Optimization and Mathematical Modeling of Biodiesel Production using Homogenous Catalyst from Waste Cooking Oil

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Abstract: ***In the present investigation, the transesterification of waste cooking oil (WCO) to biodiesel over homogenous catalyst KOH have been carried out. To optimize the transesterification process variables both response surface method (RSM) and artificial neural network (ANN) mathematical models were applied to study the impact of process variables temperature, catalyst loading, methanol to oil ratio and the reaction time on biodiesel yield. The experiments were planned with a central composite design matrix using 2^4 factorial designs. A performance validation assessment was conducted between RSM and ANN. ANN models showed a high precision prediction competence in terms of coefficient of determination (R^2 = 0.9995), Root Mean Square Error (RMSE = 0.5702), Standard Predicted Deviation (SEP = 0.0133), Absolute Average Deviation (AAD = 0.0115) compared to RSM model. The concentration of catalyst load was identified as the most significant factor for the base catalyzed transesterification. Under optimum conditions, the maximum biodiesel yield of 88.3% was determined by the artificial neural network model at 60 ℃, 1.05 g catalyst load, 7:1 methanol to oil ratio and 90 min transesterification reaction time. The biodiesel was analyzed by GCMS and it showed the presence of hexadecanoic acid, 9-octadecenoic acid, 9, 12, 15-octadecatrienoic acid, eicosenoic acid, methyl 18-methyl-nonadecanoate, docosanoic acid, and tetracosanoic acid as key fatty acid methyl esters.***

Keywords: ***Transesterification; Catalyst; Optimization; Mathematical model; Biodiesel.***

I. INTRODUCTION

The rapid expansion of the world population and the change in their lifestyle would ultimately lead to needing high energy demand. This increasing energy demand leads to the use of more non renewable energy resources like fossil based fuels. The continuous excess use of fossil fuels leads to fuel depletion, price increment and the serious environmental impacts as global warming, ozone depletion, deforestation, acidification and photochemical smog. To reduce this gap different research has been done to develop the exploration of renewable energy resources. Renewable energy sources have strongly developed for two main reasons, first to reduce the greenhouse gases emissions and pollutants and second for the shortage of fossil fuel reserves [1].

Biodiesel can be described as a liquid fuel consisting of mono alkyl esters and a combination of long chain methyl / ethyl fatty acid esters which can be derived from vegetable oils or animal fats [2]. It has significant environmental advantages, such as biodegradability fuel with a more favorable combustion emissions profile and reliability [3]. Transesterification reaction is used to produce biodiesel where oil or fat reacts with alcohol in the presence of a NaOH or KOH or catalyst of any kind to form alkyl ester and glycerol [4]. Using waste cooking oil as an alternative biodiesel feedstock reduces the biodiesel production cost around 70-95 % of the total cost of production. Currently around 15 million tons of waste cooking oil (WCO) produced worldwide, so 1 liter of WCO contaminates 1000 liters of water according to studies conducted by the world health organization. In addition, there is an increase in energy consumption estimated at 25% of wastewater treatment plants and a decrease in drainage performance [2]. Process optimization is an important and notable issue and, requires to enhance the biodiesel production yield and to minimize the production cost. The modeling of biodiesel production process using traditional methods, to predict the influence of process variables on biodiesel yield has shifted from complex analytical equations to powerful and efficient modern technologies. Response Surface Methodology (RSM) is one of major statistical tool used in experimental design, modeling and optimization process variables. It provides a relationship between one/more responses with independent variables. It also determines the impact of independent variables on the whole process, including individual impacts and interaction effects among factors.

The Artificial Neural Network model (ANN) is the most extensively accepted artificial learning tools to classify and predict the response. It was commonly recognized as an alternative method to represent the relationship between input and output of the process variables [5]. ANN has been proven to be far more effective in anticipating than other standard methods such as regression analysis. In the current study, biodiesel production was investigated by employing a homogeneous catalyst using waste cooking oil from household origin. The primary objective of this experimental analysis was to create a mathematical model with RSM and ANN to explore the interactions between the process variables (temperature, methanol to oil ratio, catalyst load and reaction time) and biodiesel yield to attain

Revised Manuscript Received on October 20, 2019.
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optimal transesterification reaction conditions for a greater yield of biodiesel. In addition, the predictive capacities of both models were statistically evaluated and compared with multiple statistical parameters.

II. MATERIALS AND METHODS

A. Sample Collection

Waste Cooking Oil (WCO) was collected from the boy's hotel, Koneru Lakshmaiah Education Foundation hostel, Guntur, Andhra Pradesh, India as a substrate for biodiesel production. The oil was found to contain 2.05 mg KOH g^{-1} Oil of an acid value and stored at room temperature before the beginning of the transesterification process.

B. Oil Pretreatment

The waste cooking oil was filtered more than three times using filter paper (Whatman No.1) to remove any insoluble impurities until it becomes clear which would interfere during the reaction. Then the oil was well preheated (105ºC) to eliminate the moisture content. The adsorption treatment was used to get rid of any colored impurities present in the sample by using charcoal (wt. % to oil) as an adsorbent [6].

C. Experimental Design

The central composite design (CCD) based response surface method model (RSM) was used to design the experimental matrix and optimize the process conditions for transesterification of biodiesel from WCO. A 2^4 factorial experimental design matrix was applied to investigate the optimal process conditions for transesterification reaction. The four major independent operating variables including temperature (A), catalyst load (B), methanol to oil ratio (C) and the reaction time (D) were studied at both high and low levels. Table 1 shows the selected independent variables with their ranges. The predictable response parameter is biodiesel yield and it was interrelated to the selected independent variables using a polynomial quadratic mathematical model. Thirty experimental runs were carried out in a randomized order, based on the experimental design matrix, and table 2 shows the design matrix for the 2^4 factorial designs. In order to evaluate the variance (ANOVA) of the experimental model was executed with the help of design expert software version 11 (State Ease Inc., USA).

Table 1. The selected independent process variables with their ranges

Operating Variables	Symbol Coded	Levels	
		Minimum	Maximum
Temperature (ºC)	A	40	80
Catalyst load (g)	B	0.58	1.72
Methanol to oil ratio	C	5:1	9:1
Reaction time (min)	D	60	120

D. Artificial Neural Networks Modeling

The MATLAB tool (R2015a), was applied to predict the biodiesel yield. To train the network, the design of experiments data and the respective experimental response values were taken (Table 3). Three layer feed forward neural network was used to train the network. In this case, the optimum ANN model architecture (4:10:1) is shown in Fig. 1. In this study one input layer consists of four input variables, hidden layers and one output layer were considered for training the neural network. For network training, the Marquardt Levenberg (ML) back propagation algorithm was implemented. The algorithm data is separated into three components, such as training (60%), testing (20%) and validation (20%) in the ANN the data set [7]. The predictive efficiency of both RSM and ANN models are statistically evaluated by various parameters including, correlation coefficient (R), determination coefficient (R^2), adjusted R^2, standard error of prediction (SEP), root mean square error (RMSE) and absolute average deviation (AAD).

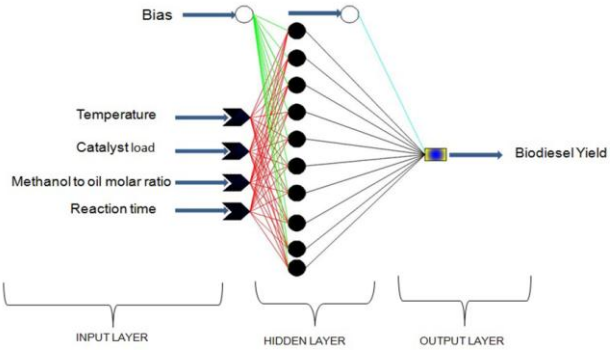

Fig. 1 Optimized Artificial Neural Network (ANN) topology

E. Validation of Models

The prediction of accuracy of an optimization tool can be examined for any process by comparing the model's predicted response to experimental response values. The comparison between RSM and ANN's predictive ability was made using the statistical formula [8]. In addition, the modeling capabilities of both the models for predicting optimal process conditions for transesterification conditions were evaluated by plotting a graph against predicted values contrast corresponding experimental value.

F. Transesterification

The catalyst KOH was added in the required quantity of methanol as mentioned in the experimental design matrix to form a potassium methoxide solution. In a typical transesterification reaction, 100 g of oil was added in all reaction mixtures to a round bottom flask and then preheated potassium methoxide solution was added as per the design matrix. The mixture was refluxed at specific temperature conditions under continuous magnetic stirring (600 rpm). The product mixtures were separated into two separate layers after settled down 24 hrs. The obtained biodiesel phase was cleansed of various impurities such as residual unconverted methanol, soap, catalyst and traces of glycerol. The unconverted methanol was removed by evaporation and other residues removed by washing with three times of warm distilled water. Later it was dried in an oven at 110 °C to remove residual moisture content. The biodiesel yield

conversion was computed by standard formula. The biodiesel sample was examined by GCMS (Model: AccuTOF GCV) for its compositions.

III. RESULT AND DISCUSSION

A. RSM analysis of Transesterification

RSM ANOVA analysis

Homogeneous catalyst based transesterification was performed using the pretreated WCO. Thirty experimental runs were performed to optimize these variables and the obtained biodiesel yield were recorded (Table 2). The predicted model equation referring to the response parameters in terms of coded factors suggested by CCD is shown below equation.

Biodiesel Yield = 87.10 -1.03 A - 5.92 B + 0.2021 C - 1.39 D - 0.0194 AB + 0.5881 AC - 2.29 AD + 1.16 BC - 0.0781 BD - 1.07 CD - 6.36 A^2 - 7.93 B^2 -8.04 C^2 - 6.70 D^2

Y is the output variable (biodiesel yield %), A (temperature), B (catalyst load), C (methanol to oil ratio) and D (reaction time) are the input variables. The parity plot compares the biodiesel yield observed with the values anticipated (Fig. 2). The experimental model F values presented in Table 3 and it implies the model is significant. The F and P value represents the significance and accuracy for the regression model. The lack of fit P value was 0.2742, indicating that it was not significant. Model fitness was evaluated using the equation of regression and the coefficient of determination (R^2). The predicted R^2 and adjusted R^2 the values were 0.9590 and 0.9845 respectively.

There is a rational agreement with the predicted and adjusted R^2 value. The high value of the two coefficients justifies an exceptional correlation among the independent variables and supports the model's high accuracy [10]. In addition, the reduced value of the coefficient of variation (CV=2.61%) shows a higher accuracy and consistency of the experimental model [11]. The optimal transesterification process conditions for predicted by the model were the maximum biodiesel yield of 88.3% at 60 ° C, 1.05 g catalyst load, 90 min reaction time and 7:1 methanol to oil ratio.

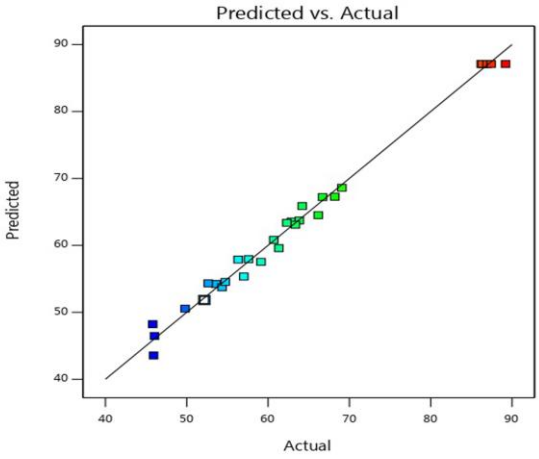

Fig. 2 Experimental Vs predicted biodiesel yield

Table 2: Experimental CCD matrix with experimental values and predicted responses

Run	Biodiesel Yield (%)		
	Experimental	Predicted	
		RSM	ANN
1	62.3	63.38	62.12
2	86.32	87.10	87.13
3	63.85	63.73	63.60
4	66.7	67.22	66.462
5	45.82	48.24	45.82
6	64.2	65.88	64.01
7	61.32	59.59	61.16
8	52.16	51.84	52.08
9	52.64	54.33	52.61
10	57.6	57.94	57.49
11	59.13	57.55	59.09
12	63.36	63.09	63.07
13	53.6	54.24	53.54
14	66.17	64.51	67.51
15	45.92	43.55	45.92
16	86.21	87.10	87.13
17	56.32	57.88	56.31
18	89.2	87.10	87.13
19	86.93	87.10	87.13
20	54.74	54.55	54.57
21	54.34	53.75	53.37
22	57.02	55.36	57.00
23	49.8	50.55	49.73
24	86.53	87.10	87.13
25	68.21	67.28	68.73
26	60.7	60.82	60.51
27	87.42	87.10	87.13
28	62.89	63.55	62.67
29	69.08	68.61	68.86
30	46.04	46.47	46.10

Interaction effects

The response 3D surface plots and the two dimensional contour plots are usually the graphical representations of the empirical model (Fig.3). Fig.3a shows the relationship between temperature and catalyst load at 90 min and 7:1 methanol ratio on biodiesel production. There was a drastic increase in biodiesel yield in fig. 3a by raising the temperature of the reaction and catalyst load to a significant value. The elevated temperature would inactivate the catalyst activity and it reverse the reactions because in general both transesterification and esterification reactions are reversible reactions in nature. Srilatha et al. [12] was reported the similar finding in the biodiesel production using niobia catalyst. However, the increase in KOH load

significantly affected the transesterification process. These increases in biodiesel yield are anticipated due to the quantity of catalytic site is rising with increasing catalyst load [13].

Table 3. ANOVA analysis for the predicted quadratic model of biodiesel yield

Source	Mean Square	F-value	p-value	Significance
Model	368.21	132.45	< 0.0001	Significant
A	25.69	9.24	0.0083	
B	840.05	302.18	< 0.0001	
C	0.9801	0.3526	0.5615	
D	46.18	16.61	0.0010	
AB	0.0060	0.0022	0.9635	
AC	5.53	1.99	0.1787	
AD	83.86	30.17	< 0.0001	
BC	21.60	7.77	0.0138	
BD	0.0977	0.0351	0.8538	
CD	18.25	6.57	0.0217	
A^2	1109.66	399.16	< 0.0001	
B^2	1724.53	620.34	< 0.0001	
C^2	1771.60	637.27	< 0.0001	
D^2	1229.62	442.32	< 0.0001	
Residual	2.78			
Lack of Fit	3.54	2.83	0.1314	not significant
Pure Error	1.25			
R^2 = 0.9920; Predicted R^2 = 0.9590			Adjusted R^2 = 0.9845; CV = 2.61 %	

The interface effects of temperature and the proportion of methanol oil ratio on biodiesel yield is shown in fig. 3b at 1.25 g catalyst load and 90 min reaction time. The biodiesel yield was increased to the maximum level after that decreased at lengthy reaction time following overloading of methanol concentration. The contour plot's elliptical nature revealed a significant impact on biodiesel yield between temperature and methanol oil ratio. The biodiesel formation can be increased by adding methanol in excess quantity, which interfere the equilibrium of the reaction and it move the balance towards the production side. However, addition of excess methanol may increase glycerol's solubility; thus interpreting glycerol's separation from the reaction mixture [14]. Fig. 3c illustrates the interaction between reaction time and temperature. The contour plot's elliptical nature showed that the relationship between reaction time and temperature against biodiesel yield was significant. The production of biodiesel was increased up to a certain critical level with increasing temperature and reaction time. With increasing reaction temperature, mass transfer restrictions occurred between the liquid and solid phase in the heterogeneous catalytic system [15,16]. The higher quantity of methanol in the reaction mixture facilitated the rate of diffusion of methanol towards the catalyst pore. The catalyst concentration of 1.25 g KOH is beneficial for diffusion which leads to increase in biodiesel yield due to the bigger pore size of the catalyst [17]. Further, the higher reaction temperature leads to a limitation in mass transfer, resulting in less biodiesel yield [10]. The circular and elliptical nature of biodiesel response curve was observed at 1.25 g catalyst load and a methanol to oil ratio of 7:1 (Fig.3d). The contour plot showed considerable interaction effects on biodiesel yield for catalyst load and methanol oil ratio. Biodiesel conversion was low at greater catalyst concentration and reduced methanol oil proportion as well as at low temperature.

Fig. 3e shows the response and contour plot for catalyst load and reaction time interface at 90°C and 7:1 methanol to oil ratio. The amount of catalyst load seemed to have a mild impact on the biodiesel yield. Meanwhile, the increasing reaction time increases biodiesel production up to the critical limit and beyond that, it considerably affects the transesterification process [11,18]. In addition, over reaction time may lead to a small decline in the yield of biodiesel due to the reversible transesterification reaction [19]. Fig. 3f depicts the consequence on biodiesel formation against methanol to oil ratio and reaction time interaction in the presence of 60 ° C and 1.25 KOH. The interaction between these two factors suggested a considerable impact on the biodiesel production as shown in the contour plot's elliptical nature.

B. ANN Modeling

The ANN model depends on the decisive optimal neuron numbers. Fig. 4 shows the spread plot of experimental versus the computed ANN data. The regression, R^2 values for training (0.9994), validation (0.9999), test (0.99979) and all prediction set (0.99908) were showed the accurate prediction of the model. Nearly each and every data points have been scattered around the 45° line indicating remarkable compatibility between the experimental and predicted output data values by ANN. In all cases, the R^2 value between the experimental and ANN response implies that the predicted ANN model was more precise in predicting the yield of biodiesel [8].

C. The predictive capability of models

The predictive competence of the developed RSM and ANN models in prediction of the biodiesel yield were evaluated in terms of various statistical parameters including R, R^2, adjusted R^2, SEP, RMSE and AAD. The obtained results of the above parameter are presented in Table 4 for both RSM and ANN models. If the R^2 value is near to 1 then the experimental and expected response values are well correlated (ie., 0.9992 and 0.9998 for RSM and ANN, respectively) [20]. The very high values of R^2, demonstrate the authentic suitability of RSM and ANN models. The RMSE value (0.5702) of ANN model is much smaller than

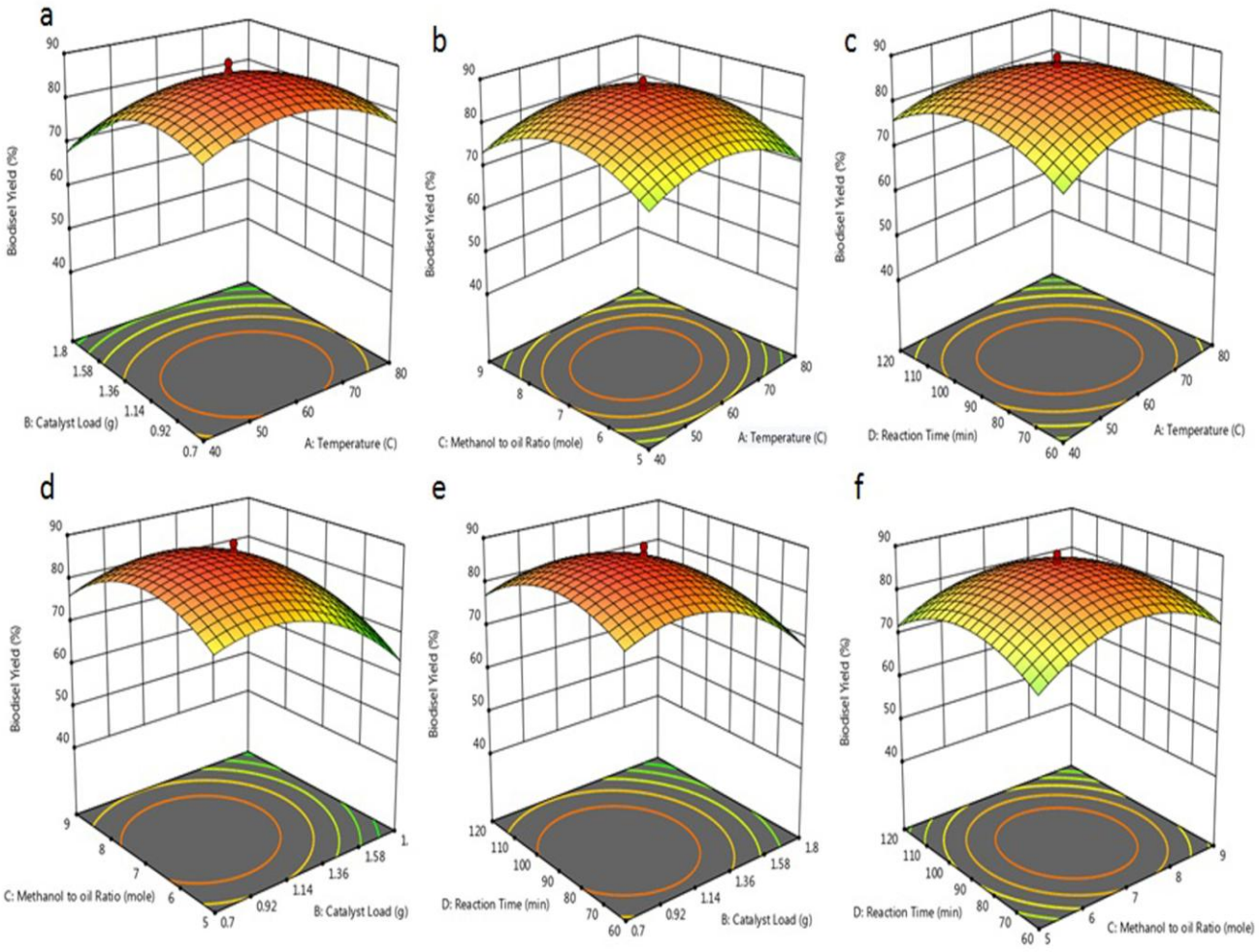

Fig. 3 RSM surface plot of the process variables a) temperature and catalyst load b) temperature and methanol to oil ratio c) temperature and reaction time d) catalyst load and methanol to oil ratio e) catalyst load and reaction time f) methanol to oil ratio and reaction time on biodiesel yield.

the RSM model (1.1786). These findings confirm that the model ANN is better than the RSM model. SEP and AAD check the significance and accuracy of the models [21]. The low values of the mentioned statistical parameters indicates the better the performance of the predicted model. The ANN model showed that higher predictive accuracy and generalization capability than the RSM model [22].

Table 4: Statistical parameter assessment of RSM and ANN models

Parameters	RSM	ANN
Determination Coefficient (R^2)	0.9976	0.9995
Root Mean Square Error (RMSE)	1.1786	0.5702
Standard Predicted Deviation (SEP %)	1.8449	1.3323
Absolute Average Deviation (ADD %)	4.6037	1.1516

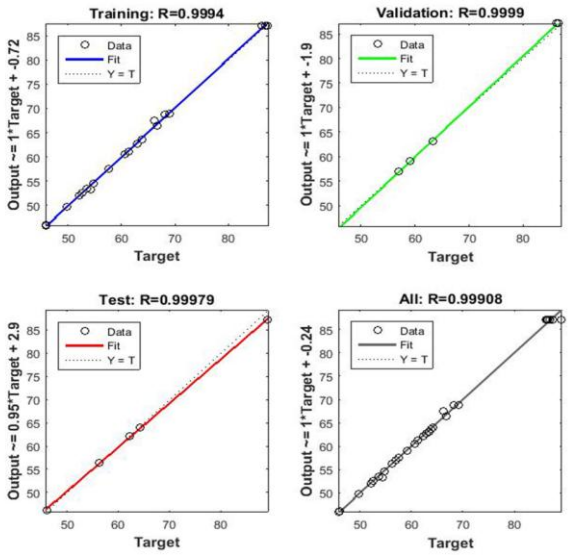

Fig. 4 Artificial Neural Network plot of experiment versus predicted value on biodiesel yield

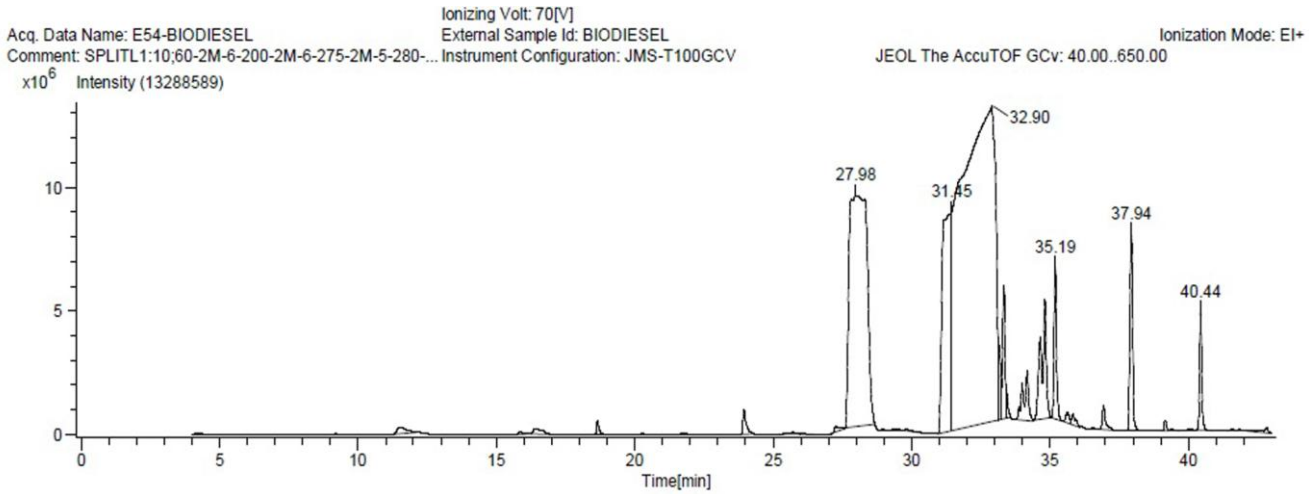

Fig. 5 GC/MS chromatogram of the biodiesel

D. GC-MS Analysis

The biodiesel generated by the experiment is a transparent yellow color liquid. The GC-MS analysis was performed to know the fatty acid methyl esters present in the biodiesel product. Fig 5 demonstrates the GC-MS chromatogram of obtained biodiesel product. It clearly showed that the biodiesel contained mainly, hexadecanoic acid, 9 - octadecenoic acid, 9,12,15 - octadecatrienoic acid, eicosenoic acid, methyl 18- methylnonadecanoate, docosanoic acid, and tetracosanoic acid methyl esters.

IV. CONCLUSION

The optimization and modeling of the parametric study of biodiesel production through transesterification from waste cooking oil was effectively. ANN model were developed to predict the transesterification process conditions based on CCD and feed forward multilayered perceptions. High values of R, R^2, predicted R^2 (> 0.99) clearly show the high precision of both the models. Statistical parameters value of R^2 (0.9995), RMSE (0.5702), SEP (0.0133), AAD (0.0115) for the validation information sets, the ANN model was shown to be more effective in both data fitting and prediction capacities than the RSM model. Under optimal transesterification conditions the production of biodiesel by KOH was achieved above 88.3%. The composition of fatty acid methyl esters present in the biodiesel was analyzed by GCMS. The current research shows that the waste cooking oil is quite appropriate as a low cost feedstock for the manufacturing of biodiesel.

ACKNOWLEDGMENT

We are thankful for their instrumental and technical assistance to the Sophisticated Analytical Instrument Facility (SAIF), IIT Bombay for GC/MS analysis.

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