 (concentration of catalyst) was the most influencing parameter for both performance parameters. The factor X_2 (concentration of catalyst) contributed 51.1% of the total effect of contribution, and among the rests, factors X_4 (reaction temperature), X_1 (methanol-to-oil molar ratio), X_3 (time of reaction) contributed 32.8%, 12.6% and 3.4%, respectively, for the performance parameter Y1 (biodiesel yield). On the other hand, factors X_2, X_1, X_4 and X_3 were provided 50.8%, 22.0%, 18.0% and 9.1% contribution, respectively, for the performance parameter Y_2 (viscosity).

Table 11: Percentage of contribution of process parameters

Factor	Sum of square, S_f	Contribution of factor, %
For Y_1		
X_1	39.53	12.6
X_2	160.21	51.1
X_3	10.72	3.42
X_4	102.96	32.8
For Y_2		
X_1	8.51	22.0
X_2	19.67	50.8
X_3	3.54	9.1
X_4	6.99	18.0

Regression analysis has been carried out by the obtained data of biodiesel yield (Y_1) and is specified as the equation given below. Regression Equation:

$$Y_1 = 88.1 - 1.25X_1 + 4.47X_2 + 1.34\,X_3 + 3.06\,X_4$$
$$R^2 = 0.62$$

IV.CONCLUSION

The purpose of this experimental study was to optimize the process parameters which exert significant effect on the transesterification process. Three process parameters, the methanol-to-oil molar ratio, catalyst concentration and reaction time, have been taken into consideration for the optimization of biodiesel yield and its viscosity using the Taguchi approach.

(1) It was found that the optimum conditions for enhancing the biodiesel production by transesterification were the methanol-to-oil molar ratio of 6:1, catalyst concentration of 1 wt%, reaction time of 90 min and reaction temperature of 55 ^{0}C.

(2) As per the objective of performance parameter Y2 (viscosity), the optimum conditions found from the graph of S/N ratio were methanol-to-oil molar ratio of 4:1, catalyst concentration of 1.5 wt%, reaction time of 60 min and reaction temperature of 60^{0}C.

(3) The concentration of catalyst was found to be the most influencing factor for both performance parameters Y1 and Y2. The percentage of contribution of the concentration of catalyst for Y1 and Y2 was 51.1% and 50.8% of the total effect of contribution, respectively.

(4) Kinematic viscosity is one of the most important properties of biodiesel. So, optimization of the transesterification process was based on the both performance parameters, i.e., biodiesel yield and viscosity. As per the design of experiments, the experimentally obtained maximum biodiesel yield was 95.5% with viscosity 7.14 cSt, whereas the minimum viscosity of biodiesel produced was 5.60 cSt for which the yield of the biodiesel was 93.8%. As the viscosity of the former sample was beyond the standard ASTM limit, the process parameters of the latter, which were methanol-to-oil molar ratio of 4:1, catalyst concentration of 1.5 wt%, reaction time of 90 min and reaction temperature of 60^{0}C, were considered as the optimized conditions for this transesterification reaction.

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