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Optimisation of Biodiesel Production from Waste Frying Oil Via Response Surface Methodology (RSM) and Artificial Neural Network (ANN)

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ABSTRACT: This work was aimed at determining the suitability of using Waste Frying Oil (WFO) to produce biodiesel which is a very much needed energy product in our world today. This was achieved through a transesterification process in which time, methanol/oil ratio (MOR), catalyst weight and temperature were used to optimize the yield of biodiesel from waste frying oil. RSM and ANN have been used to carry out this process. RSM had an R^2 value of 0.967 and ANN had an R^2-value of 0.9000. This result does not suggest that ANN is less a predictive tool but this could be as a result of the number of neurons used or the number of training the ANN model was subjected to. The RSM model generated was optimized and the optimum yield obtained after 100 iterations was 88.694% at optimum conditions of MOR (7.720), Catalyst weight (1.393), Temperature (59.567), and Reaction time (92.707). The GC-MS results validates the biodiesel produced from WFO and shows that the biodiesel is typical of what is obtainable from similar research.

KEYWORDS: waste oil, optimization, biodiesel, ANN, RSM, transesterification

INTRODUCTION

The rise in the demand for energy with consequent depletion of fossil fuels as well as environmental degradation of the oil reserves to meet the energy needs of mankind has led to the search for alternative renewable sources of energy [1]. Unlike non-renewable fossil fuels, a class of renewable energy such as biofuels (biodiesel, biogas, bioethanol) has emerged. Because it is made from renewable resources like fresh or fried vegetable oils (both edible and waste), biodiesel is currently receiving more attention as an alternative fuel [2]. Biodiesel is a biodegradable diesel, a light yellow liquid having viscosity similar to fossil diesel [3]. It can be used alone in a diesel engine but can still be blended with certain additives, for better quality, prior to its use in the diesel engine. Biodiesel is a cleaner energy source than fossil fuel and mitigates global warming due to decreased emission of greenhouse gases such as carbon dioxide, sulphur (IV) oxide, carbon monoxide and hydrocarbons [4]. There are generally two main steps involved in the manufacture of biodiesel, the extraction of the oil and the conversion of same to biodiesel, done by employing various ways [5]. Large-scale manufacture of biodiesel today is majorly done by transesterification method using acid or base catalysts. Transesterification is a chemical reaction between triglycerides in the form of oil or fat and an alcohol, either methanol or ethanol in the presence of a base catalyst which may be potassium or sodium hydroxide to give biodiesel and glycerol [2]. High Free Fatty Acid content (greater than 1%w/w) reduces the quantity and quality of the biodiesel because soap is formed in the process making it uneasy to separate the biodiesel from glycerol. However, using acid as the catalyst in the esterification process or carrying out esterification with a base catalyst followed by transesterification processes has solved the problem of high FFA during the manufacturing process [6]. In another sense, it has been discovered that the use of household waste oils instead of the usual fresh oil is more cost-effective in the manufacture of biodiesel [7]. In addition, it is equally eco-friendly and renewable. Response Surface Methodology (RSM) is a very useful tool for improving the yield of biodiesel. It shows how the process responds to the independent variables when considered alone or when combined with the other variables [8]. This work is focused on optimization of the biodiesel production from Waste Frying Oil (WFO) using RSM. It involves the transesterification of the WFO to biodiesel using methanol as the base catalyst in the different variable combinations obtained using RSM. The design of the experiment was utilized in order to assess and understand the effect of each variable on the biodiesel yield, calculate the optimum values for these operating variables and in turn, obtain the values of the biodiesel yield.

MATERIALS AND METHOD

Pretreatment of WFO

WFO is filtered into a beaker using a sieve/mesh screen to remove particulate matter and food residue. In a bid to reduce the amount of the FFA (via Acid-catalysed esterification), a known amount of oil is poured into the beaker and then heated to 60°C on the hot plate with a magnetic stirrer. Methanol was added in a 3:1 methanol-oil ratio before adding 1wt% acid catalyst, similar to the procedure employed in [9]. The biodiesel was washed with hot water, separated using a separating funnel then dried in an oven at 105°C.

Transesterification Reaction.

The approach adopted in this study is similar to the one used by [10], but with slight modifications. The transesterification reaction of WFO and Methanol was performed using a varied table of values of 30 runs generated using the RSM software for design and optimisation of the parameters and factors. A known weight of the pretreated WFO was measured into a round-bottom flask and placed on a hot plate with a magnetic stirrer, and the reaction mixture was heated to a certain temperature as obtained from the central composite design (CCD). Also, a known weight of NaOH pellet was dissolved in a known volume of methanol and was quickly transferred into the pretreated WFO in the round-bottom flask. The reaction was monitored according to the design variables. At the end of the reaction, the reaction mixture was transferred to a separating funnel for glycerol and Waste Frying Oil Biodiesel (WFOB) separation. This process was repeated till the 30th run, following the respective parameters generated from a four-factor-four-level central composite design (CCD) [10].

Washing, Separation and Drying.

The biodiesel was placed in a separating funnel for the glycerol to settle before separation, and then the biodiesel was washed with warm water to remove any remnant glycerol or soap formed before drying in the oven at 105°C.

Yield of the biodiesel

The biodiesel yield or percentage conversion of the biodiesel can be calculated using the formula given below:

$$Yield\% = \frac{weight\ of\ biodiesel\ produced}{weight\ of\ oil\ used} \times 100 \qquad (1)$$

Characterization of WFO sample

Fourier transform infrared (FTIR) technique

The chemical structure of the WFO sample and any change in it such as the presence of foreign materials etc. is identified and confirmed by this technique. This method is based upon the excitation of the bonds present in the structure of the sample by absorbing IR radiations of corresponding frequencies. The FTIR spectra provide a 'fingerprint' of the sample. The scanning is usually performed in the frequency range of 500–4000 cm^{-1} and at a resolution of 4 cm^{-1}. By comparing the bands of two samples e.g., sample before or after the adsorption of pollutant [11, 12].

Gas-Chromatography analysis.

The chemical composition of the studied samples was performed using a Gas chromatograph with a direct capillary column 30 m x 0.25 mm x 0.25 µm film thickness. The column oven temperature was initially held at 80 °C and then increased by 5°C /min to 200°C with holding time of 2 min then increased to 280 with a hold time of 10 C/min. The GC injector temperature was set up to 270 °C. Helium was used as a carrier gas at a constant flow rate of 1 ml/min. Electro Ionization (EI) mass spectra were collected at 70 eV ionization voltages over the range of m/z 40–550 in full scan mode. The ion source and transfer line temperatures were set at 200 and 250°C respectively [13].

Response surface methodology

Four input factors were chosen for the study which include; reaction time (min), reaction temperature (°C), catalyst concentration (%wt/wt) and methanol/oil ratio which are denoted by A, B, C and D with the output factor as yield (%). The upper and lower limits were set for the factors as seen in Table 1 which shows a range of 4 factors and used in the experiment. These factors with their selected range were used to predict the response which is the yield of biodiesel and to determine the model that best fits the data. This was achieved using the *Design Expert Version 10* by entering the number of factors as 4 and the response as 1 on the Central Composite Design (CCD) data entry page. The numerical factors were set to 5 levels which are plus and minus alpha (axial points), plus and minus 1 (factorial points) and the centre point. The number of centre points was 6 while non-centre points was 24 bringing the total number of runs to 30 and the alpha value was set at 2 to ensure that the axial points are not negative or in decimal.

The Eq. (2) represents various effects of process variables on response

$$Y = \beta_0 + \beta_1 A + \beta_2 B + \beta_3 C + \beta_4 D + \beta_{12} AB + \beta_{13} AC + \beta_{14} AD + \beta_{23} BC + \beta_{24} BD + \beta_{34} CD + \beta_{11} A^2 + \beta_{22} B^2 + \beta_{33} C^2 + \beta_{44} D^2 \quad (2)$$

where Y is the response variable, which denotes the yield of biodiesel, β_0 the intercept term, β_1, β_2, β_3 and β_4 are linear coefficients, β_{12}, β_{13}, β_{14}, β_{23}, β_{24} and β_{34} are interactive coefficients, β_{11}, β_{22}, β_{33} and β_{44} are quadratic coefficients and A, B, C and D are independent factors.[14]

Table 1: Parameters and their levels for CCD for Transesterification of WFO to biodiesel

Process parameters	Units	Coded Factor Levels				
		-α	-1	0	+1	+α
Methanol: oil A	-	3.5	5	6.5	8	9.5
Catalyst conc. B	wt/wt%	0.4	0.8	1.2	1.6	2
Temperature. C	^{0}C	45	50	55	60	65
Reaction time. D	Min	25	50	75	100	125

Artificial neural networks (ANN)

The ANN application called Neural Network Fitting of MATLAB (R2015a) software was used. The ANN technique was used in this study to optimize the model generated from the RSM analysis by using a network of neurons just like the information processing system of the human brain equipped with a high correlation profile through the log sigmoid function [15]. It involves three stages; the training, testing and validation stages (Figure a). The total samples considered were 30 in which the training stage had 70% and the testing and validation had 15% each. The ANN architecture has the input (4), hidden layers (9), and output layer (1). The hidden layers were varied from 9 to 15 neurons with 9 neurons giving the best prediction for the experiment. The log sigmoid function was used since it has a high correlation profile The performance of the ANN process can be expressed in terms of the MSE and correlation coefficients (R) [16].

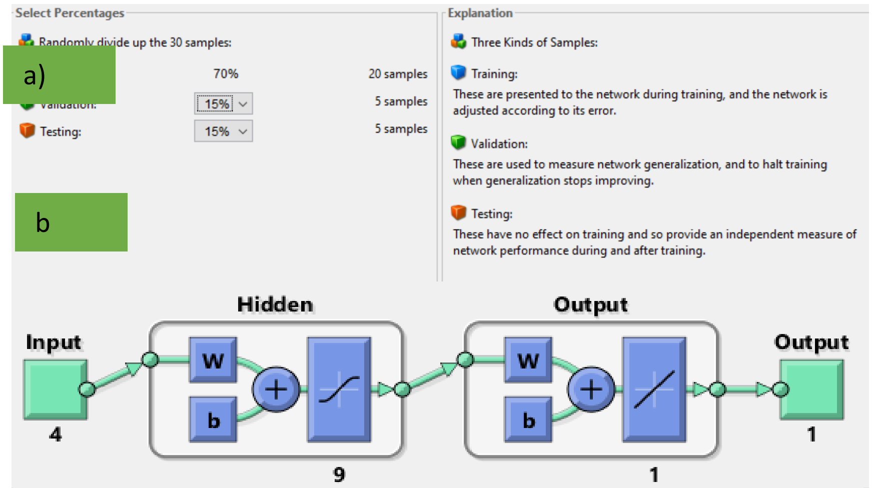

Figure: a) Data entry page of the Neural Network Fitting
b) Diagram showing the architecture of ANN.

RESULTS AND DISCUSSION

Biodiesel yield in the transesterification process using RSM

According to the Central composite design (CCD), a total of 30 runs were done for the transesterification process (as shown in Table 2) using the pretreated WFO, with a minimum yield of 54.62% on run 8 and a maximum yield of 88.56% on run 5 (Table 2). The response variable (the yield of biodiesel) was collected for all the runs together with the predicted values. Based on the 30 runs, a model was developed by RSM for the response variable (Y) as a function of four selected independent factors within the range selected as shown in Table 1. The model generated (Eq. 3) is quadratic and includes terms for interactions among the factors.

$$Y = -187.77472 + 5.67018A + 91.11792B + 5.4926C + 0.26688D + 1.832AB + 0.012464AC - 0.02489AD - 0.30728BC - 0.092295BD + 6.06e^{-03}CD - 0.36354\,A^2 - -27.85204B^2 - 0.045219\,C^2 - 1.59e^{-03}D^2$$

(3)

Where Y is the yield of biodiesel, A is the methanol to oil ratio, B is catalyst concentration, C is temperature and D is the reaction time respectively. These represent the actual factors considered and not the coded factors.

Table 2: Design of experiment, experimental, Predicted RSM and ANN values

Run	Factor1 A: MOR	Factor 2 B: Catalyst Conc. %wt/wt	Factor 3 C: Reaction Temperature °C	Factor 4 D: Reaction Time Minutes	Response 1 Yield %	Predicted rsm Value	Response ANN
1	6.5	1.2	55	125	82.73	82.84	82.09
2	6.5	1.2	55	75	86.42	82.36	81.35
3	8	0.8	50	50	68.04	66.27	67.66
4	5	1.2	50	100	75.21	75.55	73.13
5	8	1.6	60	100	88.56	87.98	87.66
6	8	0.8	60	100	78.24	78.97	82.18
7	6.5	1.2	55	75	82.45	82.36	81.35
8	6.5	0.4	55	75	54.62	54.65	59.57
9	3.5	1.2	55	75	73.31	73.21	74.72
10	6.5	1.2	65	75	85.31	84.69	81.15
11	5	0.8	60	50	68.21	67.28	70.24
12	5	0.8	60	100	77.67	76.97	73.10
13	6.5	1.2	55	25	74.54	73.93	76.21
14	6.5	1.2	55	75	81.2	82.36	81.35
15	9.5	1.2	55	75	85.37	84.97	83.96
16	6.5	1.2	55	75	82.95	82.36	81.35
17	5	1.6	60	50	74.29	75.59	74.92
18	5	1.6	60	100	80.53	81.58	76.19
19	8	2	60	50	79.3	78.71	78.28
20	6.5	1.2	55	75	81.53	82.36	81.35
21	5	0.8	50	50	59.1	60.90	58.48
22	8	1.6	50	50	79.5	81.43	79.43
23	5	1.6	50	100	75.32	74.63	74.81
24	6.5	1.2	45	75	70.87	70.98	73.82
25	6.5	1.2	55	75	79.02	82.36	81.35
26	8	0.8	50	100	69.26	69.19	67.13
27	6.5	1.2	55	75	83.49	82.36	81.35
28	8	0.8	60	50	71.57	73.02	84.23
29	8	1.6	50	100	80.45	80.65	79.96
30	5	1.6	50	50	73.12	71.66	73.81

ANOVA studies

The analysis of variance (ANOVA) is one of the methods for checking the accuracy of the model developed. The results of the ANOVA of RSM are given in Table 3 below. The Model F-value of 34.25 implies the model is significant. There is only a 0.01% chance that an F-value this large could occur due to noise. The Lack of Fit F-value of 0.45 implies the Lack of Fit is not significant relative to the pure error. There is an 86.15% chance that a Lack of Fit F-value this large could occur due to noise. It also shows that the lack of fit for the model is not significant implying that the dependent variable was well predicted by the independent variables. This was further explained by the "Pred R-Squared" of 0.8851 which is in reasonable agreement with the "Adj R-Squared" of 0.9414 i.e. the difference is less than 0.2, however, a difference greater than this limit shows that the lack of fit is significant and the model does not fit (Table 4)

Table 3: ANOVA

Source	**Sum of Squares**	**df**	**Mean Square**	**F Value**	**p-value Prob > F**	
Model	1680.93	14	120.07	34.25	< 0.0001	significant

A-MOR	200.24	1	200.24	57.12	< 0.0001	
B-Catalyst Conc	434.06	1	434.06	123.81	< 0.0001	
C-Temperature	271.84	1	271.84	77.54	< 0.0001	
D-Reaction Time	114.90	1	114.90	32.77	< 0.0001	
AB	19.01	1	19.01	5.42	0.0343	
AC	0.14	1	0.14	0.039	0.8466	
AD	13.54	1	13.54	3.86	0.0682	
BC	5.94	1	5.94	1.69	0.2126	
BD	13.40	1	13.40	3.82	0.0695	
CD	8.92	1	8.92	2.55	0.1315	
A^2	18.48	1	18.48	5.27	0.0365	
B^2	449.95	1	449.95	128.34	< 0.0001	
C^2	35.30	1	35.30	10.07	0.0063	
D^2	27.30	1	27.30	7.79	0.0137	
Residual	52.59	15	3.51			
Lack of Fit	21.32	9	2.37	0.45	0.8615	Not significant
Pure Error	31.27	6	5.21			
Cor Total	1733.52	29				

Table 4: Model Summary Statistics

Source	**Std Dev**	R-Squared	Adjusted R-Squared	Predicted R-Squared	PRESS	
Linear	5.22	0.6075	0.5447	0.4146	1014.88	
2FI	5.57	0.6600	0.4811	-0.2454	2158.98	
Quadratic	1.87	0.9697	0.9414	0.8851	199.25	Suggested
Cubic	2.28	0.9820	0.9128			Aliased

Effect of Reaction Parameters on Biodiesel Yield.

The 3D response surface plots in Fig 5(a–c) investigated the interaction effect of important process variable combinations on biodiesel yield. The interactive effect of process factors namely; temperature, reaction time, catalyst concentration, and methanol to oil ratio shown in Fig 5(a–c). The diagrams agree with the equation that gives the yield of biodiesel which has already been shown as quadratic. The concave slope confirms that the optimum value for biodiesel production is a maximum point and had the reverse been the case, then we would have had a minimum point. This shape depicts effective interaction among the independent variables, for instance, the catalyst concentration (B) and the reaction time (D) played a huge role in ensuring that the side reaction of soap formation does not occur by allowing sufficient time and adequate catalyst concentration. In addition, the catalyst concentration increased the energy sites which led to the production of more alkyl esters and ultimately the production of biodiesel [17]. The graduation from minimum to maximum and back again brings several reasons to mind; the activation energy prevailed during the initial stages of the reaction, however, the catalyst was on hand to ensure the reaction reached its climax within the time allowed. In the same vein, the methanol-oil ratio (MOR) (A) and temperature (C) interaction led to the achievement of the maximum yield of 88.56% at an MOR of 8%w/w and a temperature of 60^{o}C. For the fact that the transesterification reaction is reversible, high MOR will help to ensure that all the oil present is converted into valuable product at a slightly high temperature. Also, the temperature will aid in pushing the reaction beyond the energy barrier by ensuring frequent collision of the reactant molecules leading to the efficient transformation of the reactants into the final product. The graphical results suggest that all the biodiesel increased up to a maximum of 88.56% as all the independent variables increased.

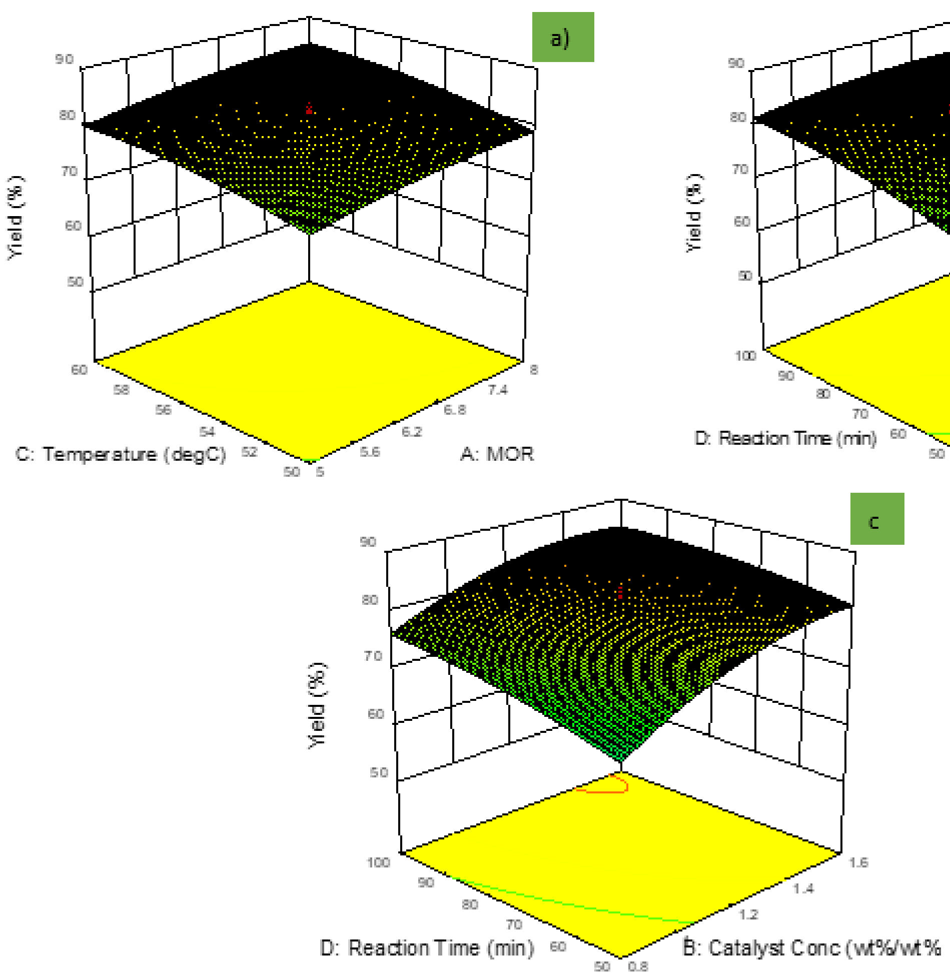

Figure 5(a-c): 3D interaction of the various factors that affect the yield of biodiesel

Optimization studies for RSM

This was achieved using the optimization option of the *Design Expert Version 10* the numerical sub-option was used to achieve the optimization process. The goal for the yield which should be maximized was set at "maximize" while the four independent factors were kept in range according to the entry data on the application entry page. The optimization process was run and about 100 iterations were generated but the optimum value of the yield was 88.694% while the optimal conditions obtained were MOR (7.720), Catalyst weight (1.393), Temperature (59.567), and Reaction time (92.707).

Modelling of Biodiesel yield using ANN

ANN is a valuable tool which employs data-driven approach for simulating multifaceted or nonlinear relationships which are demanding to represent using physical models [18]. In this study, several training algorithms were established with hidden layers and neurons, Lavenberg- Marquardt backpropagation algorithm was to find performance functions based on RMSE and MSE as it minimizes a linear combination of squared errors and weights [8]. Figure. 6 shows the ANN architecture for the investigation of biodiesel production from WFO comprised of an input layer with four neurons (methanol to oil ratio, catalyst concentration, temperature and reaction time), 9 neurons of hidden layer and a single neuron an output layer (biodiesel yield).

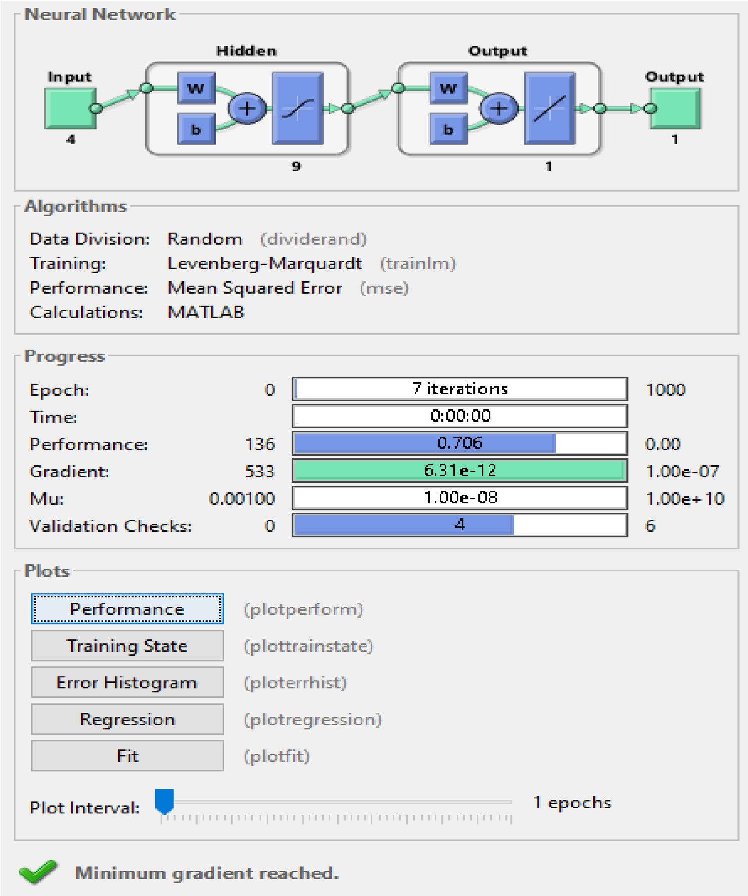

Fig 6: Data Output pane on ANN

The output regression coefficient values for the training, validation, and testing with corresponding R values of 0.96105, 0.93527 and 0.97492 are shown in Figure. 7 The R-value obtained from these experiments indicated good agreement between both the experimental and estimated values. The good fit of the model was confirmed by the low values of MSE [16]. The values of MSE reported a low error % between actual and ANN predicted. Beyond the limit of 9 neurons, the problem of overfitting resulted leading to varied results in the yield and the regression. Therefore, the study used variation from 1 to 9 Neurons under 7 iterations to avoid the divergence of the mean square error.

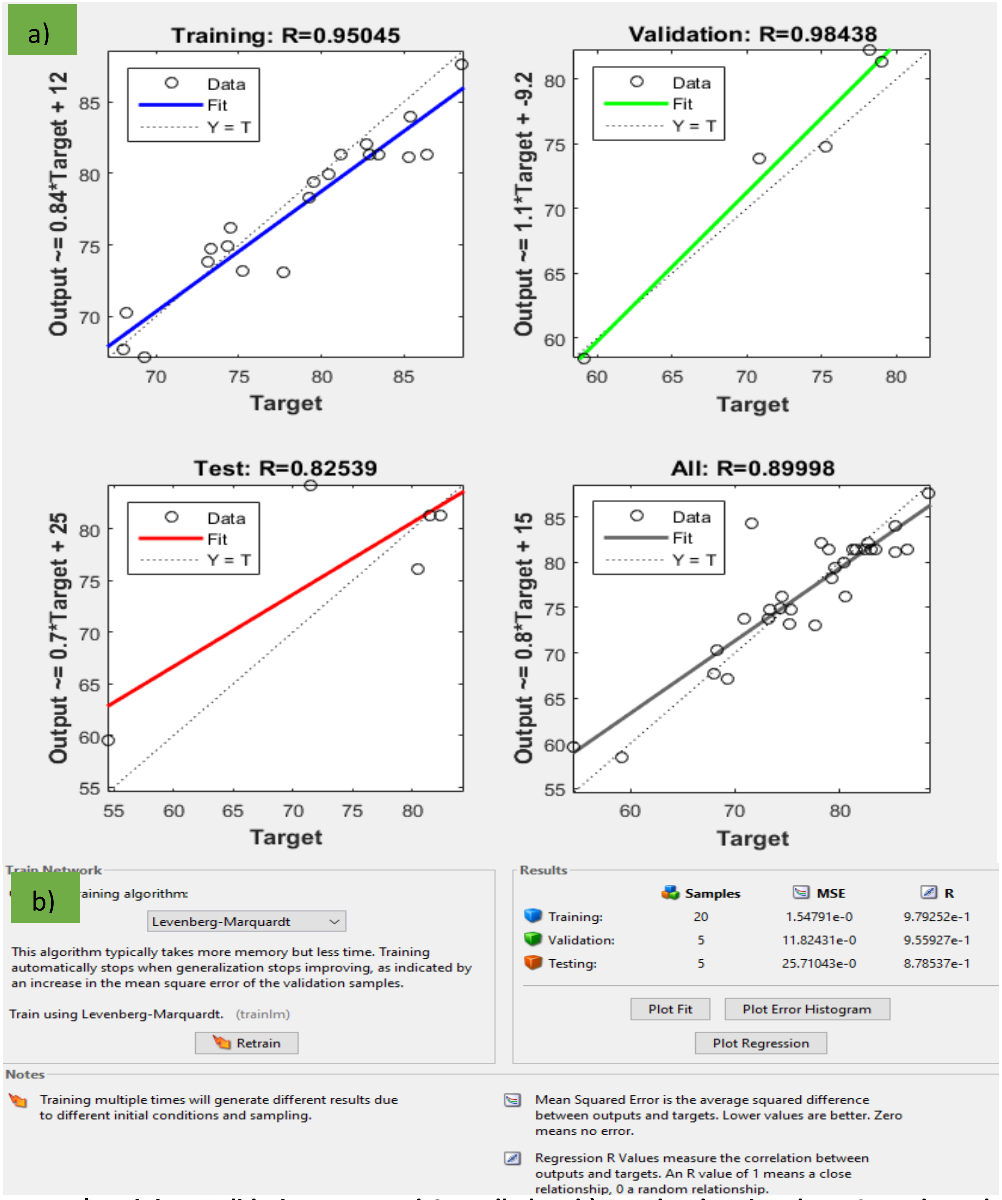

Figure 7: a) Training, Validation, Test and Overall plots b) Results showing the MSE and R-values

Comparative Analysis of ANN and RSM

Among the optimization models, the ANN and RSM model results show high accuracy [19] and were used to investigate the predicted values and verify their actual values. The correlation coefficient R^2 values expressed the model's accuracy and fitness. The R-value and R^2 values for ANN and RSM were 0.9000 and 0.9697, respectively. The error was greater in ANN which shows that the values predicted by RSM were closer to the laboratory values than were those of ANN [17]. Table 2 shows the predicted values of both models and the actual response values experiment. It could be detected that there is much less deviation of points between actual response and predicted values of ANN. The ANN model illustrated that the obtained results were highly robust and superior for prediction and complex problem-solving solving, especially biodiesel production. [20]. The bar chart plot below (Fig. 8) compares the yield of biodiesel as obtained from RSM, ANN and RSM predicted. This plot shows that the yield of biodiesel as determined was very close as the height of the bar for each of them is almost of the same length. However, the height of the bar for ANN showed a significant deviation from those of RSM and RSM predicted for about 8 samples with only a little error for the rest of the samples implying that ANN is a better optimization tool than RSM.

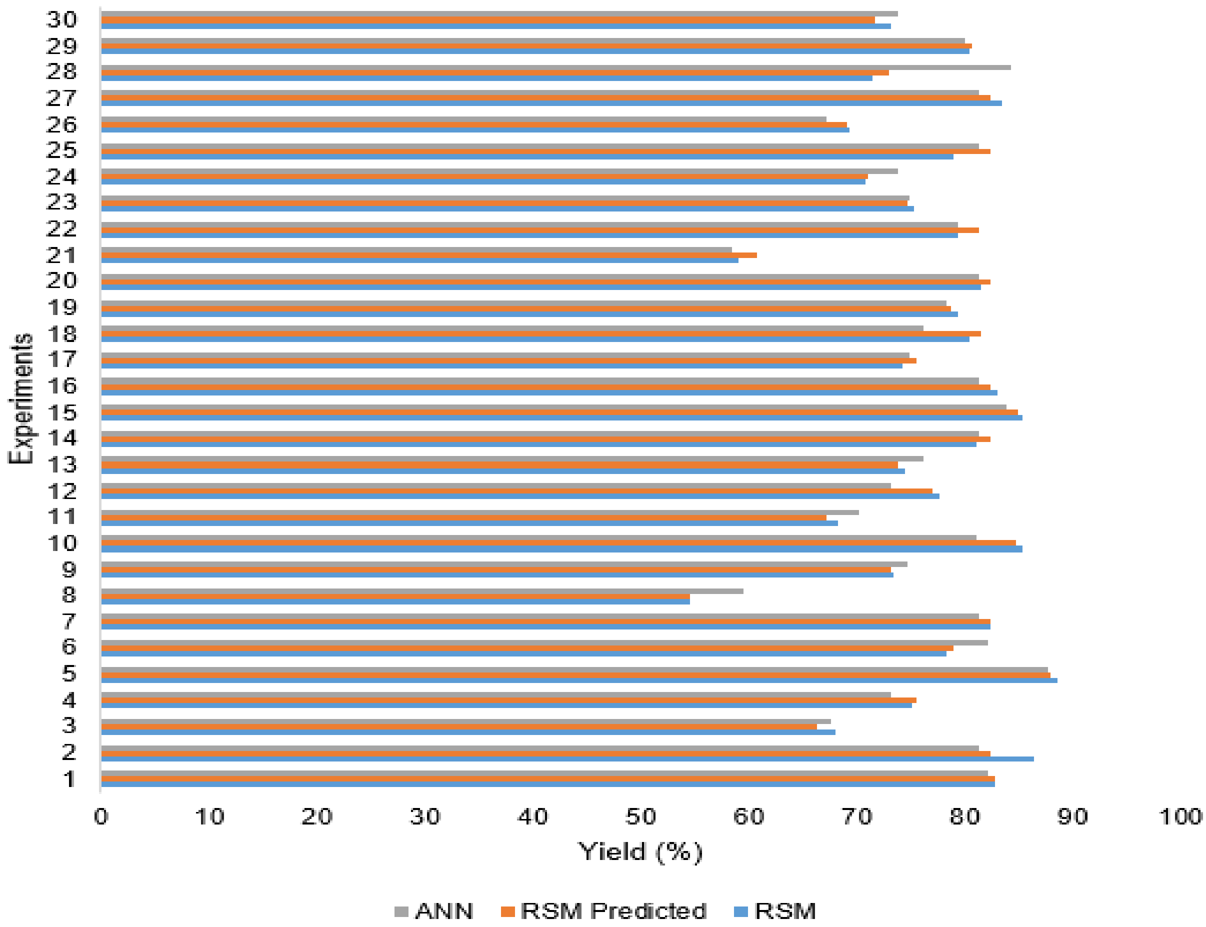

Fig 8: Experiments plotted against against ANN, RSM predicted and RSM

Validation Analysis (GC-MS)

The fatty acid chromatogram showing the different components present in WFOB is shown in Figure 9. The peaks were recorded which showed different fatty acid methyl esters and few non fatty acid components present. The major fatty acid component present in WFOB is oleic acid followed by linolenic acid and palmitic acid. Other organic compounds detected by the GC–MS in SASOME include Alcohol, monoglyceride, diglyceride and triglyceride. With these results, WFOB is expected to have low thermal efficiency and viscosity, and emit lower HC, CO and less smoke than other higher saturated diesel fuels.

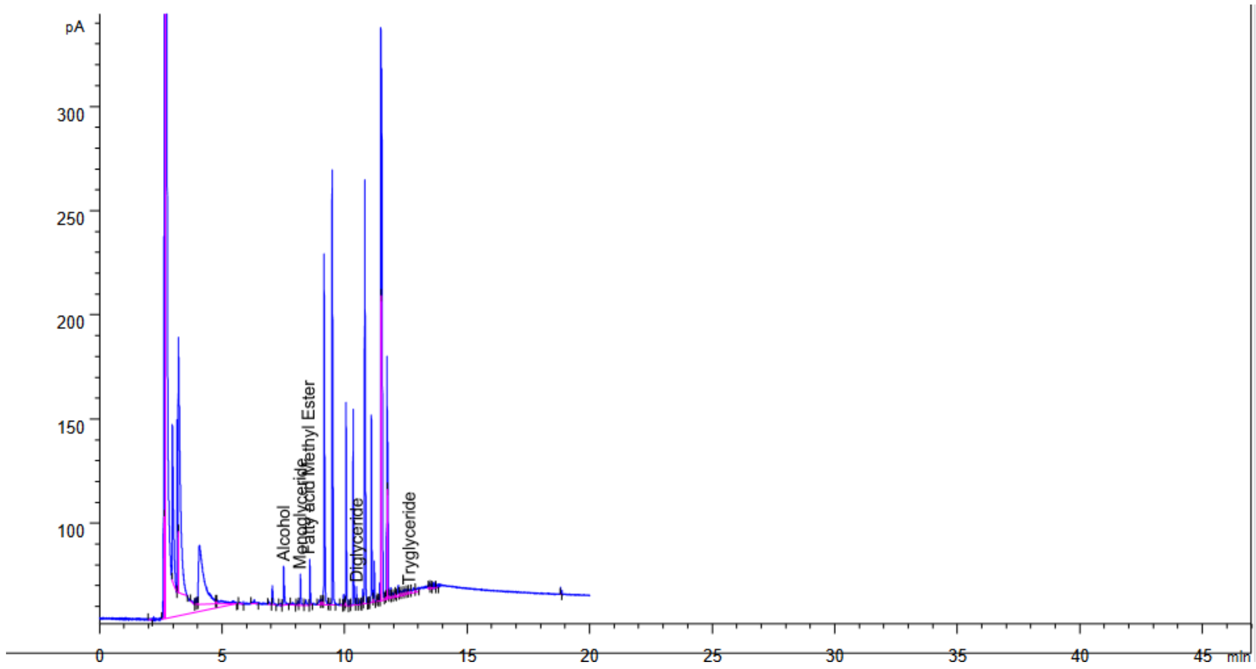

Figure 9: GC-MS chromatogram showing the components of Biodiesel.

CONCLUSION

The study aimed to optimise biodiesel production from WFO using RSM and ANN which is aimed to solve two problems at a go namely; the reduction of biowaste dumps in our environment and filling the gap created by shortage of supply by fossil fuel. The accuracy of the model generated on RSM was analysed using ANOVA which showed a better prediction R^2 of 0.9697 as compared with the ANN which gave an R-value of 0.9000. This doesn't necessarily mean that the ANN is a less accurate model but that the result could also be due to the number of neurons used and the amount of training done. Also, the bar chart used for the yield from RSM, RSM predicted and ANN confirms that RSM is a much better tool for modelling of biodiesel yield. The maximum yield according to the RSM results is 88.56% while that of ANN is 87.66% which agrees with the R-value and the R^2 values of the models, respectively. The optimization of the process was achieved with RSM where the yield was 88.694% while the optimal conditions obtained were MOR (7.720), Catalyst weight (1.393), Temperature (59.567), and Reaction time (92.707). These discoveries have significant implications for the biodiesel production sector, offering potential enhancements in operational efficiency, cost-effectiveness, and environmental sustainability by employing waste cooking oil as a viable raw material. Additionally, the study guides future research, emphasizing the need for refining process parameters, investigating alternative catalysts, evaluating scalability, and conducting techno-economic feasibility analyses to support widespread biodiesel production.

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