mesi
e-issn. 2798-5245

Mechanical Engineering
for Society and Industry
Vol. 1, No. 1 (2021) pp 22-30
https://doi.org/10.31603/mesi.5320

Type of contribution:
Editorial
▶ Research Paper
Case Study
Review Paper
Report of Tech. Application

Biodiesel Production from Waste Cooking Oil: Characterization, Modeling and Optimization

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This article contributes to:

7 AFFORDABLE AND CLEAN ENERGY

SUSTAINABLE DEVELOPMENT GOALS

Highlights:

- The high viscosity of waste cooking oil (WCO) was converted to biodiesel (WCOBD) through a transesterification process
- Fuel properties are measured and observed within the scope of ASTM standards (ASTM D6751) and fatty acid profiles captured by chromatography
- RSM optimization and ANN modeling for biodiesel production have been tested experimentally
- Both RSM and ANN techniques are accurate in predicting WCOBD results based on coefficient correlation

Abstract

In this study, waste and discarded cooking oils (WCO) of palm, sunflower, rice bran and groundnut oils are collected from local restaurants. The high viscous WCO was converted into waste cooking oil biodiesel (WCOBD) by a single-stage transesterification process. During the transesterification process, the important parameters which show a significant change in biodiesel yield are studied using the optimization tool of response surface methodology (RSM). Results reported that 91.30% biodiesel yield was achieved within L18 experiments and NaOH catalyst was identified as the most influential parameter on WCOBD yield. Artificial Intelligence (AI) based modeling was also carried out to predict biodiesel yield. From AI modeling, a predicted yield of 92.88% was achieved, which is 1.70% higher than the RSM method. These results reveal the prediction capabilities and accuracy of the chosen modeling and optimization methods. In addition, the significant fuel properties are measured and observed within the scope of ASTM standards (ASTMD6751) and fatty acid profiles from chromatography reveal the presence of high unsaturated fatty acids in WCOBD. Therefore, utilizing the waste cooking oils for biodiesel production can mitigate the global challenges of environmental and energy paucity.

Keywords: Biodiesel, Waste cooking oil, Transesterification, High viscous biodiesel

Article info
Submitted:
2021-05-20
Revised:
2021-07-24
Accepted:
2021-07-25

Publisher
Universitas Muhammadiyah Magelang

1. Introduction

Energy resources play a very vital role in improving the country's economy and development. Unfortunately, the available energy resources, especially the petroleum reserves, are restricted to certain parts of the globe and currently, they are declining at a faster rate due to rapid utilization with an increase in the global population [1]–[3]. Statistical predictions on the crude oil reserves reveal that the available resources may be extinct within a short period [4]. On the other hand, environmental air pollution is also increasing due to harmful emissions from petro-diesel engines. Therefore, the search for renewable and environment-friendly fuels is gaining wide popularity.

Biofuels, obtained from different plant and animal oil feedstocks, resemble a better solution to mitigate energy and environmental challenges [5], [6]. Research on alternative fuels, especially biodiesels, is gaining significance due to good quality combustion, resulting in low exhaust emissions and eco-friendly fuel. In general, biodiesels are referred to as esters formed from mono-alkyl extracted from various plants and animal oils. These oils are insoluble in water due to hydrophobic substances related to the plant and animal kingdom [7]. Due to the presence of high free fatty acids in raw oils, the kinematic viscosity is more. Hence, it was not advised to use directly in diesel engines. Therefore, viscosity reduction techniques are used to lower the high viscosity in raw oils [8].

On the other hand, the cost analysis for biodiesel production reveals that 60-70% was related to the type of oil used for production [9]. Therefore, low-cost feedstocks like non-edible oils of jatropha, neem, pongamia, mahua, animal fat, etc., are preferred for biodiesel production [10]. Similarly, used or waste cooking oils (WCO) are also gaining more significance for biodiesel production because the availability of WCO is more in populated countries like India and China.

After utilizing the oils for cooking purposes, they are discarded, which results in contamination and environmental pollution. Huge litres of WCO from hotels, restaurants and households are discarded every year results in polluting land and marine life. Therefore, converting the WCO into a useful energy source helps to reduce the reliance on imports and promote local employment. Literature [11], [12] on WCO for biodiesel utilization reveals that the cost for biodiesel production is reduced with the utilization of WCO. Furthermore, the waste cooking oil biodiesel (WCOBD) properties are also meet the international standards of ASTM and EN [12].

Due to the wide range of advantages in using WCO for biodiesel production, an attempt was made to characterize its important properties and achieve a profitable yield. For this purpose, statistical tools in optimization and modern artificial intelligence (AI) modeling tools in Matlab-R19 are used. Optimization tools are traditional and are always play a significant role in achieving the maximum output with limited experiments [13]. Therefore, to gain maximum biodiesel yield from WCO, the response surface method (RSM) optimization technique was implemented with the help of statistical software Minitab®18. AI has entered every field of science, engineering, and technology; biodiesel production was not an exception. In recent times AI modeling was used [14] in biodiesel production due to its high accuracy and great prediction capabilities. Therefore, AI modeling was also carried out in this endeavour to compare the prediction capabilities.

2. Material and Methods

2.1. Material used

For biodiesel production, waste cooking oils from palm, sunflower, rice bran and groundnut oils are collected from different restaurants and households near the college campus (17.9942° N, 83.4150° E). Methanol (CH_3OH) and Sodium hydroxide (NaOH) of analytical grade were purchased from Merck industrial and lab chemicals, India. Inbuilt temperature and RPM controlled magnetic stirrer and different glass beakers of Borosil make are used for experimentation.

2.2. Transesterification process

The used cooking oils of palm, sunflower, rice bran and groundnut, which are about to discard are collected from different restaurants near the college campus. They are initially filtered with filter paper (Whatman-541) and washed with warm deionized water to remove unwanted and suspended impurities. After the filtration process, oils are mixed with a temperature-controlled magnetic stirrer until an even mixture appears. A sample of WCO from the mixture was tested for kinematic viscosity at room temperature and observed as 52 mm^2/s. Now the objective was to lower this high kinematic viscosity of the raw oil. For this purpose, the transesterification process [8] was followed.

Several factors influence the biodiesel yield during the transesterification process, namely the type of oil used for production, its acid number, viscosity, density, etc. Apart from the physical properties, other important parameters like type of catalyst (heterogeneous/homogeneous), alcohol used, reaction temperature and time show a significant change in biodiesel yield [15]. Therefore, these parameters are analyzed with the response surface method (RSM) optimization technique. L18 experiments are conducted based on Box-Behnken design in Minitab®18 statistical software. The experiments are performed by varying methanol to oil ratio (4:1, 8:1 & 12:1), NaOH catalyst (4, 6 & 8 grams), and reaction temperature (50°C, 55°C and 60°C) as shown in Table 1.

Table 1. Varying the methanol to oil ratio, catalyst, and reaction temperature

Property	Notation	L-I	L-II	L-III
Molar ratio	A	4:1	8:1	12:1
Catalyst used (grams)	B	4	6	8
Reaction temperature (°C)	C	50	55	60

Figure 1. Result of separataion: (a) Methyl ester and (b) Glycerol

During the transesterification process, the conversion of triglycerides to methyl ester will take place. The triglyceride reacts with methanol and NaOH to form biodiesel and glycerol. The heavy mass glycerol settles in the bottom, as shown in Figure 1, is removed and the methyl ester is separated and washed with warm deionized water until clear visibility of biodiesel and water and a neutral pH. The experimental biodiesel yield is calculated using Equation (1) and a total of L18 experiments are conducted randomly, as shown in the Table 2.

$$Yield = \frac{Weight\ of\ the\ biodiesel\ produced}{Weight\ of\ the\ raw\ oil} \times 100 \qquad (1)$$

Table 2. Experimental and RSM biodiesel yield

No	A	B	C	Exp Yield (%)	RSM Yield (%)
1	8	6	50	83.47	83.11
2	8	4	60	79.43	77.93
3	4	4	60	74.34	75.04
4	8	8	50	87.92	88.87
5	12	4	55	80.02	80.96
6	4	6	50	74.41	73.87
7	4	4	60	75.41	75.04
8	12	4	50	83.61	82.93
9	12	8	60	90.42	91.30
10	8	6	60	81.53	82.82
11	4	6	55	77.65	78.45
12	4	8	50	79.56	78.18
13	12	6	55	86.75	85.67
14	4	8	50	77.42	78.18
15	8	6	55	85.91	85.23
16	8	6	60	83.77	82.82
17	12	8	60	90.42	91.30
18	8	8	50	88.54	88.87

3. Result and Discussion

3.1. Biodiesel fuel property analysis

Biodiesel fuel properties are important to analyze before their real-time application in diesel engines. Therefore, WCO biodiesel from the transesterification process was tested for important fuel properties by following the international standards of ASTM and the reported results are compared with neat diesel and presented in Table 3. From the Table 3, it was observed that kinematic viscosity was recorded relatively higher than neat diesel fuel. This is due to the presence of highly unsaturated fatty acid composition within WCOBD. However, a drastic reduction in viscosity was observed from raw oil to methyl ester and approximately 91% reduction was witnessed post transesterification process. The obtained kinematic viscosity results from the Redwood viscometer were 4.29 mm^2/s, which can be used in compression ignition (CI) engine with or without blending.

The density measured with the help of a mass-comparator was observed as 880 kg/m^3, which is relatively more than the mineral diesel. Similarly, the flash and fire point measured with the silver Cleaveland (VT463) operator is also observed high for WCOBD. The heating value measured in the Bomb calorimeter (Toshniwal-CC01-M3) was reported as 38 MJ/kg, which is relatively near to the diesel value (42 MJ/kg). The cetane number was calculated using the fatty acid composition profiles and Equation (2) was used for this purpose, where the saponification and iodine values are represented by SV & IV. Similarly, the standard formulas used for various fuel properties are shown in Equation (3) and (4), Where A= 0.26 and B= 179. The obtained fuel properties of WCOBD are compared with the other researchers [16], [17] and observed within the limits of specified standards.

$$CN = 46.3 + \left(\frac{5458}{SV}\right) - (0.225 \times IV) \tag{2}$$

$$Kinematic\ viscosity = (A \times time) - \left(\frac{B}{time}\right) \tag{3}$$

$$Density = \frac{Mass\ of\ the\ Biodiesel}{The\ volume\ of\ the\ Biodiesel} \tag{4}$$

Table 3. WCOBD fuel properties

Fuel Property	Diesel	WCOBD	ASTM-D
Density (kg/m^3) at 18^{o}C	832	880	1298
Kinematic viscosity (mm^2/s) at 40^{o}C	2.9	4.29	445
Calorific value (MJ/kg)	42.32	38.08	240
Flash point (^{o}C)	58	168	1310
Cetane number	49	52	-
Fire point (^{o}C)	66	178	1310

3.2. Analysis of fatty acid composition

The fatty acid composition (FAC) analysis reveals saturated and unsaturated fatty acids in the biodiesel, and they always vary from oil to oil and depend on the oil crop cultivation location and climatic conditions. The main significance of measuring their levels will help estimate the important fuel properties and estimate the heterogeneous combustion. Several studies [18] reveal that measuring the fatty acids composition (FAC) in biodiesel can reveal the significant fuel properties, especially cetane number, based on the fatty acid structure, chain length and bonding. The presence of saturated and unsaturated may vary in the biodiesel. However, high NOx formation was evident in highly unsaturated FAC in the biodiesel as per the investigation from Talamala V et al. [19]. Therefore, in the present endeavour, biodiesel obtained from the transesterification process was tested for the presence of fatty acid levels with a gas chromatograph (Agilent-HP 6890 model). Table 4 reported the fatty acid composition presence in waste cooking oil biodiesel (WCBD) and approximately 56.33% unsaturated and 41.76% saturated were reported. Among the traced FAC, poly-unsaturated fatty acids are more dominant with maximum share (56.33%) and they are identified as Octadecadienoic acids was shown in Figure 2a. Similarly, Hexadecanoic acid, which is a saturated fatty acid with 31% share, was also shown in Figure 2b.

Table 4. Fatty acids in WCOBD

FAME	Wt (%)
Octanoic acid	4
Tetradecanoic acid	3.36
Hexadecanoic acid	31
9,12-Octadecadienoic acid	10.45
10,13-Octadecadienoic acid	45.88
Dodecanoic acid	1.7
Nonanedioic acid	1.7
Other	1.91
Saturated	41.76
Unsaturated	56.33

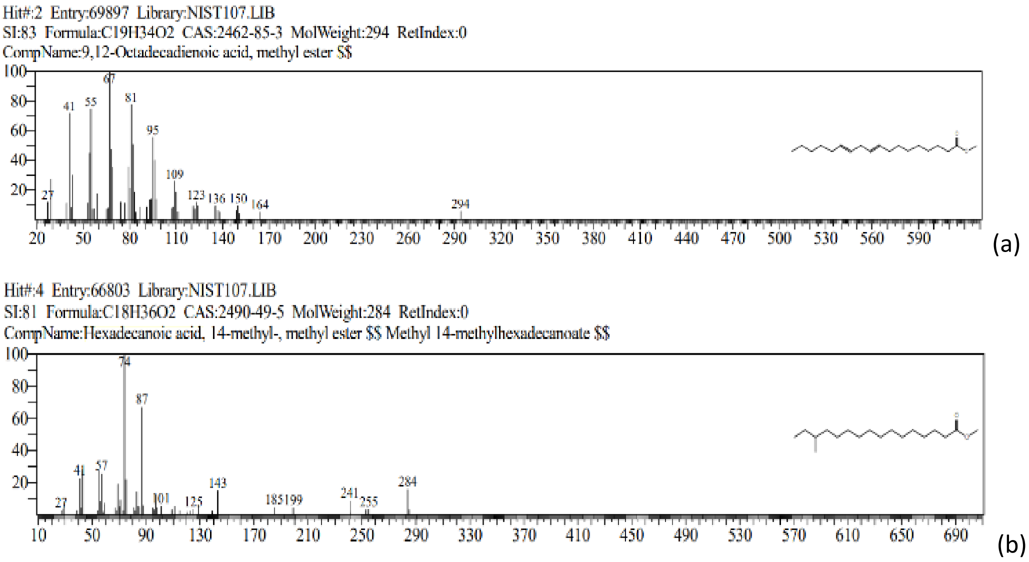

Figure 2. Chromatography profiles of WCOBD: (a) Octadecadienoic acids and (b) Hexadecanoic acid

3.3. ANOVA analysis

To determine the influence of process parameters in the biodiesel yield, an analysis of variance (ANOVA) test was carried out. The output results reveal the importance of each process parameter in achieving better yield by verifying with the F-test and P-test. Table 5 represents the detailed information of ANOVA analysis for the three process parameters. The maximum value in F-test and minimum value in P-test represent a significant contribution parameter on WCOBD yield and it was evident that the catalyst usage was recorded with a maximum F-value (65.23) and minimum P-value. The model's accuracy was represented by the coefficient of correlation (R2) which is 96.43%. This shows that the model was accurate in estimating biodiesel yield from WCO at 95% confidence levels. A maximum biodiesel yield of 91.30% was gained at a molar ratio of 12:1, catalyst usage of 8 grams, and reaction temperature of 60°C.

Table 5. ANOVA analysis for process parameters

Source	Df	Adj SS	Adj MS	F-Value	P-Value
Model	9	518.381	57.598	29.98	<0.0001
A	1	68.588	68.588	35.70	<0.0001
B	1	125.324	125.324	65.23	<0.0001
C	1	0.133	0.133	0.07	0.798
A2	1	36.64	36.64	19.07	0.001
B2	1	13.658	13.658	7.11	0.024
C2	1	11.367	11.367	5.92	0.035
A*B	1	7.223	7.223	3.76	0.081
A*C	1	18.929	18.929	9.85	0.011
B*C	1	12.081	12.081	6.29	0.031
Lack-of-Fit	5	10.649	2.130	1.24	0.408
R^2= 96.43%, R^2(Adj)= 93.21% and R^2(Pred) = 81.61%					

3.4. Interaction effect of process parameters

From the ANOVA analysis, it was evident that catalyst concentration shows a significant role in WCOBD yield compared to molar ratio and reaction temperature. The same can be witnessed graphically with the support of the Pareto chart, as shown in Figure 3. On the other hand, it was evident that reaction temperature has less significance in WCOBD production. The effect of process parameters in achieving better WCOBD yield can also be witnessed with the 2D contour plots as shown in figures (4-6). In these plots, the relation between any two process parameters which show a significant change in WCOBD yield can be examined.

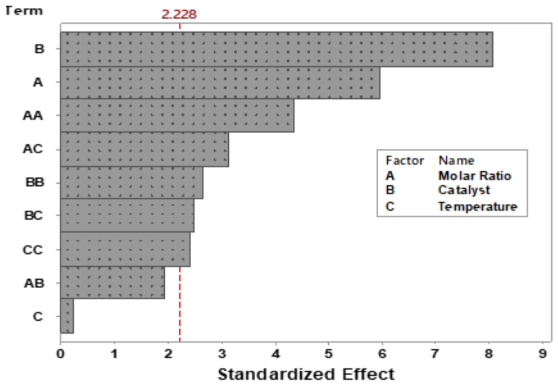

Figure 3. Pareto chart for WCOBD yield

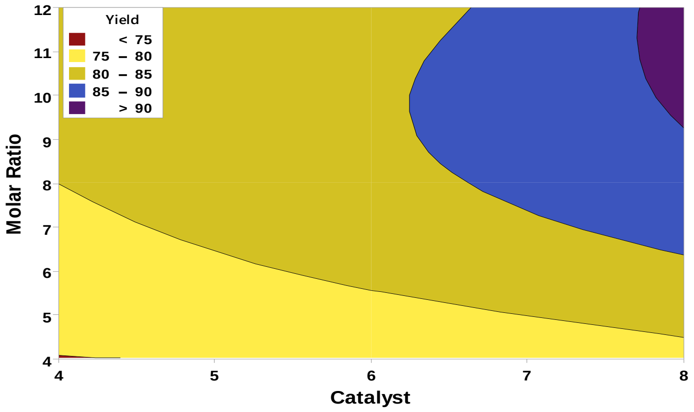

Figure 4. 2D Counter plot for molar ratio and catalys

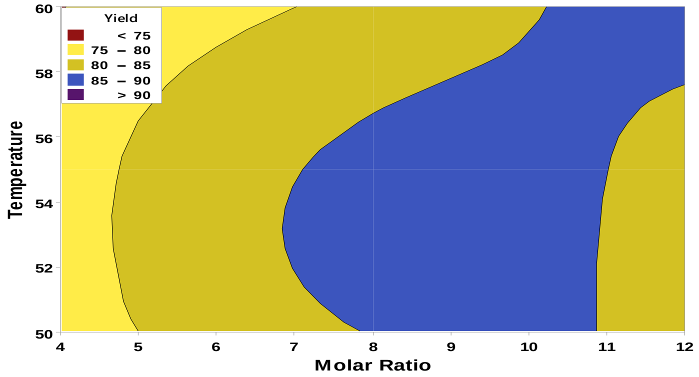

Figure 5. 2D Counter plot for molar ratio and temperature

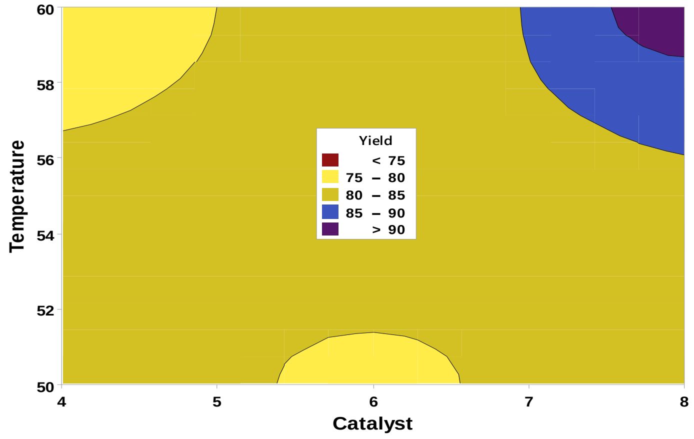

Figure 6. 2D Counter plot for catalyst and temperature

Then, the variation of catalyst and molar ratio shows a better yield at high molar ratios and catalyst usage as pesented in Figure 4. A maximum biodiesel yield of above 90% was obtained at a molar ratio of 12:1 and catalyst usage of 8 grams. The oils used for biodiesel production are of different fried cooking oils; hence their molecular bonding and chain lengths may completely be dispersed. Due to this, more amount of alcohol and catalyst are required to convert them into methyl esters.

Similarly, from the reaction temperature versus catalyst and molar ratio plots (Figure 5 and Figure 6), it was evident that better WCOBD biodiesel yield was achieved at maximum temperatures, as shown in Figure 6. Similar results are reported by Mohammad Anwar et al. [17] with the increase in temperature of about 55oC can achieve a biodiesel yield of about 95%. However, above 65°C there is a drastic fall in the biodiesel yield due to the low boiling point of alcohol. In general, alcohol possesses low boing points, and it was 64.7°C for methanol and 78.37°C for ethanol. Therefore, in this experimental investigation, methanol was used as alcohol to initiate the reaction with NaOH catalyst, and the maximum temperature maintained was 60°C to avoid the escape of methanol during the transesterification process.

3.5. ANN modeling

Artificial intelligence (AI) modeling was gaining more importance nowadays, especially in biofuel production, due to its performance popularity in solving complex non-linear problems [20]. The widely used tools in AI are genetic algorithms (GA), artificial neural networks (ANN), fuzzy logic, etc. These techniques can solve complex non-linear problems with high accuracy. Due to this extraordinary capability of AI tools, an attempt was made in this investigation by training the experimental results for maximum biodiesel yield prediction. For this purpose, a neural network fitting (NF) tool with a two-layer, feed-forward network was used. The neural network structure consists of an input layer, hidden layer, and an output layer. The L18 experimental results were trained to the input layer and the target biodiesel yield to the output layer. During the processing of data from input to hidden layer, 20 neurons are chosen by the trial-and-error method. For training the network, Levenberg-Marquardt was used, and the training was continued multiple times until low mean square error (MSE) was achieved. Figure 7 represents the correlation between the desired input and output by the regression (R) value which shows that the training (R=0.98542), validation (R=0.9493), testing (0.96521) and overall (R=0.97844) are closed to 1, which shows a good agreement between the actual and predicted results. A comparison graph (in

Figure 8) was drawn for experimental yield, RSM optimization yield and ANN modeling yield reveals that both the RSM and ANN results follow a similar experimental result. This shows that chosen models are highly accurate in estimating the maximum biodiesel yield from the set of eighteen experimental runs.

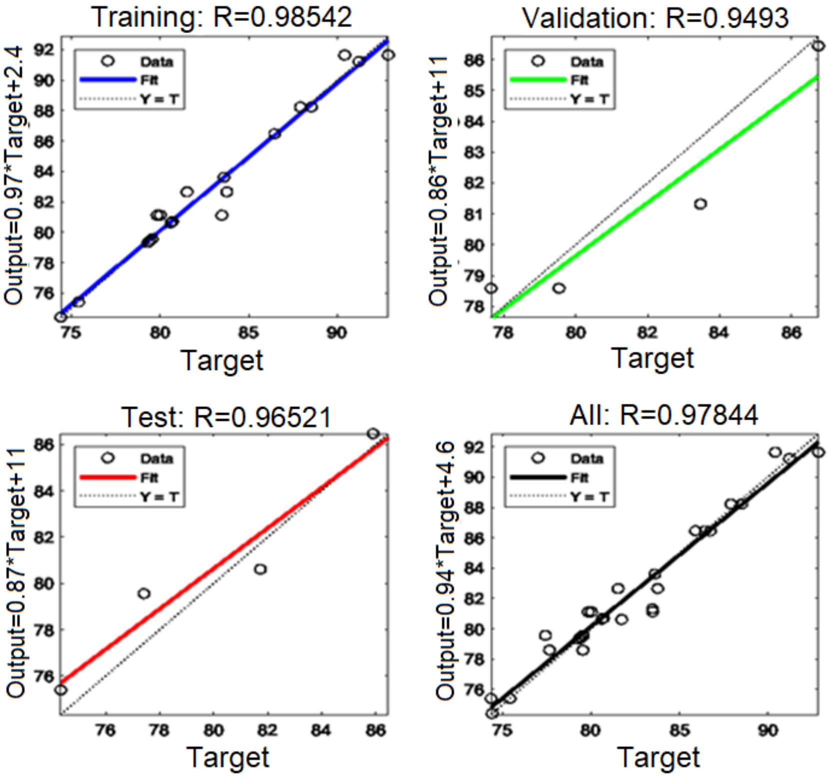

Figure 7. Regression plot for training, testing, and validation

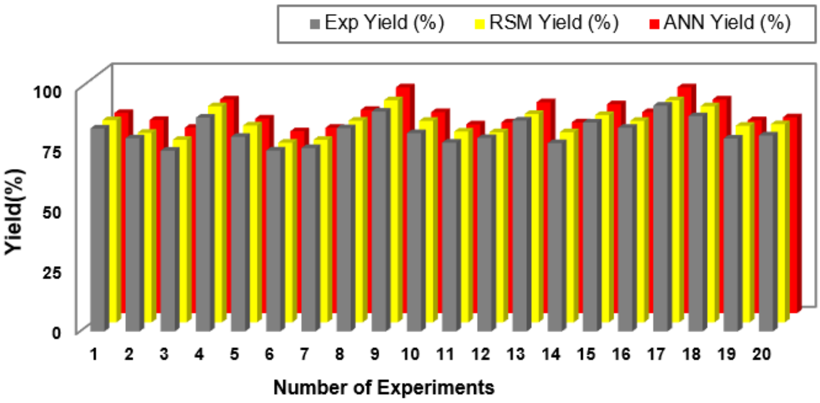

Figure 8. Comparison of WCOBD yield from chosen methods

4. Conclusion

Waste cooking oil biodiesel production from the transesterification process with the RSM optimization and ANN modeling has been experimentally examined in this study. The significant parameters which contribute to achieving maximum biodiesel yield were investigated individually and observed catalyst (NaOH) was most significant among the reaction temperature and molar ratio. As a result, a maximum biodiesel yield of 91.30% was gained at a molar ratio of 12:1, catalyst usage of 8 grams, and reaction temperature of 60°C in RSM optimization. Compared to RSM optimization, ANN modeling predicts the biodiesel yield more accurately (92.88%). Furthermore, the important fuel properties match with the requirements of international standards of ASTMD 6751. Both the techniques of RSM and ANN are accurate in the prediction of WCOBD yield based on the coefficient of correlation values (R and R^2). It was concluded that waste cooking oils are the best choice for biodiesel production, which can eliminate their disposal and with the latest optimization and modeling tools, better yield can be achieved.

Authors' Declaration

Authors' contributions and responsibilities - The authors made substantial contributions to the conception and design of the study. The authors took responsibility for data analysis, interpretation, and discussion of results. The authors read and approved the final manuscript.

Funding - No funding information from the authors.

Availability of data and materials - All data are available from the authors.

Competing interests - The authors declare no competing interest.

Additional information - No additional information from the authors.

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