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Data Article

Dataset on optimized biodiesel production from seeds of *Vitis vinifera* using ANN, RSM and ANFISV. Hariram^{*}, A. Bose, S. Seralathan

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ABSTRACT

This dataset disclose the investigational data on the extraction of bio-oil from seeds of *Vitis vinifera* through combination of mechanical pressing and soxhlet solvent extractor. Biodiesel is produced through single stage base catalysed transesterification process due to lower free fatty acid content in the *Vitis vinifera* bio-oil. Independent variable process parameters like molar ratio, reaction time and catalyst concentration are optimized using Artificial Neural Network, Response Surface Methodology and Adaptive Neuro-Fuzzy Interference System to predict the maximum biodiesel yield and the results are compared with the experimental data. Response Surface Methodology predicted a maximum *Vitis vinifera* biodiesel yield of 97.62% at methanol to oil molar ratio 0.2758 v/v, catalyst concentration 1.045 gm of NaOH and reaction duration of 1.11 hrs which is also confirmed with experimental results.

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1. Data

Based on the previous literatures [1,2], it was noticed that, production of biodiesel from seeds of *Vitis vinifera* was not attempted by many researchers. Further, optimization of the biodiesel production process using soft computing tools was also reported by few studies. This data article explores the comparative advantage of using Adaptive Neuro-Fuzzy Interference System (ANFIS), Artificial Neural

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Specifications table

Subject area	Bio-energy
More specific subject area	Biodiesel
Type of data	Figures, Tables and Surface plots
How data was acquired	Experimental analysis of biodiesel from <i>Vitis vinifera</i>
Data format	Tabulated, Raw as well as Processed data
Experimental factor	Optimized biodiesel production from <i>Vitis vinifera</i> bio-oil by varying molar ratio, catalyst concentration and reaction time by using soft computing tools like ANN, ANFIS and RSM.
Experimental features	<i>Vitis vinifera</i> bio-oil is extracted through soxhlet extraction process and subjected to transesterification process at varying degree of input variable parameters.
Data sources	Antoine Lavoisier (FLL) Lab, HITS
Data accessibility	Data is available within this article

Value of the data

- This dataset portrays the ability of extracting *Vitis vinifera* bio-oil extraction and its biodiesel production capability which can be potential alternate to mineral diesel.
- This dataset can be used as a benchmark to compare the improvements in physio-chemical properties of *Vitis vinifera* biodiesel when blended with oxygenated additives and nano particle.
- It also benefits the researchers in further enhancing the *Vitis vinifera* biodiesel yield by using analytical and soft computing tools.
- The process operating parameters which includes concentration of catalyst concentration, molar ratio and reaction time were optimized using Adaptive Neuro-Fuzzy Interference System (ANFIS), Artificial Neural Network (ANN) and Response Surface Methodology (RSM) tools to considerably bring down the overall time and cost for *Vitis vinifera* biodiesel production.

Network (ANN) and Response Surface Methodology (RSM) on estimating and optimizing the biodiesel production yield from *Vitis vinifera* bio-oil through single stage base catalysed transesterification process.

This dataset divulges the procedural approach for extracting bio-oil from crushed *Vitis vinifera* seeds through soxhlet process in presence of *n*-hexane solvent and converting it into biodiesel through single stage base catalysed transesterification process using methanol and sodium hydroxide [3,11]. Five different types of dataset are presented in this data article. Firstly, the crushed seeds of *Vitis vinifera* is processed in soxhlet apparatus, a solvent extraction methodology to extract bio-oil in the presence of *n*-hexane as shown in Fig. 1. Secondly, the distillate (bio-oil) is subjected to single stage base catalysed transesterification process with sodium hydroxide and methanol for the transformation of mono-alkyl triglycerides of *Vitis vinifera* oil into fatty acid methyl esters as shown in Fig. 1. Thirdly, the optimization of input variables namely, catalyst concentration, molar ratio and reaction time is carried out using model developed through ANN, ANFIS and RSM approach as shown in Fig. 3. Fourthly, the efficiency of the developed model is validated and compared in Table 3. Finally, the derived biodiesel is categorized and characterized using Gas Chromatography and Mass Spectrometry analysis to classify and understand the various FAMES present in *Vitis vinifera* biodiesel as shown in Table 4 and Fig. 5.

2. Experimental design, materials and methods

2.1. Materials

Vitis vinifera is one of the significant grape species cultivated in Indian sub-continent. About 79.6 thousand hectares of land area is cultivated out of which around 25.6 lakh tonnes of grapes in India alone. On an average, *Vitis vinifera* contains seeds at 4.3% by weight (approximately 1.02 lakh tonnes) making it a potential bio-resource. *Vitis vinifera* seeds are supplied by Noyer Overseas India Pvt Ltd,

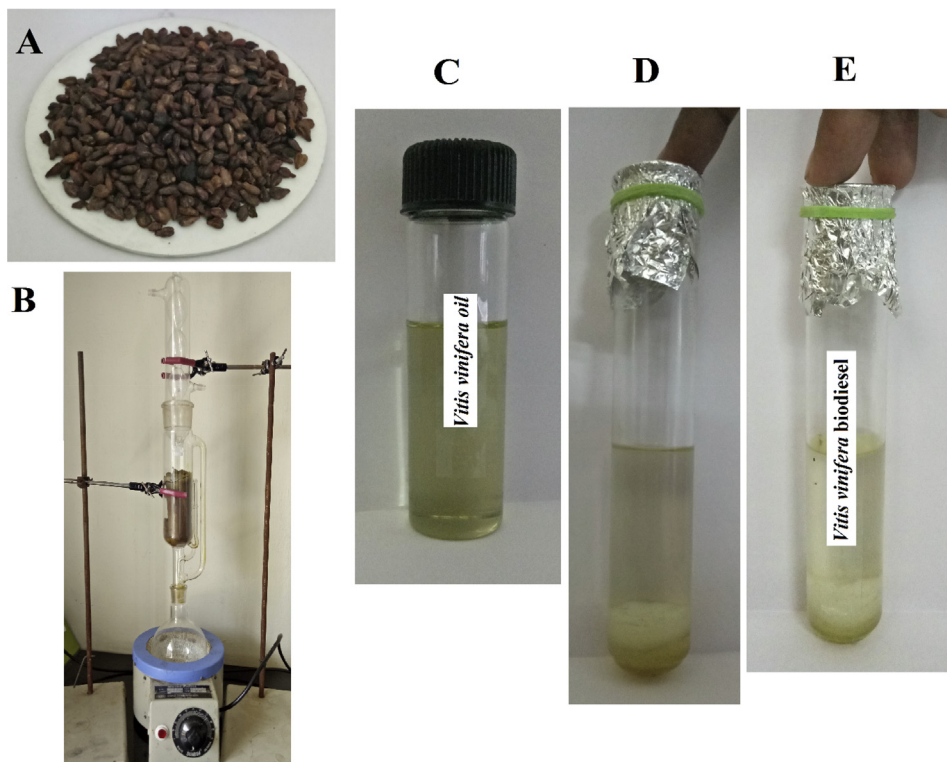


Fig. 1. *Vitis vinifera* seeds (A), Soxhlet bio-oil extraction (B), Extracted bio-oil (C), Transesterification process (D) and *Vitis vinifera* biodiesel (E).

Pune, India. National Petrochem, Chennai, India supplied laboratory grade methanol and sodium hydroxide is purchased from Prime Laboratories, Hyderabad, India. Pre-processing of *Vitis vinifera* seeds is performed using Sonar SA 2009 pressing machine. Soxhlet apparatus with *n*-hexane solvent is used to extract bio-oil from pro-processed seeds. Flat bottom flask, side arm layered apparatus and Erlenmeyer flask are the other apparatus used in this investigation to extract and transesterify the bio-oil from *Vitis vinifera* seeds. Further, process parameter optimization studies using tools namely RSM, ANN and ANFIS are carried out using Design expert and Matlab-2018a software.

2.2. Methods

The procured *Vitis vinifera* seeds are thoroughly segregated based on their size and shape and the adhered sand particles if any, along with other impurities are removed. The seeds are then washed with distilled water. The seeds are then sundried in ambient conditions for 48 hrs and it is followed by hot oven drying kept at 60 °C for 12 hrs for the removal of moisture. Sonar SA 2009 pressing machine is fed with one batch (100 gms) of processed *Vitis vinifera* seeds for crushing and cell wall membrane rupturing. This process resulted in oil extraction of around 3.5% by weight. The crushed seeds are then placed in the thimble of soxhlet apparatus and *n*-hexane is filled in the sedimental region as shown in Fig. 1 *n*-hexane solvent is heated upto 70 °C during which it vaporized and occupied the upper layer followed by condensation reaction in the side arm making the solvent to reach the thimble and react with the processed seeds for the extraction of remaining bio-oil. This cycle is repeated 6–8 times during which 6.4% by weight of *Vitis vinifera* bio-oil was collected. The entire cyclic process is repeated several times to extract the bio-oil from seeds of *Vitis vinifera*. In this present study, the combinational

oil extraction procedure yielded the bio-oil at an efficiency of 9.54% by weight. Few literatures [2,5,9] also supported the oil extraction efficiency between 9% and 11% through mechanical and solvent extraction methods in their studies.

2.3. Single stage transesterification

Analytical investigation with phenolphthalein based titration method estimated the free fatty acid content in *Vitis vinifera* seed oil to be 1.52%. Therefore, single stage base catalysed transesterification method is adopted to reduce the kinematic viscosity of the bio-oil and also to transform the mono-alkyl triglycerides of *Vitis vinifera* oil into fatty acid methyl esters. Investigations are conducted by considering the independent variable process parameters such as methanol to oil molar ratio (0.17–0.33 v/v), catalyst concentration (0.45–1.35 g/g) and reaction duration (0.5–1.5 hrs). Based on the literature report [1,9,10], the biodiesel formation majorly depends on variable process parameters as mentioned above. This may be due to boiling point of *n*-hexane and methanol solvent being closer to 60 °C which is also the reaction temperature of transesterification process. This causes negative and reversible reaction creating a sludge soapy formation when the reaction temperature was brought above 60 °C [4,5,7]. Fig. 1 depicts the methodological approach for bio-oil extraction and transesterification process for the yield of *Vitis vinifera* biodiesel.

2.4. Transesterification reaction - process optimization

This data set portrays that the yield of *Vitis vinifera* biodiesel mainly depends on independent variable process parameters like molar ratio, catalyst concentration and reaction time. Variation in the degree of process parameters significantly affects the biodiesel yield. In order to reduce the time and energy consumption, there is a need to optimize these parameters to obtain maximum biodiesel yield with minimal wastage of energy [6]. Therefore, a comparative study using soft computing tools like RSM, ANN and ANFIS approach has been carried out to maximize the yield of *Vitis vinifera* biodiesel. The experimental trials were performed for the pre-determined experimental condition and the results obtained were used in the mathematical tools. The central composite rotatable design (CCRD) has been used to design the experimental trials. The experiment consists of 3 variable factors and the experiment is designed with parameters in three different levels (+1, 0, -1). The number of data points in the design was estimated based on the equation (1). A three factor five level RSM based central composite rotatable design is formulated with eighth factorial six axial point and six centre point. The factorial points and axial points for particular data set will be constant while the number of centre points varies. The axial point 1.68 is chosen for the orthogonality of the model. Fig. 2 represents the variation between molar ratio and catalyst concentration at constant reaction time (A), reaction time and molar ratio at constant catalyst concentration (B) and catalyst concentration and reaction time at constant molar ratio (C) using RSM. ANN model is developed using non-linear relationship between the process variable and the experimental biodiesel yield. A feed-forward propagation loop with multi-layered perception neural network is employed to predict the results.

The dataset of the experimental yield is divided into training dataset, testing dataset and validation dataset. The experimental data obtained is fitted into model using different datasets to prevent the over-fitting of model, reduce the error and increase the reliability of the developed model. The dataset of the experimental yield is divided into training data 70% (The training dataset are the dataset which are initially used to develop a relation between the input and the output.), testing data 15% (The testing data is used to check the over-fitting of the data) and validating data 15% (The validation data is used to check the error in the developed model using training data.), which totals to 100%. The experimental data's which have been obtained can be used to predict the yield for an unknown dataset and also to predict the experimental condition for optimal yield. Training dataset usually consists of input experimental values or data's and output experimental results which is used to learn the relation between the input and output functions. The training done using a part of the experimental dataset which is known as the training dataset. Fig. 3 represents regression analysis fitting data for training, validation, testing and whole set corresponding to ANN approach [8].

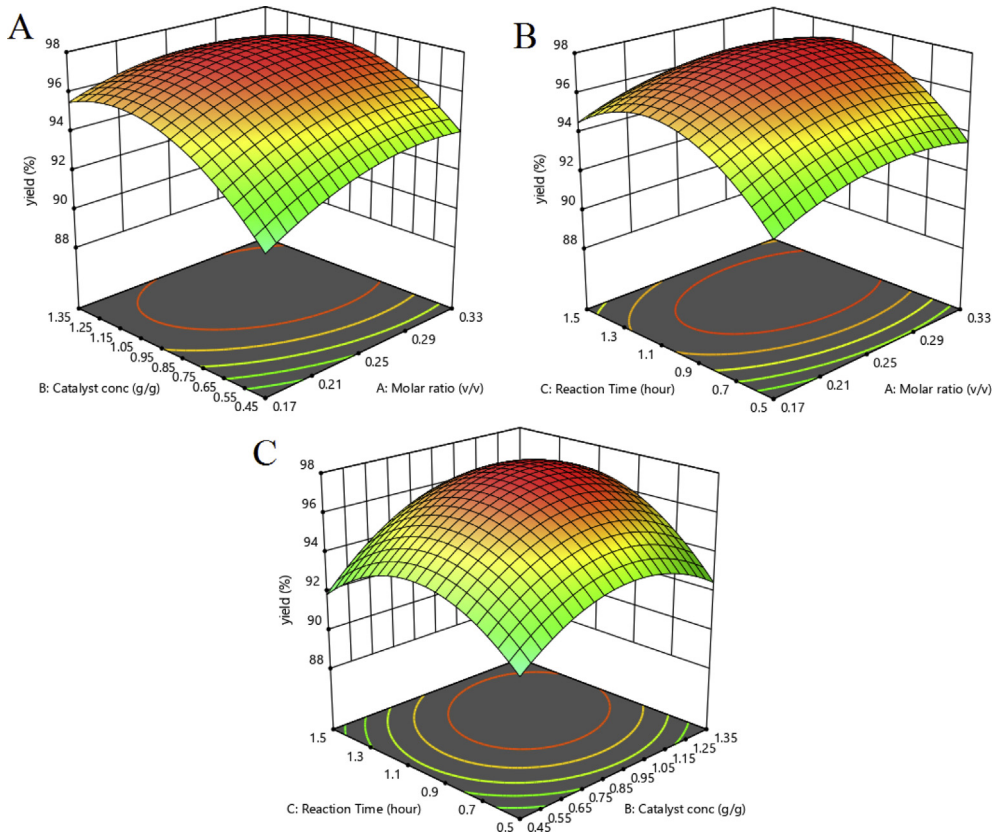


Fig. 2. Variation of Molar ratio and Catalyst concentration (A), Molar ratio and Reaction time (B) and Catalyst concentration and Reaction time (C) at constant reaction time, catalyst concentration and molar ratio respectively using RSM.

$$n = 2^k + 2(k) + \text{no. of centre points} \quad (1)$$

Where k is number of factorial data's.

ANFIS is a combinational approach of fuzzy-layer and ANN having the advantage of both the tools. Sugeno-fuzzy model having IF-then rules predicting the outcomes of transesterification reaction and biodiesel yield. Fig. 4 showcases the influence of molar ratio and catalyst concentration at constant reaction time (A), molar ratio and reaction time at constant catalyst concentration (B) and catalyst concentration and reaction time at constant molar ratio (C) respectively using ANFIS. Table 1 compares the variation in methanol to oil molar ratio, catalyst concentration and reaction time with the experimental results and the predications of RSM, ANN and ANFIS [8]. It can be seen that methanol to oil molar ratio of 0.25 v/v, 0.9 gm of NaOH catalyst concentration and 1 hr reaction time yielded a maximum experimental biodiesel of 97.4% which is in comparison with the predicted biodiesel yield of 97.184, 97.3322 and 97.18333 for RSM, ANN and ANFIS respectively as tabulated in Table 1.

The data points in 7, 10, 11, 18 and 20th experimental trial are the centre points used in the Central Composite Rotatable Design which determines the variability of the results with respect to the input. With the same experimental condition (Trial no 5, 7, 10, 11, 18 and 20) there always be errors and uncertainties which may be due to humans and instruments. There is a need to estimate the uncertainty of the experiments conducted during the transesterification process, so that the predicted

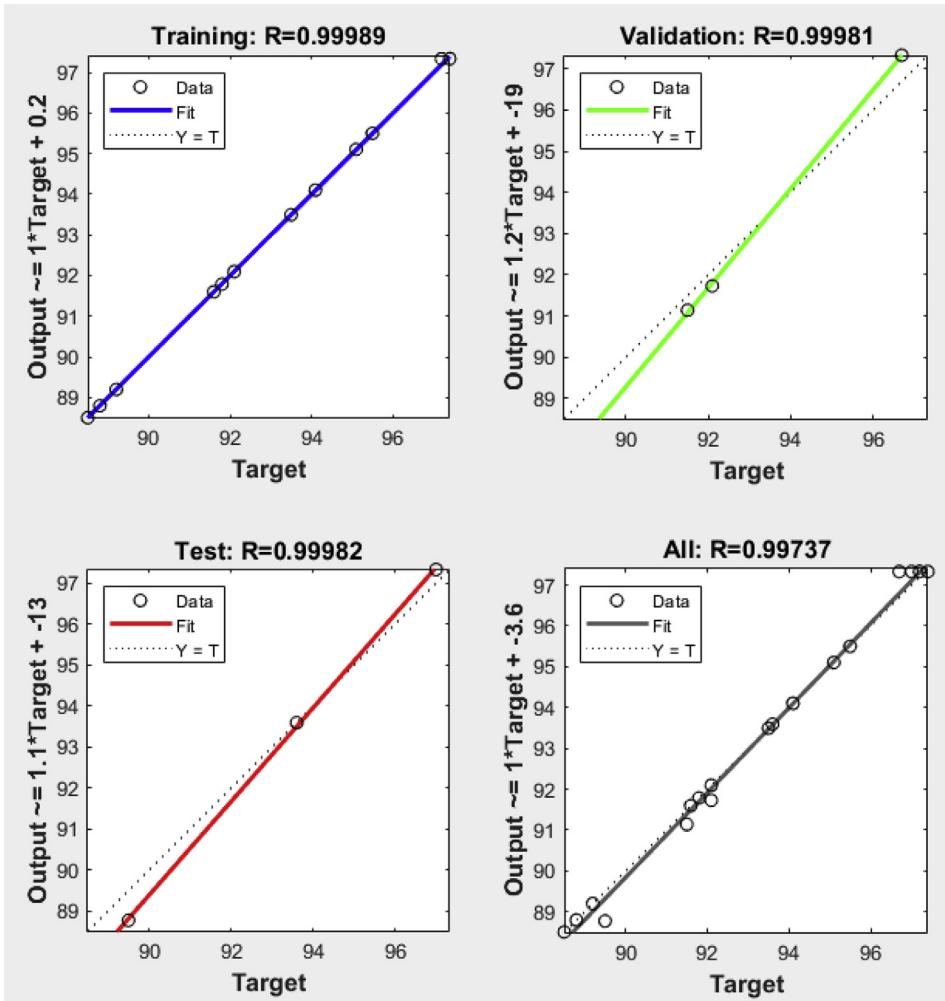


Fig. 3. ANN fitting data for training, validation, testing and whole set - Regression study.

optimum yield is with more reliability. By including same data's, the variance of the design can be calculated and the reliability of the optimum value increases. Figs. 2–4 are plotted based on the data's available in Table 1 and also with the supplementary data which predicts the RSM, ANN *Vitis vinifera* biodiesel. The plots represents the variability of any two factors keeping the third parameter as constant.

2.5. Statistical parameters

The statistical approach using parameters of Analysis of Variance (ANOVA) namely R , R^2 , Adjusted R^2 , SEP, MAE, MSE, RMSE, and MRPD are used to assess and evaluate the predictive capability of ANN, RSM and ANFIS as tabulated in Table 2. The values of MSE, RMSE, SEP and MAE are used to estimate the error in the predicted data while MRPD value measures the accuracy of the developed models [6]. The comparative data of statistical parameters are tabulated in Table 2. The transesterification experiment

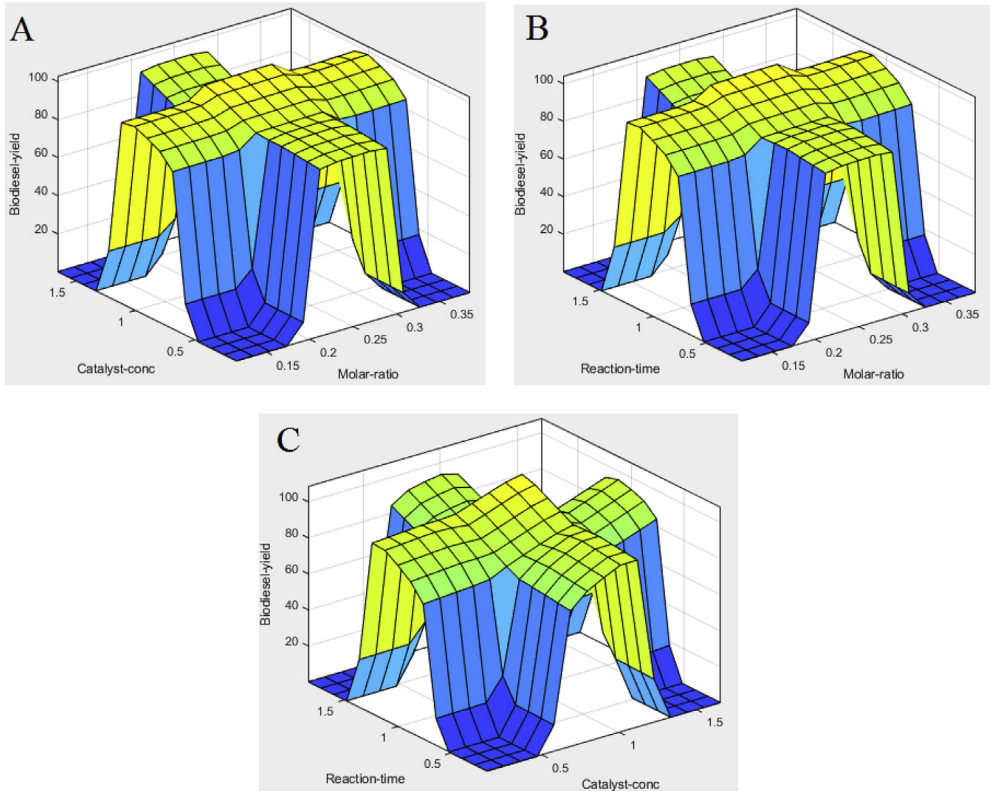


Fig. 4. Variation of Molar ratio and Catalyst concentration (A), Molar ratio and Reaction time (B) and Catalyst concentration and Reaction time (C) at constant reaction time, catalyst concentration and molar ratio respectively using ANFIS.

was performed as per the CCRD experimental design and values are tabulated. The regression analysis was performed using Response Surface Methodology. The model equation in terms of the variable parameters is given in Equations (2) and (3).

$$y = b_0 + b_1x_1 + b_2x_2 + b_3x_3 + b_{11}x_1^2 + b_{22}x_2^2 + b_{33}x_3^2 + b_{12}x_1x_2 + b_{13}x_1x_3 + b_{23}x_2x_3 \quad (2)$$

Where Y represents the biodiesel yield, b_1 , b_2 and b_3 are linear coefficients, b_{11} , b_{22} and b_{33} are quadratic coefficients, b_{12} , b_{13} and b_{23} are interactive coefficients, x_1 , x_2 and x_3 are independent variables in the process which represents methanol to oil ratio, catalyst concentration and reaction duration.

$$Y = 63.04314 + 95.13385x_1 + 21.17361x_2 + 18.71924x_3 - 13.19444x_1x_2 - 0.625x_1x_3 + 3.0x_2x_3 - 146.60416x_1^2 - 9.95854x_2^2 - 9.76347x_3^2 \quad (3)$$

Where Y is the biodiesel yield with respect to the methanol to oil ratio, catalytic concentration and reaction time.

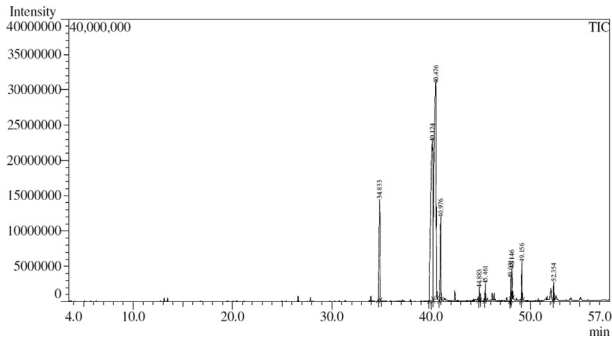


Fig. 5. GC MS chromatogram of *Vitis vinifera* seed biodiesel.

The statistical significance of the model can be analyzed by the ANOVA test. The model has F-value of 141.92 and P-value of 0.0001 which defines the model is significant with the confidence level of 95% ($p < 0.05$). The significance of the variable parameters can be analyzed through ANOVA.

2.6. Biodiesel yield optimization using RSM

The desirability function in the RSM model is used to optimize the yield of *Vitis vinifera* biodiesel. At a methanol to oil molar ratio 0.2758 v/v, catalyst concentration 1.045 gm of NaOH and reaction duration of 1.11 hrs, RSM predicted a yield of 97.62% *Vitis vinifera* biodiesel as shown in Table 4. The predicted biodiesel yield is validated experimentally which shows a yield of 97.6% with an error of 0.8% [6,8].

Table 1

Comparison of Experimental and Predicted biodiesel yield.

Trial No	Methanol to oil molar ratio (v/v)	Catalyst concentration (g/g)	Reaction duration (hrs)	Experimental Biodiesel yield (%)	Predicted Biodiesel yield		
					RSM	ANN	ANFIS
1	0.17	0.45	1.5	89.2	89.458	89.1998	89.19997
2	0.1154566	0.9	1	93.6	93.275	93.5954	93.59997
3	0.25	1.656807	1	93.5	93.713	93.4946	93.49998
4	0.3845434	0.9	1	95.5	95.786	95.4996	95.49997
5	0.25	0.9	1	97	97.184	97.3322	97.18333
6	0.25	0.143193	1	89.5	89.248	88.7745	89.49996
7	0.25	0.9	1	97.4	97.184	97.3322	97.18333
8	0.25	0.9	1.840896415	92.1	91.74	92.0976	92.09998
9	0.33	0.45	1.5	91.6	91.851	91.6015	91.59997
10	0.25	0.9	1	97.4	97.184	97.3322	97.18333
11	0.25	0.9	1	96.7	97.184	97.3322	97.18333
12	0.25	0.9	0.159103585	88.5	88.82	88.505	88.49995
13	0.33	1.35	0.5	92.1	91.87	91.7299	92.09997
14	0.17	1.35	0.5	91.5	91.27	91.1417	91.49997
15	0.17	0.45	0.5	88.8	89.02	88.7998	88.79994
16	0.33	0.45	0.5	91.8	91.515	91.7916	91.79994
17	0.33	1.35	1.5	95.1	94.9	95.1039	95.09998
18	0.25	0.9	1	97.2	97.184	97.3322	97.18333
19	0.17	1.35	1.5	94.1	94.412	94.1028	94.09998
20	0.25	0.9	1	97.4	97.184	97.3322	97.18333

Table 2

Comparison statistical parameters - ANFIS, RSM and ANN.

Parameters	ANFIS	RSM	ANN
R	0.9989	0.996	0.9976
R ²	00.9978	0.992	0.9933
Adjusted R ²	0.9974	0.991	0.9921
MSE	0.0242	0.0718	0.0667
RMSE	0.1429	0.2678	0.2582
SEP%	0.16	0.29	0.28
MAE	0.0667	0.2531	0.136
MRPD	0.0687	0.2716	0.1459

Table 3ANOVA table for *Vitis vinifera* transesterification process optimization.

Source	Sum of Squares	Df	Mean Square	F-Value	P-Value
Model	183.30	9	20.37	141.92	<0.0001 significant
A-MOR	7.61	1	7.61	53.04	<0.0001
B-CC	24.06	1	24.06	167.66	<0.0001
C-TI	10.29	1	10.29	71.70	<0.0001
MOR*TI	0.0050	1	0.0050	0.0348	0.8557
MOR*MOR	12.69	1	12.69	88.40	<0.0001
CC*CC	58.61	1	58.61	408.38	<0.0001
MOR*CC	1.81	1	1.81	12.58	0.0053
TI*TI	85.86	1	85.86	598.28	<0.0001
CC*TI	3.65	1	3.65	25.40	0.0005
Lack of fit	1.03	5	0.2054	2.51	0.1672 Not significant
Pure error	0.4083	5	0.0817	–	–

Table 4

Optimal parameter predicted by RSM.

Parameters	Values	Units
Molar ratio	0.2758	v/v
Catalytic concentration	1.045	g/g
Reaction Duration	1.11	hour
Predicted value - RSM	97.62	%
Experimental value-validation	97.7	%

Table 5Fatty acid profile of *Vitis vinifera* biodiesel.

S.No	Retention time (min)	Name of the esters	FAME's	% Conc.
1	34.833	Hexadecanoic acid, methyl ester	Palmitic acid	9.46
2	40.124	Methyl 10-trans,12-cis-octadecadienoate	Linoleic acid	36.65
3	40.476	9-Octadecenoic acid, methyl ester,(E)-	Oleic acid	42.73
4	40.976	Methyl stearate	Stearic acid	5.36
5	44.883	11-Eicosenoic acid, methyl ester	Gondoic acid	0.53
6	45.461	Eicosanoid acid, methyl ester	Gondoic acid	0.68
7	48.031	9,12-octadecadienoic acid (z, z)-,2.	Linoleic acid	0.89
8	48.146	9-Octadecenoic acid, 1,2,3-propanetriyl ester	Oleic acid	1.33
9	49.156	Methyl 20-methyl-heneicosanoate	Heneicosylic acid	1.60
10	52.354	Tetracosanoic acid, methyl ester	Lignoceric acid	0.78

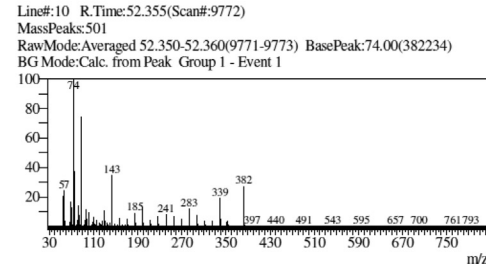
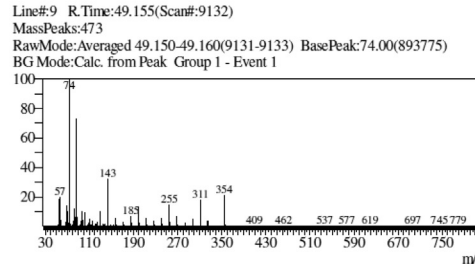
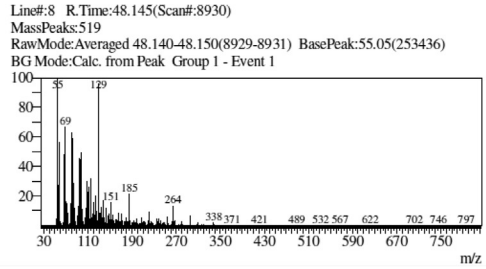
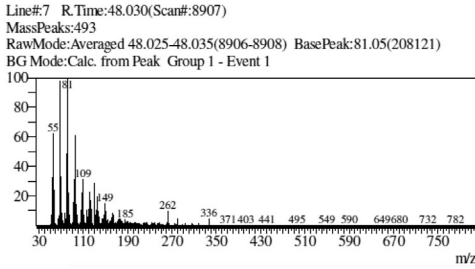
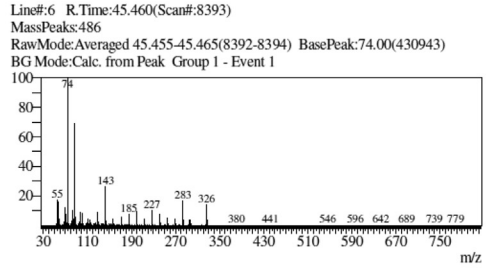
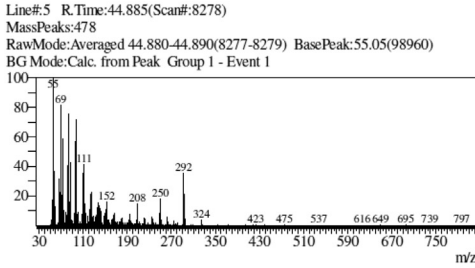
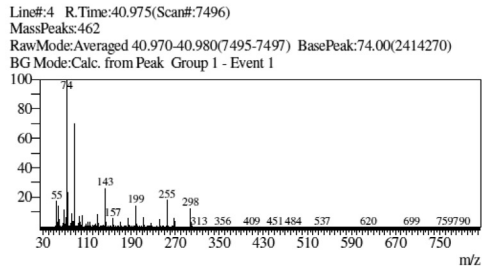
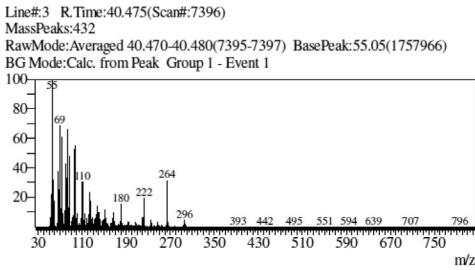
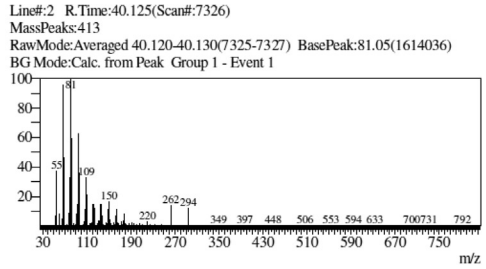
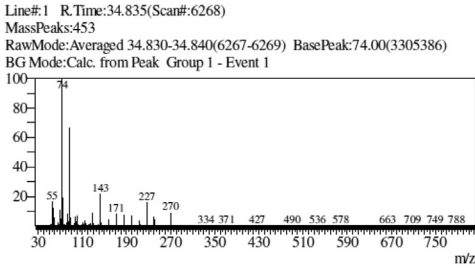


Fig. 6. GC MS fragmentation pattern of *Vitis vinifera* seed biodiesel.

2.7. Gas Chromatography Mass Spectrometry analysis

The obtained *Vitis vinifera* biodiesel was washed with distilled water and the distillate was heated upto 100 °C to remove the existence of water molecules and excess methanol. GC/MS–QP 2010 ultra with split type AoC-20i auto injector and FameWAX column is used to identify the various fatty acid methyl esters present in *Vitis vinifera* biodiesel. NIST11 library is compared and confirmed for the existence of Palmitic acid at RT 34.833, Linoleic acid at RT 40.124, Oleic acid at RT 40.476, Stearic acid at RT 40.976, Gondoic acid at RT 44.883 and 45.461, Linoleic acid at RT 48.031, Oleic acid at RT 48.146, Heneicosylic acid at RT 49.156 and Lignoceric acid at RT 52.354. The concentration of Oleic acid at 44.06% and Linoleic acid at 37.54% is found to be notable proportions in *Vitis vinifera* biodiesel.

Retention time is the time duration of each component of the *Vitis vinifera* biodiesel spends in the column after the injection of the *Vitis vinifera* biodiesel. Each component has different amount of retention time which is based on the mass, boiling point, pressure and temperature of the *Vitis vinifera* biodiesel. The *Vitis vinifera* bio-oil was also subjected to GC/MS analysis which revealed the presence of saturated fatty acids (palmitic, stearic, heneicosylic and Lignoceric acids) at 17.2% and unsaturated fatty acids (Linoleic, oleic and Gondoic acids) at 82.81%. Table 5 illustrates the presence of various fatty acid methyl esters and its concentration in *Vitis vinifera* biodiesel. The spread area of the chromatogram, retention time and peak value were used to estimate the percentage of FAME in *Vitis vinifera* biodiesel. Fig. 5 and Fig. 6 depicts the fragmentation patterns and GC/MS chromatogram of the *Vitis vinifera* biodiesel [9,10].

Conflict of interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.dib.2019.104298>.

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