

Prediction of Biodiesel Yield from Moringa Seed Oil Using Factorial Design Model and Artificial Neural Network Model

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ABSTRACT

In this study, oil was extracted from Moringa seed using mechanical and solvent methods. To transesterify the oil into biodiesel, factorial design of experiment of 2⁴ was used to obtain different combination factors at different level of reaction temperature, catalyst amount, reaction time and alcohol to oil ratio, giving rise to 48 experimental runs. The oil sample was transesterified in 48 experimental runs, in each case the biodiesel yield was recorded in percentage. The biodiesel was then characterized according to ASTM test protocol. Factorial design model was developed using Design Expert 7.0, the model generated R of 0.987 and Mean Square Error (MSE) of 5.0453 and was used to predict and optimize biodiesel yield. Artificial Neural Network (ANN) model from MATLAB R2016a was developed using 4 input variables and 30 runs, the remaining 18 runs were tested with the ANN model to predict and compare the biodiesel yield with the experimental biodiesel yield, the model generated R value of 0.99687 and MSE of 3.50804. It was found that solvent method yielded more oil than mechanical method, the biodiesel has good thermo-physical property, optimum biodiesel yield of 91.45 % was obtained at 5:1 alcohol/ oil molar ratio, 18.89 wt% catalyst amounts, 45 minutes reaction time and at 45°C reaction temperature. The experimental validation yielded 88.33 % biodiesel. The ANN model adequately predicted the remaining 18 runs with R² value of 0.99649 and MSE of 4.914243. Both models proved adequate enough to predict biodiesel yield but ANN model proved more adequate.

Keywords: Artificial neural network model Biodiesel, Biodiesel, Factorial design model, Optimization, Prediction

I. INTRODUCTION

The growing concern over fast depletion of the worlds' crude oil reserve, fuel price volatility and stringent pollution regulation has placed the future of fossil fuels under serious threat; this has generated research interest in production of supplementary and surrogate fuels. Biodiesels have been found to be promising substitute fuels and supplementary fuels in internal combustion engines (1,2). Biodiesel is a preferable fuel option because it is renewable, biodegradable and non-toxic, with high flash point and reduces green house gas emission (3). Biodiesel can be produced from plant oils or animal fat via transesterification of triglyceride using short chain alcohol mostly methanol and ethanol as solvents in the presence of catalyst (4), however, Verma, et al. (5) reported that the choice of the solvent that gives the highest yield depends on the feedstock used thus it is necessary to identify the best solvent for each feedstock.

In relation to petro diesel, the cost of production and commercialization of biodiesel is relatively higher at pilot scale (6) thus the idea of increasing the yield with relatively lower input has become a worthwhile endeavour to minimize cost. Several researchers adopted factorial design in optimizing biodiesel yield with maximum success, this approach has become widely used because it allows the influence of a factor to be predicted at different interaction level of other factors, that is, it allows three, four or more input variables to be studied simultaneously (7).

Production of biodiesel from plant oil should be in such a way that it does not pose threat to food security, the oil derived from Moringa seed is classified as non-edible oil [8,9,10] and its oil has been identified as a potential biodiesel resources in some countries (8,9,11). Abdulkareem

(11) optimized oil yield of Moringa seed using factorial design, he employed n-hexane and bio-ethanol as solvents, this work attempts to find the optimal yield of biodiesel from moringa seed oil using methanol as solvent.

In scientific studies, larger sample size helps researchers to efficiently avoid error from testing and effectively determine the average values of their data (12), thus during experimental design, many experimental runs may be desirable as this gives larger sample size to the data. In solvent extraction of biodiesel from oils, catalyst such as KOH, NaOH and CH₃ONa are cheap and available, however, consequent higher energy consumption may cause increase in capital cost for equipment and safety concern (13,14) this limitation may increase with increasing data size in addition to making it tedious, thus it is necessary to develop and test a model that can efficiently predict biodiesel yield using previous experimental data.

II. EXPERIMENTAL METHOD

Factorial Experimental Design and Plan of the Experiment

Factorial design formulates the interaction of the dependent and independent variables prior to experiment. If the combinations of three independent variables (factors, k) are to be investigated at two different levels, the factorial design will be in the form of 2^k giving rise to 12

experimental runs. The lower level of these factors are represented by minus (-1) and the upper level by plus (+1), that is, all the variables are normalized to fall between -1 and +1. After experimental runs have been carried out, the obtained response variables at different combination and interaction level of the input variables are used to generate regression model that describe the relationship between them and this model is used to optimize the independent variable. The input variables may be constrained within the experimental range, minimized or maximized depending on what it is intended for [15]. The regression model equation will take the form of

$$y = \beta_0 + \sum_{k=1}^2 \beta_i x_i + \sum_{k=1}^2 \beta_{ii} x_i^2 + \sum_{k \neq j}^2 \beta_{ij} x_i x_j \dots$$

i=1, 2, 3...n; k=1, 2, 3...n [1]
y is the dependent variable (biodiesel yield), x_i and x_j are the independent variables, β₀ and β_i, β_{ii} and β_{ij} are the intercepts and coefficients of linear and quadratic terms respectively.

Design Expert 7.0 was used to carry out the factorial design, 2⁴ factorial design of four (4) factors namely alcohol to oil ratio, catalyst amount, reaction temperature and reaction time was used. The factors were varied at two levels; low and high level to determine the interactive factors for highest biodiesel yield. The varied parameters are presented in Table 1.

Table 1. Variation of parameters for 2⁴ factorial design

S/N	Name	Lower Limit	Upper Limit	Lower Weight	Upper Weight	Desirability
1	Alcohol/oil ratio	5:1	6:1	1	1	3
2	Catalyst amount	10	20	1	1	3
3	ReactionTemperature	50	60	1	1	3
4	ReactionTime	45	60	1	1	5
5	Biodiesel yield	75	98	1	1	5

Extraction and transesterification of Moringa seed oil

Two methods of oil extractions were used to determine the oil yield; mechanical and solvent extraction approaches were employed.

Oil extraction by mechanical method

The seeds were extracted from the pod and dried under open sun, the weight of the seeds were continually measured until no further weight change was observed. 1000g of moringa seeds were grinded into powder, hot water at approximately 105°C was poured on the powder and thoroughly mixed with a homogenizer to form paste, the paste was then tied in a filter material and

pressed gradually using hydraulic press to drain the oil, the drained oil was collected in a beaker. The oil collected was then heated in an open container to allow water present in the oil to evaporate; the oil was weighed continuously until no further change in weight was observed. The final volume of the oil which represents the percent oil yield was measured and recorded.

Oil extraction by solvent method

The method of Shivani et al. (16) was used. 1000g of Moringa seeds were grinded into powder and the fat was removed using soxhlet apparatus, n-hexane was utilized as the solvent. The process was allowed for 6 hours and the

solvent was removed via drying at oven temperature of 50°C utilizing vacuum evaporation.

The quantity of the oil extracted was presented as percentage of oil extracted from moringa seed.



Plate 1: Photograph of moringa and the extracted oil

Transesterification of moringa seed oil

A quantity of Moringa seed oil was poured into a flat bottom conical flask, a water bath apparatus connected to a thermostat was used to preheat the oil to a temperature of 50 °C. 10g of KOH and 5g of methanol were mixed together and dissolved; the mixture was then poured into the oil and heated on a hot plate magnetic stirrer to a temperature of 60 °C according to the experimental design. The reaction was allowed a period of 50 minutes and the process was stopped. The mixture was allowed to stand for 14 hours to stratify and form separate layer of glycerol and biodiesel, the mixture were then separated using burette.

60 mL of distil water was poured on the biodiesel and stirred gently to remove impurities that may be present in the diesel, the mixture was then allowed to stay for 14 hrs to form two phases of the liquid, the mixture was separated using burette, the process was repeated twice and the decanted biodiesel was then heated at 90 °C to dry

it, the weight was continually measured till no weight change was observed. The process was replicated for 47 more runs, in each case the biodiesel yield was calculated and recorded using equation 2.

$$\text{Biodiesel yield, \%} = \frac{V_b}{V_s} \times 100$$

[2]

Where;

V_b =Volume of biodiesel recover

(g)

V_s = Volume of the oil used (cm³)

Fuel characterization

The biodiesel samples were characterized according to standard test protocols to determine its moisture content, specific gravity, kinematic viscosity, acid value, cloud point, flash point, pour point and cetane number, the test method are presented in Table 2.

Table 2. Test methods adopted for characterizing the biodiesel

S/N	Fuel property	Test method
1	Moisture content	ASTMD6304
2	Specific gravity	ASTM D1298
3	Kinematic viscosity	ASTM D-2983
4	Acid value	ASTM D664
5	Cloud point	ASTM D-2500
6	Flash point	ASTM D-92
7	Pour point	ASTM D-92
8	Cetane number	ASTM 613

III. RESULTS AND DISCUSSION

Oil yield from moringa seed

The oil yield obtained from mechanical and solvent method of oil extraction is presented in Table 3.

Table 3. Oil yield of moringa seed

S/N	Oil extraction method	Oil yield %
1	Mechanical extraction	18.00
2	Solvent extraction	40.00

The oil yield obtained from the two extraction methods shows that the solvent extraction approach yielded 55% more oil than the mechanical method. The results obtained are in agreement with that of Anwar and Bhanger (17) and Abiola and Ato (18).

Properties of the transesterified moringa oil (biodiesel)

The results of preliminary study of the properties of moringa seed oil derived biodiesel carried out according to ASTM standard are presented in Table 4.

Table 4. Properties of moringa seed oil derived biodiesel

S/N	Properties	Moringamethyl ester	Limits of ASTM Test protocol
1	Moisture content %	0.18	min 0.05
2	Specific gravity	0.87	0.85-0.90
3	Kinematic viscosity (mm ² /s) at 40 °C	3.23	1.9-6.0
4	Acid value (mg KOH/g oil)	0.28	min. 0.80
5	Cloud point °C	4.0	-15 to 5
6	Flash point °C	250	min 100 – 170
7	Pour point °C	4.5	-35 to -15
8	Cetane number	57.90	min 47

As it could be seen, the moisture content of the biodiesel falls above the minimum recommended value, this could be attributed to the fact that biodiesels are hygroscopic in nature, and fatty acid methyl esters absorb humidity during storage (19,20), thus the biodiesel of moringa seed oil is hydrophilic as from other feedstock, such elevated moisture content could present potential microbial growth media in transport equipment and fuel tank. A further study may be required to determine the level of moisture absorption of the biodiesel under different storage medium and through a longer storage period. The specific gravity of the fuel falls within the bracket of suitable biodiesel fuel standard; this infers that the fuel contains favourable energy content which can consequently increase engine power (8,21). The kinematic viscosity of the fuel falls within the low-normal limit, this implies that the fuel will atomize into fine spray without resistance and burn efficiently in engines (22). The acid value of the fuel falls below the minimum benchmark, this

demonstrate that the fuel has no tendency of forming solid deposits in fuel injectors and will rarely present corrosion effect in fuel tank due to acidity (23). Both flash point and pour point also falls within the bracket, this infers that the fuel is suitable for use in temperate regions with no challenges of gelling. The Cetane value was also found to fall above the minimum value, this indicates that the fuel will not present any challenge during ignition.

Regression Model Evaluation

The forty eight (48) experimental runs carried out for each combinations of independent variables obtained from the design to obtain the biodiesel yield, the experimental data was regressed to obtain models that explains the relationship or the effect of all the independent variables on the yield of biodiesel, the summary of the design and yield of the biodiesel is presented in Table 5.

Table 5. 2⁴ experimental design and experimental biodiesel yield

Runs	Alcohol/oil	Catalyst amount (wt%)	Reaction temperature(°C)	Reaction time (min)	Biodiesel yield (%)
1.00	5.00	10.00	60.00	50.00	88.00
2.00	6.00	10.00	60.00	50.00	91.00

3.00	6.00	10.00	60.00	50.00	93.00
4.00	5.00	20.00	45.00	50.00	91.00
5.00	5.00	20.00	45.00	60.00	73.00
6.00	5.00	10.00	60.00	60.00	41.00
7.00	6.00	10.00	45.00	60.00	62.00
8.00	6.00	20.00	60.00	50.00	81.00
9.00	6.00	20.00	45.00	50.00	86.00
10.00	5.00	20.00	60.00	50.00	81.00
11.00	6.00	10.00	60.00	60.00	29.00
12.00	6.00	20.00	60.00	50.00	77.00
13.00	5.00	10.00	45.00	60.00	72.00
14.00	6.00	10.00	45.00	50.00	90.00
15.00	5.00	20.00	60.00	60.00	18.00
16.00	5.00	20.00	60.00	50.00	83.00
17.00	6.00	10.00	60.00	50.00	92.00
18.00	5.00	10.00	60.00	50.00	85.00
19.00	6.00	20.00	45.00	60.00	53.00
20.00	5.00	20.00	45.00	50.00	90.00
21.00	5.00	10.00	45.00	50.00	86.00
22.00	6.00	20.00	45.00	50.00	85.00
23.00	6.00	20.00	60.00	60.00	15.00
24.00	5.00	20.00	60.00	50.00	82.00
25.00	6.00	10.00	60.00	60.00	28.00
26.00	5.00	10.00	45.00	60.00	75.00
27.00	6.00	10.00	45.00	50.00	86.00
28.00	5.00	10.00	45.00	50.00	90.00
29.00	5.00	20.00	45.00	60.00	70.00
30.00	5.00	10.00	45.00	50.00	89.0
31.00	6.00	10.00	45.00	50.00	89.00
32.00	6.00	20.00	60.00	60.00	13.00
33.00	5.00	10.00	60.00	50.00	86.00
34.00	6.00	20.00	60.00	60.00	14.00
35.00	6.00	10.00	45.00	60.00	62.00
36.00	6.00	20.00	60.00	50.00	78.00
37.00	5.00	20.00	60.00	60.00	20.00
38.00	5.00	10.00	60.00	60.00	44.00
39.00	6.00	20.00	45.00	60.00	50.00
40.00	6.00	10.00	60.00	60.00	29.00
41.00	5.00	20.00	45.00	50.00	89.00
42.00	6.00	20.00	45.00	50.00	84.00
43.00	6.00	20.00	45.00	60.00	56.00
44.00	5.00	10.00	60.00	60.00	42.00
45.00	5.00	10.00	45.00	60.00	73.00
46.00	5.00	20.00	45.00	60.00	75.00
47.00	6.00	10.00	45.00	60.00	63.00
48.00	5.00	20.00	60.00	60.00	19.00

Table 6. Summary of Statistical Analysis of the Effect of the Independent Variables on Biodiesel Yield

Parameter	Value	Parameter	Sum of square	VIF	Adequate precision	Mean	Standard deviation
R ²	0.9974	Regression	31764	-	83.386	2117.6	1.62
F-stat	806.70			1.000	-	-	-
Sig. of F-stat	0.0001	-	-	1.000	-	-	-

The statistical analysis explains the fitness of the model to predict biodiesel yield after transesterification. From Table 6, it could be seen that the model generated R^2 value of 0.974, which demonstrates that 97.4 % changes in the biodiesel yield can be explained by changes in the independent variables. Larger F-statistics of 806.70 and sum of square of regression of 31764 indicate that the model accounts for most of the changes in the biodiesel yield. It can be inferred that the model significantly predicts the independent variable, since the significance level is 0.0001 which is below 0.05 at 95% level of confidence. Adequate precision or signal to noise ratio of 83.486 is far above the benchmark of 4 which indicates very minimal noise in the model; the variation inflation factor (VIF) of 1 is below the benchmark of 10, this negates the possibility of collinearity or multi collinearity among the independent variables thus the model can be declared adequate enough to estimate the relationship between the independent variables and the biodiesel yield.

The model generated is a factorial model of 4FI order at 95% level of confidence; the model

equation was modified to eliminate all independent variables that are insignificant in terms of actual factor and is given as;

$$\begin{aligned} \text{Biodieselyield} \\ (Y) = & 2114.66667 - 432.33333 * A - 194.53333 * B - 4 \\ & 8.28889 * C - 38.40000 * D \\ & + 10.53333 * A * C + 8.16667 * A * D + 4.20444 * B * C + 3. \\ & 86000 * C * D + 0.90667 * C * D + 0.014222 * A * B * C * D \\ [3] \\ (7) \end{aligned}$$

While the equation in terms of coded factor is given as;

$$\begin{aligned} y = & 66.00 - 3.25 * A - 4.21 * B - 10.63 * C - 20.33 * D - 0. \\ & 87 * A * B + 1.21 * A * C - 2.92 * A * 2.75 * B * C - 1.79 * B * \\ & D - 9.04 * C * D + 0.83 * A * B * C + 0.87 * A * B * D + 0.29 * \\ & A * C * D - 0.75 * B * C * D + 1.33 * A * B * C * D \\ [4] \end{aligned}$$

Where A-Alcohol/molar ratio; B-catalyst amount; C-Reaction time; D-Reaction temperature

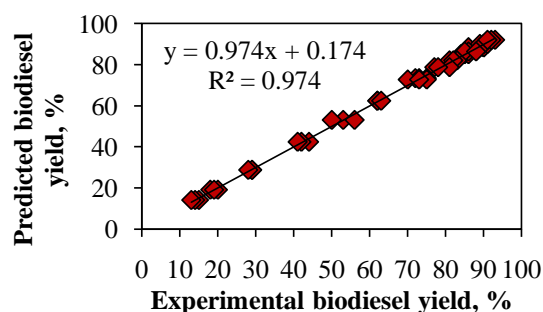


Figure 1. Correlation between predicted and experimental biodiesel yield

Optimization and Validation of Biodiesel Yield

Having substantiated the adequacy of the regression model, the generated model was utilized by to maximize biodiesel yield, the goal of the optimization is to keep alcohol/ oil molar ratio, catalyst amount and reaction time within the experimental domain and then minimize reaction temperature to save energy cost. The algorithm randomly chooses starting values for all the independent variables for 30 cycles per optimization at simplex fraction of 0.1. Maximum biodiesel yield of 91.45 % was obtained at 5:1 alcohol/ oil molar ratio, 18.89 wt% catalyst amount, 45 minutes reaction time and 45°C reaction temperature. The optimized value was validated experimentally using the optimized values of the

independent variables; the experimental biodiesel yield obtained was 88.33 %. The optimized and the validated biodiesel yield show close agreement, with deviation of 3.12%, according to Ali and Hanna (24), residual of less than 10% is the minimum allowable error, thus our finding is valid.

Artificial Neural Network Modeling

Thirty (30) of the forty eight (48) experimental run's input variables (alcohol/oil ratio, catalyst amount, reaction time and reaction temperature) and output variable (biodiesel yield) (Table 4) were used to train and develop artificial neural network (ANN) model using MATLAB R2016a. Back propagation algorithm base on Levenberg-Marquardt algorithm was used to train

the network, the data was divided into 70, 15 and 15% for training validation and testing respectively, the weight and biases were randomly initialized to adjust their value during each training session. Mean Square Error (MSE) and Correlation Coefficient (R) were used to evaluate the performance of the model. After training for different network structures and re-initializing their weight and biases severally, a model with 4-10-1-1 (4 inputs, 10 hidden layers, 1 output layer and 1 output) topography was adopted based on highest R value and lowest MSE value. The hidden layer and output layer transfer functions used were Tansig and purelin respectively, the general neural network model formulation is given as [25].

$$Y_n = f_o * \left\{ b_o + \sum_{k=1}^n [w_k * f_h (b_{hk} + \sum_{i=1}^n w_{ik} x_{ni})] \right\}$$

i=1, 2, 3...n; k=1, 2, 3...n [5]

- Y_n = Output variable
- f_o = Output transfer function
- b_o = Output layer bias
- w_k = Weight connecting the kth hidden neuron and the single output neuron
- f_h = Hidden layer transfer function
- b_{hk} = Bias of the kth hidden neuron
- w_{ik} = ith input variable and kth hidden neuron connecting weight
- x_{ni} = input variable

The two transfer functions used in this model can be expressed as:

$$f_h = \text{Tansig}(n) = \frac{2}{1 + e^{-2n}} - 1$$

$$f_o = \text{Purelin} = n$$

The matrices of input weight and biases into and out of each layer is presented below

Layer 1 (Hidden layers)

$$b1(b_{hk}) = \begin{bmatrix} -2.4580808600624771 \\ 1.9310957077650097 \\ 1.3567975490561501 \\ -0.81750521301421564 \\ 0.61545737771965103 \\ -0.35789198212317752 \\ 0.88925676531620479 \\ 1.6091950339810746 \\ -1.9718313823098437 \\ -2.3995193618568629 \end{bmatrix}$$

IW1_1 (w_{ik}) =	$\begin{bmatrix} 0.73936817427474522 & -1.6884552102784856 & -1.6617059385556212 & 0.40438824895482056 \\ -0.37770502468832307 & -1.7856535474355322 & -1.2487074530154971 & 1.1637084942423246 \\ -0.23970923816821879 & -0.43412942237497421 & -1.8026407663559081 & 1.64306344450516 \\ 0.46440347785288866 & 0.84419996070169245 & -1.4499568727610679 & -1.821167265252946 \\ -1.3307363728052066 & 0.38449526042052901 & -0.885753459301001021 & 1.812535922555379 \\ -1.59578515861000650 & 0.52947213686352568 & -1.2495545419151559 & 1.3906870074923376 \\ 1.2286939280990334 & -1.6478086659112512 & -1.2911980969762069 & 0.42318404574534879 \\ 1.03657782381849480 & 0.47730791985626314 & -1.6700711799986958 & -1.125904643573306 \\ -1.305135197436373 & -1.2335251110378858 & 1.2747450634102468 & -1.1014039958961632 \\ -1.7080023074319963 & -1.5960218685447867 & 0.91161220938951792 & 0.64420908684164713 \end{bmatrix}$
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Layer 2 (Output layer)

$$b2(b_o) = [0.22348652113986431]$$

LW2_1 (w_k) =	$\begin{bmatrix} 0.031112361626798316 & 0.38123147573317367 & 0.088214252978513583 & 0.042178086956626962 \\ -0.54288368105334506 & 0.3904173612417543 \\ -0.20277581165459135 & 0.45336735796515021 & 0.17356754817338127 & 0.107341703725083531 \end{bmatrix}$
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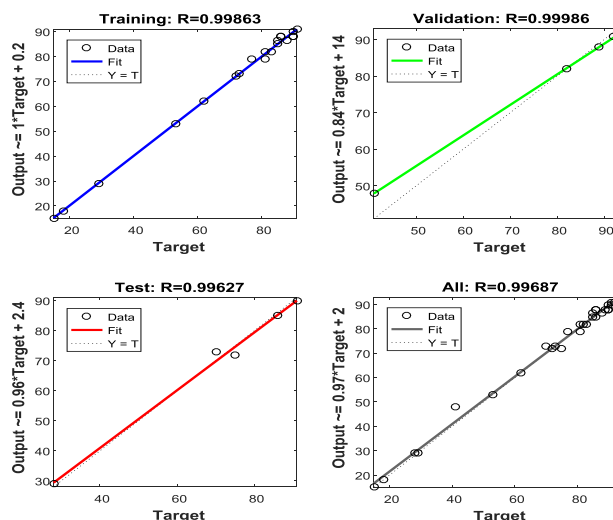


Figure 2. Correlation between ANN predicted biodiesel yield and experimental biodiesel yield for 30 runs

ANN Model validation

The generated model (Network) was saved as MATLAB file, the remaining eighteen (18) input experimental data were then used as input variables to the generated ANN model to test its ability to predict biodiesel yield beyond 30 experimental data. After the model has been used

to simulate the biodiesel yield, the ANN model predicted biodiesel yield for the 18 remaining runs were then compared with the experimental biodiesel yield. R value of 0.99649 and MSE of 4.914243 were obtained as the performance function of the ANN model's predicted biodiesel yield.

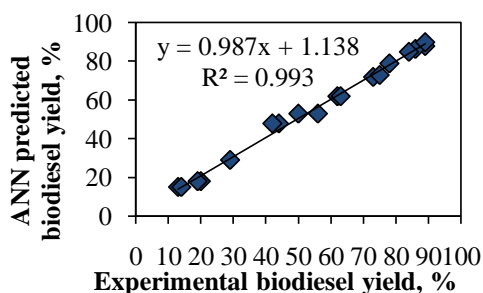


Figure 3. Correlation between ANN predicted biodiesel yield and experimental biodiesel yield for 18 runs

On the overall performance of the model as shown in Figure 4, the model satisfactorily predicts biodiesel yield with high level of accuracy. Comparatively, the ANN model generated higher R value of 0.99649 and lowest MSE of 3.50804 when

compared to R of 0.983 and MSE of 5.0453 of factorial design model, thus it can be adjudged that ANN model outperforms factorial design model, however both models showed excellent capability of predicting biodiesel yield from Moringa seed oil.

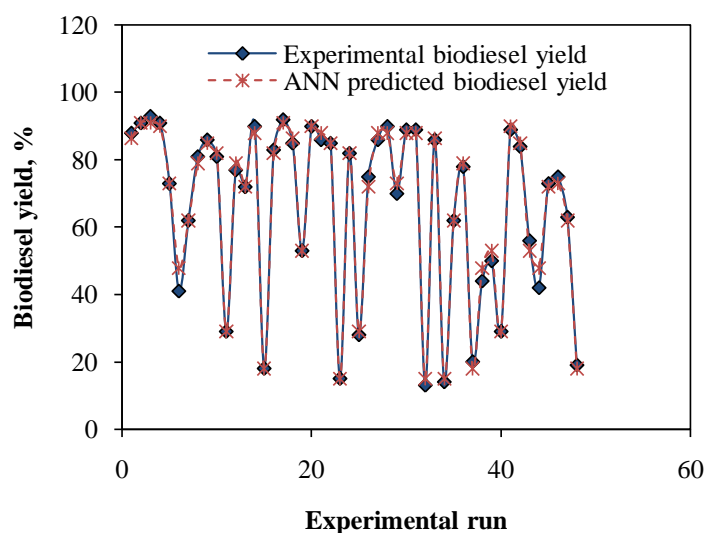


Figure 4. ANN model prediction performance for 48 runs

IV. CONCLUSION

Extraction, transesterification of moringa seed oil and characterization, optimization and prediction of biodiesel yield has been carried out; the work draws the following conclusions

1. Solvent extraction method yielded more moringa seed oil than mechanical method.
2. The biodiesel from moringa seed oil shows good properties suitable for use as diesel engine fuel.
3. The maximum biodiesel yield of 91.45 % was obtained at 5:1 alcohol/ oil molar ratio, 18.89 wt% catalyst amounts, 45 minutes reaction time and at 45°C reaction temperature.
4. Artificial neural network model satisfactorily predicts biodiesel yield with higher accuracy than factorial design model

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