



Research article

Optimization of biodiesel yield from waste cooking oil and sesame oil using RSM and ANN techniques

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ABSTRACT

In the era of global energy crises and the pressing concern of fossil fuel depletion, the quest for sustainable alternatives has become paramount. The current study aims to optimize biodiesel extraction from a combination of waste cooking oil (WCO) and sesame seed oil (SSO) through response surface methodology (RSM) and artificial neural network (ANN). The cold flow properties of biodiesel produced from WCO are a major obstacle to the commercial use of biodiesel. On the other hand, SSO possesses better oxidation stability and cold flow properties. A mixture of waste cooking oil (i.e. 70 % by volume) and sesame seed oil (i.e. 30 % by volume) has been prepared for biodiesel production via a microwave-assisted transesterification process. For biodiesel yield optimization, the interaction among the operating parameters is developed by RSM, whereas biodiesel yield is predicted by ANN. The operating parameters include reaction speed, RPM (100–600 rpm), reaction time (1–5 min), methanol to oil ratio (8:1–12:1 v/v), and catalyst concentration (0.1–2 % w/w). The highest biodiesel yield of 94 % is found at a reaction speed of 350 rpm, reaction time of 3 min, catalyst concentration of 1.05 w/w, and methanol to oil ratio of 10:1. Furthermore, it is discovered that when estimating biodiesel production rate depending on reaction constraints, ANN shows lower comparative error compared to RSM. The results show that ANN outperforms RSM in terms of percentage improvement when it comes to biodiesel production prediction.

Nomenclature

Nomenclature	Description	Nomenclature	Description
ANN	Artificial neural network	RASE	Root average squared error
AV	Acid value	RSM	Response surface methodology

(continued on next page)

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CV	Calorific value	R ²	Coefficient of determination
DOE	Design of experiment	SSO	Sesame seed oil
FAME	Fatty acid methyl ester	SSOME	Sesame seed oil methyl ester
FFA	Free fatty acid	SSE	The sum of squares error
GCMS	Gas chromatography-mass spectrum	TFEN	Total final energy consumption
HSD	High-speed diesel	WCO	Waste cooking oil
MAD	Mean absolute deviation	W70S30	70 % waste cooking oil and 30 % sesame seed oil

1. Introduction

Currently, the world is dealing with energy-related problems, such as rising energy prices, increasing energy demand, and the steady depletion of fossil fuels [1]. By 2025, the worldwide petroleum demand will be increased by 40 % [2]. Between 2013 and 2018, there was an increase in total final energy consumption (TFEC) of 25.3 exajoules (EJ), or roughly 1.4 % per year. About 32 % of the world’s total energy demand in 2017–18 came from the transportation sector, and fossil fuels were used to meet 96.7 % of these energy needs [3]. To tackle energy crises, alternative energy resources must be adopted [4,5]. Fossil fuels (petrol and diesel) combustion contribute significantly to the greenhouse gas emissions that cause climate change and environmental deterioration [6,7]. As a result, developing greener, alternative energy sources is desirable and required globally [8]. Due to these challenges, there is renewed interest in researching alternate energy sources, one of which is biodiesel [9,10]. By using biodiesel, a renewable and biodegradable fuel, the environmental and energy issues brought on by conventional fossil fuels are being mitigated [11]. Using different feedstocks, such as animal fats and vegetable oils, to transesterify triglycerides, biodiesel is a clean-burning fuel that emits fewer greenhouse gases [12]. Moreover, it does not need significant engine modifications for use in engines. The WCO offers a compelling alternative because of its cost-effectiveness and environmental benefits while producing biodiesel [12]. It is a resource that is both abundantly available and underutilized. WCO is a byproduct of the food industry that is considered renewable and sustainable. In addition, WCO is an inexpensive feedstock, making it a popular choice for manufacturing biodiesel [13]. There are challenges that need to be overcome to employ WCO to produce biodiesel. Water, free fatty acids, and sediments are among the contaminants found in WCO. These impurities could affect the biodiesel produced from WCO in terms of quality. However, sophisticated optimization techniques are required to handle the complexity of feedstock variability and reaction parameters to maximize biodiesel output from used cooking oil [14,15]. One of the challenges in using WCO for the manufacture of biodiesel is its cold flow characteristic [14]. It could be difficult to pump and inject WCO into a diesel engine because of its high viscosity at low temperatures [16]. Higher viscosity at lower temperatures can cause engine problems, including cold start cranking and wasteful fuel use. There are several methods to improve the cold flow properties of WCO, including combining two or more oils or adding surfactants and antioxidants [17]. Combining WCO with an oil that burns more readily, such as soybean oil, is one way [18]. A catalyst can also be used to convert the free fatty acids in WCO into esters.

Vegetable oil SSO is a potential feedstock for the production of biodiesel due to a number of its qualities [19]. This research work aims to explore the unexplored potential of SSO as an alternative feedstock for biodiesel production. Because of the inherent preservatives and antioxidants that fortify the oil’s resistance to rancidity, it has unique cold flow characteristics and oxidation stability [20]. It is an inexpensive oil that is widely accessible. It is an excellent fuel for diesel engines due to its high cetane number and outstanding cold flow characteristics [19]. It is simpler to pump and inject into a diesel engine since it has a lower viscosity than WCO at low temperatures. This can enhance the cold start performance and fuel efficiency of diesel engines [21,22].

This study investigates the combined use of WCO and SSO to produce biodiesel. It presents a strategy to increase biodiesel output by fusing RSM and ANN approaches [23]. RSM is a statistical technique that is used to pinpoint the variables influencing biodiesel yield and establish the ideal production environment [24]. Using RSM, this study aims to fully investigate the intricate interactions between variables that affect the transesterification process to determine the ideal circumstances for producing the highest amount of biodiesel. In addition, by identifying complex patterns and correlations in the data, the use of ANN—a potent machine-learning method inspired by the human brain—promises to substantially improve the accuracy of optimization process. Because ANNs are unparalleled in their capacity to model complex, non-linear connections and provide predictions using massive data sets, they have become indispensable tools in a wide range of research domains [25,26]. An artificial neural network (ANN) is made up of layers of linked “neurons” or nodes, and each neuron in an ANN does simple mathematical operations [27]. Since biodiesel output from WCO and SSO involves intricate and usually non-linear interactions among critical reaction parameters, ANNs offer a unique advantage. The findings have the potential to influence future studies and industrial practices in biofuel production, promoting efficient and eco-friendly alternatives to conventional fuels.

2. Materials and methods

2.1. Materials

SSO is bought at a local Pakistani market, while WCO is gathered from nearby restaurants. Sigma Aldrich is where the additional chemicals, including the alcohol and catalyst, are bought. Methanol possesses 99.9 % purity, and KOH possesses 85 % purity level.

2.2. Biodiesel yield

Determining the free fatty acid value (FFA) of any feedstock is the most critical stage in the synthesis of biodiesel. The measured acid value of SSO is 0.8976 mgKOH/g, which is acceptable for the production of biodiesel. Therefore, there is no need for the acid treatment of the feedstock. Contrary, the acid value (AV) of WCO is comparatively higher and found to be 3.8 mgKOH/g, so the acid treatment requires lowering the AV. Using mineral acid, such as H₂SO₄, the acid treatment or acid esterification procedure is carried out in two parts. 30 % SSO and 70 % WCO are combined to create a combination.

The raw oil combination is converted into biodiesel using a microwave-assisted setup in the presence of methanol and a KOH catalyst. With a catalyst concentration of 0.1–2 w/w, methanol to oil ratio of 8:1–12:1 v/v, and reaction rates ranging from 100 to 600 RPM for 1–5 min, methanol is injected into an oil mixture in the presence of KOH. The purpose of routinely washing biodiesel in hot water is to remove contaminants like catalysts and leftover methanol. Distilled water is used to wash the biodiesel; this procedure is continued until the distilled water turns transparent. Biodiesel yield is calculated using Equation (1) [28]. The schematic diagram of biodiesel generation through microwave-assisted transesterification is shown in Fig. 1.

$$\text{Yield} = \frac{\text{Amount of biodiesel produced}}{\text{Amount of oil used}} \times 100 \quad (1)$$

2.3. Biodiesel attributes analysis

A bomb calorimeter is used to determine the calorific value, and the Cleveland open cup apparatus is employed to determine the flash point. The FAME composition is ascertained using the GCMS-QP2010 plus. Helium gas is the carrier gas. The AV of the W70S30 oil mixture has been calculated by titration method. In this method, 50 ml of distilled water is taken and added to 0.5N KOH solution. The second step is to prepare the indicator for this purpose. 0.25g phenolphthalein is added to 25 ml ethanol solution. The third step is to prepare 50 ml ethyl alcohol solution by adding 95 % ethyl alcohol and 5 % distilled water. At the end pour 1 ml indicator into a solution of W70S30 and 95 % ethyl alcohol solution and titrate this mixture until it turns into light pink color. Equations (2) and (3) are used to compute the AV and FFA of W70S30, respectively [28].

$$\text{Acid Value} = \frac{56.1 \times N \times V}{W} \quad (2)$$

$$\text{FFA} = \frac{\text{AV}}{2} \quad (3)$$

where W is the weight of SSO utilized, N is the quantity of KOH, and V is the volume of KOH and distilled water used for the titration.

2.4. Biodiesel yield optimization

Biodiesel yield is optimized by using two different techniques i.e. RSM and ANN. The way the operational parameters interact is

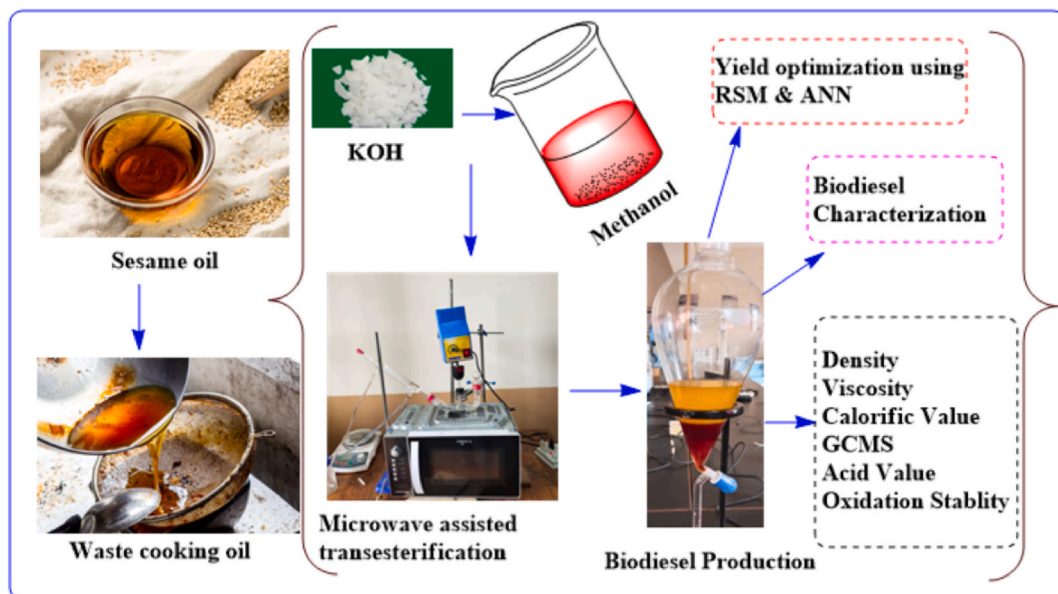


Fig. 1. Schematic diagram of biodiesel yield through microwave-assisted transesterification technique.

provided by RSM, while ANN provides the predictive model for biodiesel yield. The four primary operational parameters that affect biodiesel yield are the methanol-to-oil ratio, reaction speed, catalyst concentration, and reaction time. Using JMP Pro 16 software, a design of experiments (DOE) is created to maximize biodiesel output. Table 1 displays the ranges of these independent variables. Biodiesel production is the dependent variable, while the operational parameters of the microwave-assisted transesterification process are the independent variables. The primary analytical steps of regression analysis, response surface mapping, and analysis of variance (ANOVA) are carried out to establish ideal conditions. The yield of biodiesel is also predicted using ANN. To determine the benefits of the two approaches for yield prediction and optimization, the optimization of model prediction for RSM and ANN is compared.

3. Results and discussion

3.1. Physiochemical characteristics of biodiesel

Table 2 displays the physiochemical properties of the biodiesel produced from the SSO and WCO combination. These features are contrasted with the traditional HSD. The comparison demonstrates that no adjustments are necessary for the use of biodiesel in compression ignition engines. Another important characteristic is to determine the composition of biodiesel. It is determined by using GCMS analysis. Table 3 represents the different fatty acid methyl esters (FAMES) that compose biodiesel and can be identified with the aid of GC-MS. Peaks belonging to different FAMES may be seen in a typical biodiesel GC-MS chromatogram, with retention durations showing the relative volatility of each FAME. These FAMES' identities are verified by the mass spectra linked to each peak. Methyl palmitate, methyl stearate, methyl oleate, and methyl linoleate are examples of FAMES and their quantities.

3.2. Yield optimization

Based on Table 1, for four independent variables having three distinct levels, a full factorial DOE requires 81 experimental runs. However, an experimental matrix of 26 samples is designed here using the JMP-Pro software. The biodiesel yield is measured for each case and is tabulated in Table 4.

3.3. Impact of operating parameters on biodiesel production

To search out the effects of varying control factors on the response, surface plots are drawn. Fig. 2 (a) depicts the impact of reaction speed and reaction time on biodiesel yield. Also, it illustrates how the biodiesel yield initially decreases as response time increases, but only for a certain range. After that range, the biodiesel yield gradually decreases. Similar to reaction time, reaction speed affects the production of biodiesel. The first decrease in biodiesel yield with increased reaction speed and time is caused by the reversibility of the transesterification reaction, soap formation, and catalyst degradation. The final steady improvement in biodiesel production can be attributed to the reversibility of the saponification reaction and the progressive slowing down of the catalyst degradation. The transesterification reaction, in which oil molecules react chemically with alcohol to generate esters, is the main step in the production of biodiesel [29]. The forward reaction quickens as the response rate is increased, which moves the equilibrium position to the right. As a result, more esters are produced, which increases the amount of biodiesel produced. On the other hand, an overabundance of reaction speed increase might potentially quicken the reverse reaction. This may cause the equilibrium point to move to the left, which would reduce the amount of biodiesel produced. Fig. 2 (b) illustrates how the biodiesel yield is increased with increasing catalyst concentration and reaction speed, but it also gradually declined after reaching a maximum. Catalyst poisoning is more likely with greater catalyst concentrations. This is a result of an increased quantity of catalyst molecules available for impurity reactions. The biodiesel yield may consequently drop, and the reaction time may slow down. An increased catalyst concentration results in a greater abundance of catalyst molecules, so enhancing the rate of the reaction and ultimately yielding a higher amount of biodiesel [30]. Nevertheless, an excessive concentration of catalyst might lead to catalyst deactivation, resulting in a deceleration of the reaction. The ideal catalyst concentration typically falls within the range of 1 %–2 %. Fig. 2 (c) illustrates how the yield of biodiesel increases steadily as the methanol-to-oil ratio rises, but eventually starts to decline as a result of soap formation and methanol evaporation. This decrease is also accompanied by an increase in reaction speed. Fig. 3 (a) illustrates the significance of the two parameters: reaction time and catalyst concentration. It demonstrates how the biodiesel yield increases with increasing catalyst concentration but decreases with increasing reaction time. Both the catalyst concentration and reaction time have an impact on the biodiesel yield. Up to a certain degree, the catalyst concentration enhances the biodiesel yield. Up until a certain point, the biodiesel yield likewise rises with reaction time; after that, soap, water, and FFA production cause the yield to start declining. However, because of the esterification process's reverse, biodiesel production can begin to rise once more at extremely high reaction times. Fig. 3 (b) illustrates how biodiesel production rises

Table 1
Process parameters with their levels.

Operating parameters	Units	Values levels
Catalyst concentration	w/w	0.1, 1.05, 2
Reaction speed	RPM	100, 350, 600
Methanol-to-oil ratio	v/v	8:1, 10:1, 12:1
Reaction time	Minute	1, 3, 5

Table 2
Physicochemical characteristics of biodiesel and HSD.

Properties	HSD W70S30	biodiesel
Density at 15°C (kg/cm ³)	839.4	879.5
Acid Value (mg KOH/g oil)	0.15	0.3966
Flash Point (°C)	150	78.50
Viscosity at 40°C (mm ² /s)	2.87	5.08
Calorific Value (MJ/kg)	45.67	39.42
Oxidation Stability (h)	13.20	6.50

Table 3
Chemical composition of biodiesel.

Chemical Name	Chemical Formula	W70S30 biodiesel
Palmitic Acid	C16:0	7.85
Methyl Erucate	C22:1	2.2
Linoleic Acid	C18:2	34.66
Linolenic Acid	C18:3	4.33
Palmitoleic Acid	C16:1	0.6
Methyl Arachidate	C20:0	0.57
Oleic Acid	C18:1	46.94
Stearic Acid	C18:0	2.85

Table 4
Interaction between the operational parameters and the yield from the experiment and RSM, both real and predicted.

Run	Catalyst concentration	Reaction speed	Methanol-to- oil ratio	Reaction time	Actual yield	Predicted yield
	w/w	RPM	v/v	Minute	(%)	(%)
1	2	600	8	1	82	82.0460
2	0.1	600	12	1	77	76.6571
3	0.1	600	12	5	75	75.7543
4	2	100	8	1	80	80.9765
5	0.1	350	10	3	75	79.8539
6	0.1	600	8	1	76	76.9765
7	0.1	100	8	5	79	80.7543
8	2	600	12	1	78	77.9765
9	0.1	100	8	1	79	76.6571
10	1.05	350	8	3	86	85.2984
11	2	100	12	1	78	78.1571
12	2	100	12	5	77	77.7543
13	1.05	350	10	3	83	86.7714
14	0.1	600	8	5	84	81.8238
15	0.1	100	12	5	78	75.9349
16	1.05	100	10	3	82	85.8539
17	1.05	600	10	3	89	86.2984
18	2	350	10	3	87	83.2984
19	1.05	350	12	3	79	80.8539
20	1.05	350	10	1	86	87.9650
21	2	600	8	5	85	88.1432
22	2	600	12	5	78	78.3238
23	1.05	350	10	3	94	84.7714
24	2	100	8	5	88	86.3238
25	0.1	100	12	1	79	77.5876
26	1.05	350	10	5	91	90.1873

with a rise in the methanol-to-oil ratio but progressively declines for a while due to soap formation and other factors. Reaction time has a significant effect on biodiesel yield as well. As reaction time increases, biodiesel yield first decreases before steadily increasing again after that. At very high response times, the biodiesel yield can begin to increase once more. This is due to the reversibility of the esterification reaction [31]. The esterification reaction will begin to reverse and transform the triglycerides back into glycerol and biodiesel if the reaction period is sufficiently long. The relevance of the two parameters, i.e., the methanol-to-oil ratio and catalyst concentration, is demonstrated in Fig. 3 (c). As these parameters rise, so does the biodiesel yield.

A statistical method ANOVA is employed to ascertain whether one or more independent variables, such as transesterification reaction conditions, and a dependent variable, such as biodiesel yield, have a significant relationship. The model is regarded as statistically significant if the F-statistic is higher than the crucial F-value. Table 5 indicates the ANOVA of independent variables and the dependent variable used in biodiesel yield optimization via RSM. This indicates that the independent variables and the dependent

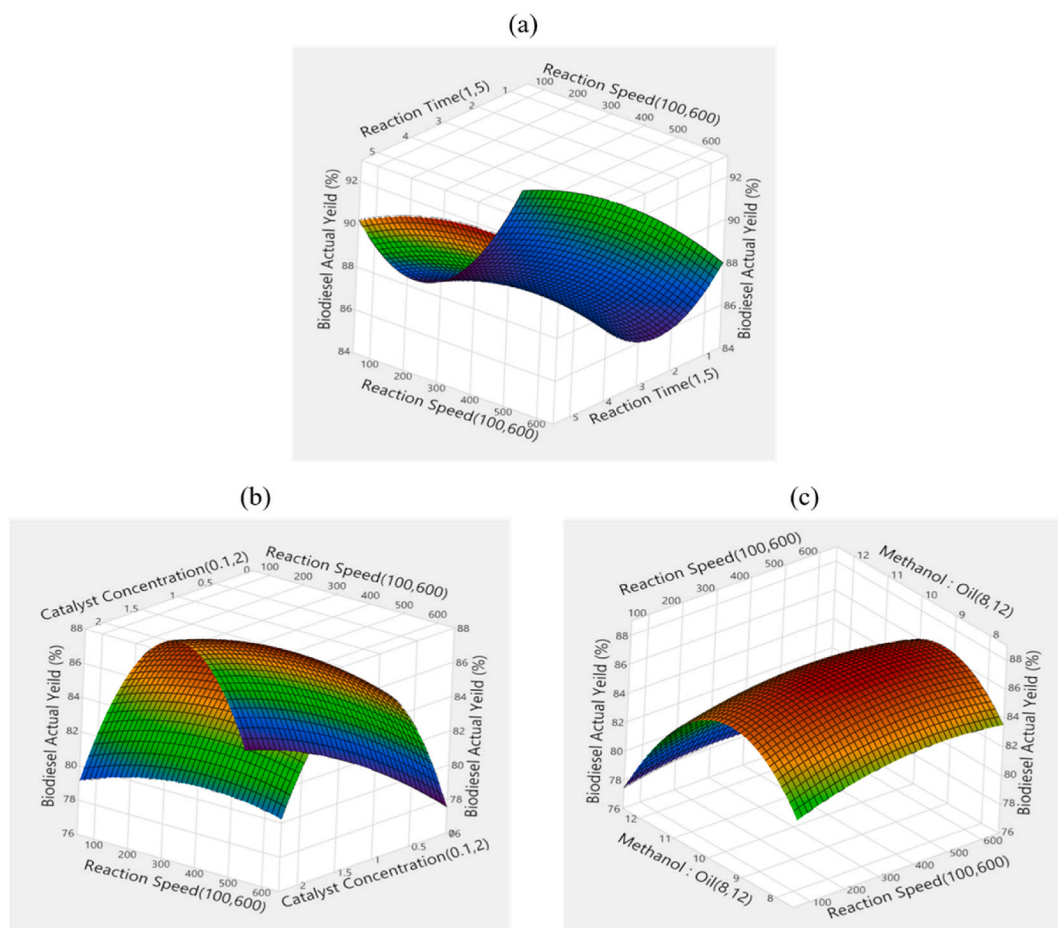


Fig. 2. (a) Impact of reaction speed and reaction time (b) Impact of reaction speed and catalyst concentration (c) Impact of methanol-to-oil ratio and reaction speed on biodiesel yield.

variable have a substantial relationship.

In addition, optimal operating conditions for a given response is carried out using the desirability function approach. The desirability function measures how well a set of input variables meets the response's specified objectives. Fig. 4 shows the optimum operating parameters for getting optimum biodiesel yield. Interestingly the optimum operating parameters determined by the desirability function is the same as achieved by the experimental findings i.e. reaction speed 350 rpm, catalyst concentration (1.05 w/w), reaction time (3 min), and methanol-to-oil ratio (10:1).

3.4. Validation of ANN predictive model

In the final section of the present study, the ANN technique is used to forecast the biodiesel yield. A prediction model of an ANN is validated using K-fold cross-validation. In the K-fold cross-validation method, the ANN model is trained on K-1 folds after the training data has been divided into K folds [32]. After that, the model is assessed using the remaining fold. The validation metric is the model's average performance across all K folds, and this process is repeated K times. In holdout validation, the training data is divided into two sets using this method: a training set and a holdout set. After the ANN model has been trained on the training set, it is evaluated using the holdout set. The holdout set should not be used to train the model, as this might lead to overfitting. Although the validation set is usually smaller than the holdout set, this technique is comparable to holdout validation. The number of hidden layers and neurons in each layer—two hyper-parameters of the ANN model—are adjusted using the validation set. The holdout set is used to evaluate the model after the hyper-parameters have been adjusted. The network diagram is illustrated in Fig. 5. The R^2 and standard deviation values for the training and test sets are displayed in Table 6. For training, the R^2 value is almost equal to 0.802063, and for validation, it is 0.9554803. As a result, it validates a precise model that is derived from the training and validation datasets. Fig. 6 (a, b) depicts the training and validation models of biodiesel predicted yield. All the points in the training segment lie on a straight line with a slope of 1. An optimization technique is employed on the ANN to accurately forecast a maximum yield i.e. 94 %. The operating parameters used for this optimization are 1.05 w/w for catalyst concentration, 350 RPM for reaction speed, 10:1 v/v for methanol: oil ratio, and 3 min

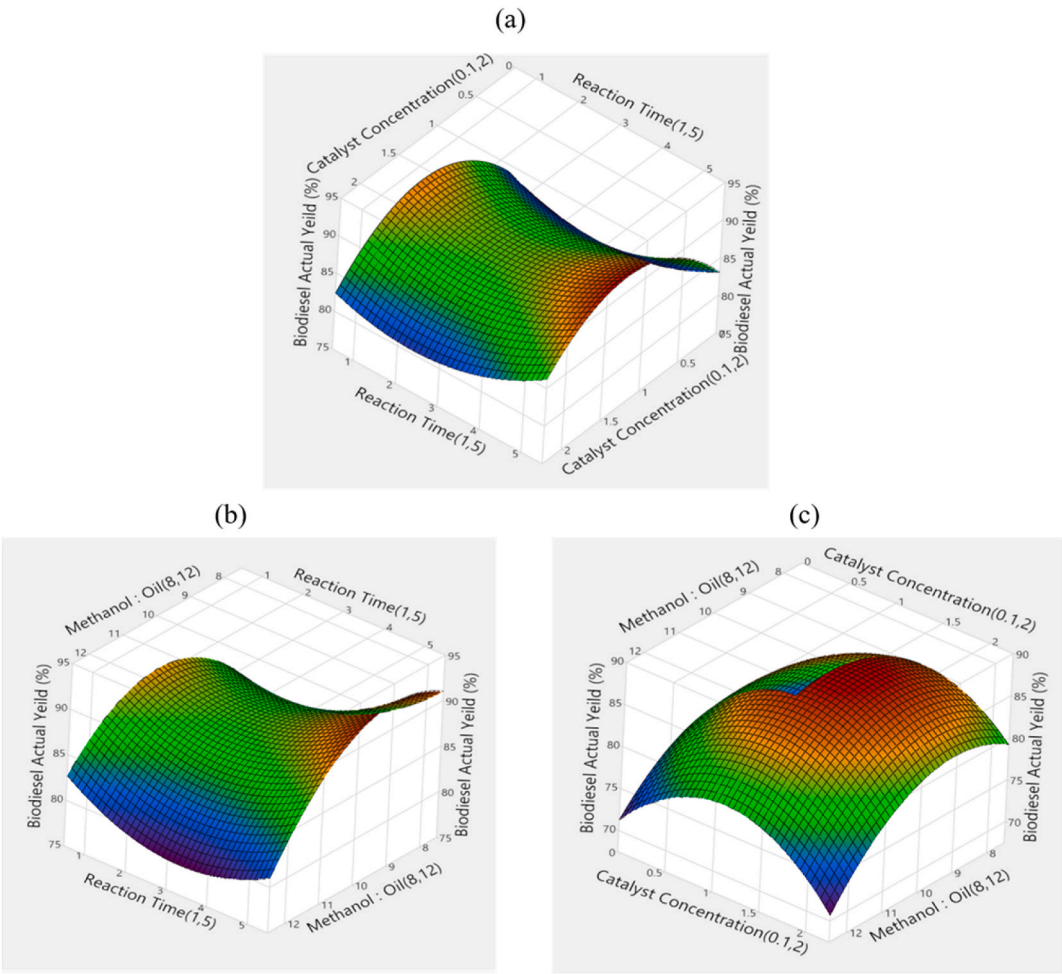


Fig. 3. (a) Impact of reaction time and catalyst concentration (b) Impact of reaction time and methanol-to-oil ratio (c) Impact of catalyst concentration and methanol-to-oil ratio, on biodiesel yield.

Table 5
ANOVA results.

Source	Nparm	DF	Sum of Squares	F Ratio	Prob > F
Reaction Speed (100,600)	1	1	0.888889	0.0576	0.8148
Methanol: Oil*Methanol: Oil	1	1	34.969570	2.2642	0.1606
Reaction Time (1,5)	1	1	22.222222	1.4389	0.2555
Reaction Time*Methanol: Oil	1	1	33.062500	2.1407	0.1714
Catalyst Concentration (0,1,2)	1	1	53.388889	3.4568	0.0899
Methanol: Oil (8,12)	1	1	88.888889	5.7554	0.0353*
Catalyst Concentration* Catalyst Concentration	1	1	69.122009	4.4755	0.0580
Reaction Speed*Reaction Time	1	1	0.562500	0.0364	0.8521
Reaction Speed*Catalyst Concentration	1	1	0.562500	0.0364	0.8521
Reaction Time* Catalyst Concentration	1	1	1.562500	0.1012	0.7564
Reaction Speed*Methanol: Oil	1	1	1.562500	0.1012	0.7564
Reaction Speed*Reaction Speed	1	1	1.237863	0.0801	0.7824
Catalyst Concentration*Methanol: Oil	1	1	14.062500	0.9105	0.3605
Reaction Time*Reaction Time	1	1	13.603717	0.8808	0.3681

for the reaction time.

4. Conclusions

A transesterification method with microwave assistance is used to turn a combination of two oils—30 % sesame oil and 70 % waste

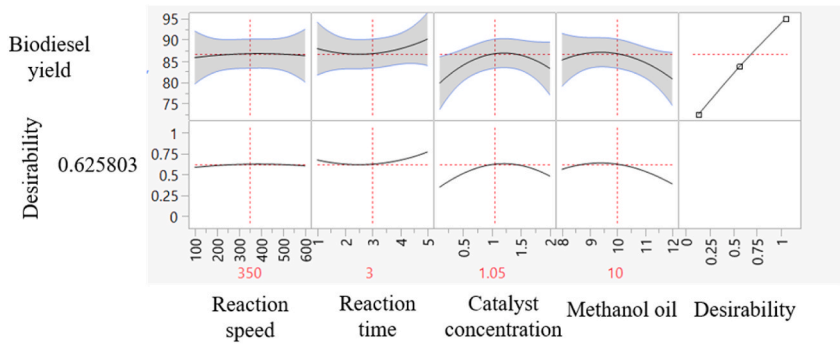


Fig. 4. Optimum operating parameters for biodiesel yield.

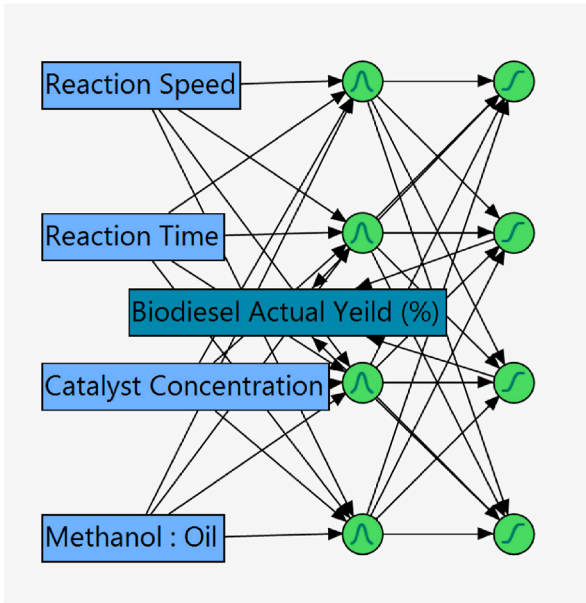


Fig. 5. Network diagram of ANN model.

Table 6
Training and validation models of ANN technique.

Training		Validation	
Biodiesel Actual Yield (%)		Biodiesel Actual Yield (%)	
Measures	Value	Measures	Value
R Square	0.802063	R Square	0.9554803
RASE	2.2268133	RASE	1.0484517
MAD	1.5486167	MAD	0.8877195
LogLikelihood	37.731671	LogLikelihood	13.196277
SSE	84.297857	SSE	9.8932579
Sum Freq	17	Sum Freq	9

cooking oil—into biodiesel. The biodiesel yield is determined by the operational parameters. The interaction between operational factors has been developed using the RSM technique, which not only provides the interaction between dependent variables but also predicts the yield of biodiesel. A maximum of 94 % biodiesel may be produced at a reaction speed of 350 rpm, catalyst concentration of 1.05-w/w, a reaction time of 3 min, and methanol to oil ratio of 10:1. When determining biodiesel yield based on reaction parameters, ANN performs better than RSM because RSM is limited in its ability to capture complex nonlinear relationships. The R2 value for training is approximately 0.802063, whereas for validation it is 0.9554803. The unique feedstock combination and optimization techniques highlight the practical applicability and scalability of the proposed methods.

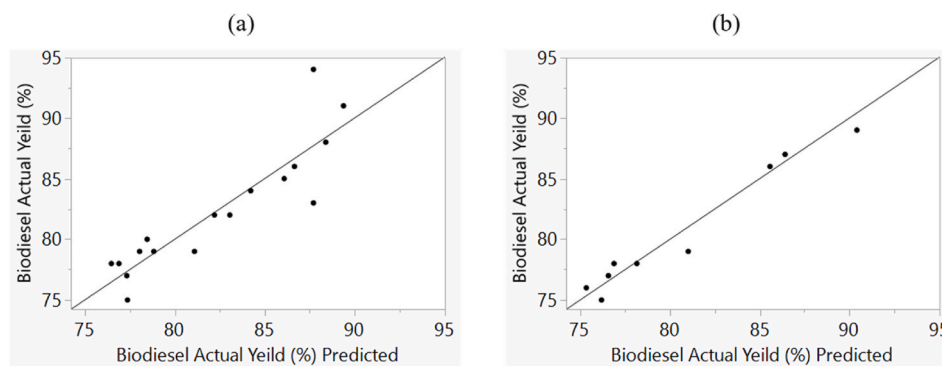


Fig. 6. Biodiesel actual yield (%) predicted by ANN technique (a) Training model (b) Validation model.

Data availability statement

Data will be made available on request.

CRediT authorship contribution statement

Waqar Bajwa: Writing – review & editing, Writing – original draft, Validation, Software, Methodology, Investigation, Formal analysis, Data curation. **Adeel Ikram:** Writing – review & editing, Writing – original draft, Visualization, Supervision, Resources, Project administration, Methodology, Investigation, Funding acquisition, Conceptualization. **Muhammad Ali Ijaz Malik:** Writing – review & editing, Project administration, Methodology, Investigation. **Luqman Razzaq:** Writing – original draft, Software, Investigation, Formal analysis, Conceptualization. **Ahmed Raza Khan:** Visualization, Methodology, Investigation. **Afsah Latif:** Writing – review & editing, Visualization, Methodology, Investigation. **Fayaz Hussain:** Writing – review & editing, Resources, Funding acquisition. **Atika Qazi:** Writing – review & editing, Visualization, Resources, Investigation.

Declaration of competing 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.

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