

Application of artificial neural networks and response surface methodology for dye removal by a novel biosorbent

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ABSTRACT

Toxic dyes found in industrial effluent must be treated before being disposed of due to their harmful impact on human health and aquatic life. Thus, *Musa acuminata* (banana leaves) was employed in the role of a biosorbent in this work to get rid of methylene blue derived from a synthetic solution. The effects of five process parameters such as temperature, pH, biosorbent dosage, initial methylene blue concentration, using a central composite design, the percentage of dye clearance was investigated (CCD). The response was modelled using a quadratic model based on the CCD. The analysis of variance revealed the most influential element on experimental design response. Temperature of 44.3°C, pH of 7.1, biosorbent dose of 0.3 g, starting methylene blue concentration of 48.4 mg/L, and 84.26% dye removal were the best conditions for *M. acuminata* (banana leaf powder). At these ideal conditions, the experimental percentage of biosorption was 76.93. The surface area of 40 m²/g was provided by *M. acuminata* and the pore volume of *M. acuminata* was observed as 0.265 cm³/g and the scanning electron microscopy images are also showed before and after biosorption. The link between the estimated results of the developed artificial neural networks (ANN) model and the experimental results defined the success of ANN modeling. As a result, the study's experimental results were found to be quite close to the model's predicted outcomes.

Keywords: *Musa acuminata*; Central composite design; Methylene blue; Artificial neural network

1. Introduction

The application of dyes like textiles, paper, rubber, plastic, food, leather and cosmetics are just a few of the sectors that use it, can result in a considerable volume of colored watery waste. Industrial effluents lose 10% to 15% of their weight during processing and manufacturing processes [1,2], with a global production of about 0.7 million tons/y. Colored wastewater dumped into natural water bodies is a major source of pollution, causing a slew of issues. The fact that there are relatively minimum amounts of dye in

the water (as little as 1 ppm for some dyes) has an impact on aesthetics, dissolved oxygen solubility, transparency, and sunlight permeability, all of which have an impact on aquatic life and the food chain [3]. The health of humans and aquatic organisms may be compromised by certain dyes and/or breakdown products of dyes (e.g., aromatic amines). Commercially available adsorbents are very expensive, even though they are available in a variety of forms. Biosorbents with new and inexpensive properties are therefore necessary to remove dyes from wastewater. Recently, plant biomass has been made from a variety of

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industry by-products and agricultural waste are examples of low-cost feedstock precursors methylene blue is removed from water using this method, as like as other colors derived from water [4–6].

Methyl blue molecular formula (MB) is $C_{16}H_{18}N_3SCl$. When dissolved in water, it looks like a dark green powder, that is solid and odorless. Basic dye, MB, is widely used as an antibiotic, as well as to color papers, hair colorants, stock of paper coating, dye, printing tannin, cotton, and leather dyeing. Due to the various features of textile effluent, methods of physical, chemical, and biological treatment for color removal include flocculation, coagulation, adsorption on activated carbon, electrochemical treatment, membrane processing and aerobic or anaerobic biodegradation. We have extensively reviewed the advantages and disadvantages of each technique [7–9].

Industrial effluents can be effectively and economically removed from dyes using biosorption. As part of the process, a liquid substance (soluble dye) is carried on solid surface, extremely porous (adsorbent) material and bonds physically or chemically [10]. Adsorption is the most efficient, cost-effective, simple, and easy to operate because of its low toxicity as a wastewater treatment method. Furthermore, the adsorbent can be selected from a wide range of materials, which is an advantage of this approach. In batch or dynamic conditions, natural materials (peat, chitin, wood, coal, chitosan, biomass and other clays industrial wastes (red mud, ash, dye hydroxide sludge, furnace blast slag, lignin, bark, sawdust, maize cob, stalks from sunflower, husk from rice, olive stones, seashell, shell of hazelnut etc.) [11].

By response surface methodology (RSM) and artificial neural network (ANN) techniques, no study has been done on optimizing the banana leaves powder for methylene blue elimination. A basic RSM design known as a central composite design (CCD) enables the optimization of with a small number of experiments, you can find the most effective parameters, in addition the examination of parameter interactions. The success of ANN modeling was determined by the relationship between the created ANN model's expected results and the experimental results. Temperature, pH, biosorbent dose, and initial dye concentration all have an impact on the process were examined to achieve a high methylene blue percentage removal from synthetic solution.

2. Materials and methods

2.1. Biosorbent

Banana leaves were harvested near Salalah in the Sultanate of Oman's Dhofar Region. To dispose of dirt particles, the leaves were cleaned numerous times by distilled water. The dried leaves were subsequently ground to a powder size of 75–212 μm using a domestic grinder and used as a biosorbent for methylene blue biosorption without any pretreatment.

2.2. Chemical

The appropriate salt was dissolved in distilled water to make methylene blue. 0.1 N HCl and NaOH were used to modify the pH of the solutions.

All the tests were carried out five times, with the average results being given. Blank trials were also carried out to guarantee that no biosorption occurred on the walls of the apparatus.

2.3. Apparatus

The concentration of methylene blue was measured using a UV spectrophotometer at 665 nm. A pH meter from SYSTRONICS and an electronic balance from Shimadzu were used to determine the pH of the adsorbate and the weight of the adsorbent.

2.4. Batch biosorption experiments

The pH was adjusted to 5.1 with hydrochloric acid and sodium hydroxide, and experimental methylene blue solutions of appropriate concentrations were made from the stock solution using the requisite dilutions.

By agitating 30 mL of varying concentrations of metal solution with 0.1 g of adsorbent of size 75 m at 180 rpm for 1 h, the effect of agitation time with different beginning concentrations of methylene blue was investigated. The sorbent was removed by centrifugation at predefined intervals, and the remaining methylene blue in solution was quantified using a UV spectrophotometer. The next tests used the same spectrophotometric approach. Based on the disparities between the amount of metal provided to the adsorbent biomass and the metal content of the



Fig. 1. Biosorbent preparation from banana leaves waste.

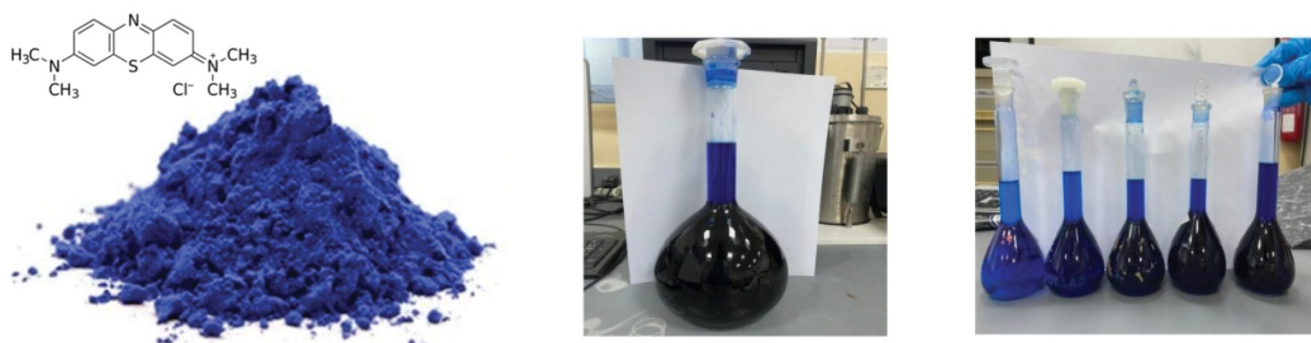


Fig. 2. Preparation of methylene blue solution.

supernatant, the amount of metal absorbed by banana leaves was determined using the following equation:

$$q = (C_0 - C_f) \times \frac{V}{M} \quad (1)$$

where metal uptake represented by q (mg/g), C_0 and C_f represent starting and final concentrations of metal in the solution (ppm), volume of solution represented by V (mL), and adsorbent mass (g) represented by M .

2.5. Experimental design for biosorption studies

Four independent parameters were chosen for the investigation to establish best a % condition of methylene blue elimination, as shown in Table 1.

Preliminary studies determined the (X_1) temperature, (X_2) pH, (X_3) biosorbent dose, and (X_4) beginning dye concentration research ranges. The association the relationship between parameters and response was established utilizing CCD in the RSM of Stat-Ease Inc. (CCD) is a Design-Expert Software Company based in the United States (version 6.0.6) (Fig. 3). In this investigation, the CCD [8] architecture was chosen because it is efficient, adaptable, and resilient. In Table 1 the four levels of the parameters are coded as -2, -1, 0, +1, +2 correspondingly. According to CCD [9], thirty trials were taken with 8-star points ($\alpha = 2$) and 6 replicas of a center point (Y) the experimental design as a response, the % of methylene blue elimination was computed as:

$$(\%)\text{Biosorption} = \frac{(C_i - C_f)}{C_i} \times 100 \quad (2)$$

The initial concentration is C_i (mg/L), while the final or equilibrium concentration is C_f (mg/L). Each experiment was carried out three times, with the average values published. When the necessary incubation period has passed, samples are evaluated using a UV spectrophotometer (Model Shimadzu AA-6650). Design-Expert Software (version 6.0.6), Stat-Ease Inc., USA, was used to conduct regression analysis, graphical analyses, and analysis of variance (ANOVA). Student's F -test and p -values were used to establish the statistical significance of the coefficient [10]. The multiple

Table 1
For methylene blue biosorption onto banana leaves, the experimental range and amounts of the independent factors were

Independent parameters	Range and level				
	-2	-1	0	+1	+2
(X_1) Temperature, K	30	35	40	45	50
(X_2) pH	4	5	6	7	8
(X_3) Adsorbent dosage, g/L	0.1	0.2	0.3	0.4	0.5
(X_4) Initial concentration, mg/L	20	40	60	80	100

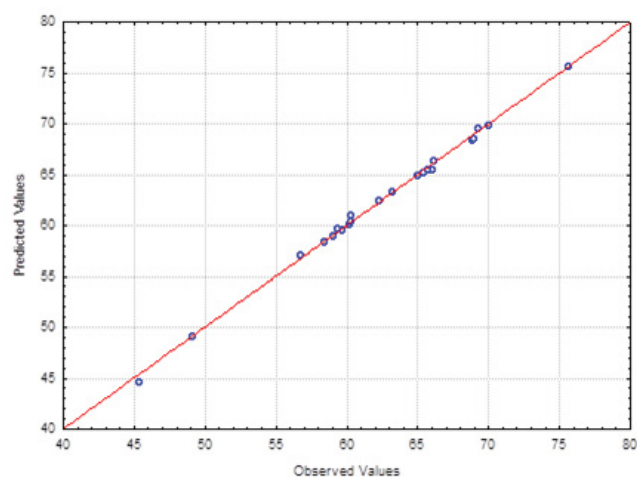


Fig. 3. With banana leaves, plot depicting the distribution of observed vs. predicted percentage biosorption of methylene blue is shown.

coefficients of determination, R^2 , explained the fraction of variance produced by the model.

2.6. Artificial neural network

A network of neural is a smart hub that can predict an output pattern based on the recognizing a specific input pattern. Before being deployed, initially, neural networks are trained by consuming many datasets. Following that, neural networks can find similarities using new patterns, resulting in an estimated output pattern [12]. In the

prediction of adsorption systems, applications of empirical models in conjunction to artificial neural networks (ANN) are recognized as powerful alternatives to numerical estimating approaches [13].

In this case, ANN was utilized to model adsorption based on data from experiments collected in a variety of situations. A simplified architecture is seen in Fig. 4, with input designated by the characters $x_1, x_2, \dots, W_{k1}, W_{k2}, \dots, W_{kn}$ indicate the weight coefficients of inputs. As a result, x_n is used to represent input signals, while W_{kn} is used to represent their weight coefficients. The weighted sum of all incoming signals is calculated the core. The throughputs in the network's function are represented by Y [14]. Depending on the problem's nature, the count of hidden ANN layers can be increased. ANN computations were performed using the MATLAB software suite.

3. Results and discussion

3.1. Statistical analysis

The parameters that have the greatest influence on the reaction must be discovered to determine an optimal condition for methylene blue elimination. The quadratic model fit the association between four independent variables and % of methylene blue elimination well in this investigation. In terms of coded factors, the % of methylene blue removal (after biosorption process) produced from CCD design is given as a quadratic regression model.

$$\begin{aligned}
 Y(\text{Percent dye bicsorption}) = & -212.740 + 12.065X_1 + 1.747X_2 \\
 & + 67.310X_3 + 0.481X_4 - 0.171X_1^2 - 1.954X_2^2 + 34.33X_3^2 \\
 & - 0.003X_4^2 + 0.574X_1X_2 - 2.085X_1X_3 - 0.005X_1X_4 \\
 & + 0.949X_2X_3 + 0.007X_2X_4 - 0.118X_3X_4
 \end{aligned}
 \tag{3}$$

where X_1 stands for temperature, pH for X_2 , biosorbent dosage for X_3 , and initial concentration for X_4 , respectively. Table 2 demonstrates the discrepancies in the corresponding coded values of four parameters and response based on experimental runs and predicted values suggested by CCD design.

The response value for methylene blue removal from aqueous solution ranged from 40.28% to 75.61%. The ANOVA of this model Eq. (3) in Table 3 was significant ($P < 0.05$) with a model F -value of 718.5. The determination coefficient in this investigation was 0.9985, with residues accounting for the remaining 0.15%. The calculated error was created for this model as 0.263. When the adverbial error was higher than 0.05, the model of quadratic was valid

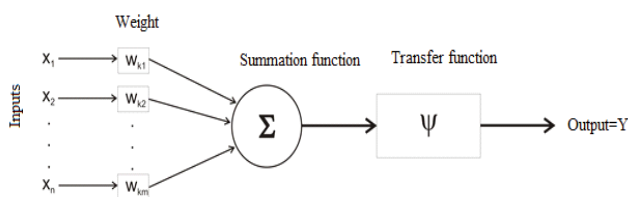


Fig. 4. ANN cell pattern.

for the current study [11]. The first order effects of temperature, pH, biosorbent dose, initial dye concentration, and second-order effects were likewise highly significant by the model, as shown in Table 4.

Temperature of 39.4°C, pH of 5.8, biosorbent dose of 0.27 g, and beginning dye concentration of 53.07 mg/L are the projected optimum conditions with 75.86% methylene blue removal, according to Table 5.

3.2. Adsorbent characterization

3.2.1. Brunauer–Emmett–Teller analysis

Brunauer–Emmett–Teller (BET) surface area and pore volume of adsorbents is tabulated in Table 6. The surface area of 40 m²/g was provided by *Musa acuminata*. BET surface area was observed less due to the particles of *M. acuminata* filled the pore. However, the pore volume of *M. acuminata* was observed as 0.265 cm³/g.

3.2.2. Scanning electron microscopy analysis

Scanning electron microscope (SEM) is a useful technique in the study of the natural sorbent morphology and its modification derived from sorbate interactions. SEM is an electron microscope which provides images of the sample surface by scanning it with a high energy beam of electrons. In this investigation, possible mechanisms involved in the sorption of the toxic elements in biomasses and differences due to the application of the amendments are investigated using SEM.

The SEM images were taken by applying 10 kV voltage with different magnification times for the clarification of surface. These SEM observations of fibrous superficial structure of untreated *M. acuminata* leaves powder are depicted in Fig. 5. It is evident from analysis that the surface area on the leaves biomass is uneven, heterogeneous and contains pores.

SEM analysis after biosorption (Fig. 6) show that the surface has irregular texture with globular, elongated grains and shiny particles over the surface of methylene blue loaded biosorbent which are absent in the fresh biosorbent. These elongated grains show that the methylene blue particles are adhered onto the surface of *M. acuminata* leaves biosorbent.

3.3. Interaction effects of four parameters on methylene blue removal

The present biosorbent's methylene blue ions biosorption capacities were visualized using three-dimensional response surface plots for various combinations of independent factors (Figs. 7–12). Figs. 5–10 show the plots as a function of two elements at a time, with the other factors held constant. All of the response surface plots demonstrated that the biosorbent's capacity was minimum at low and high levels of the variables; nevertheless, there was an area where neither a growing nor a declining trend in the biosorption capacity was detected. This phenomenon indicates that there was an optimum to the biosorption factors to enhance the biosorption dye capacity of methylene blue. Temperature and starting dye concentration (X_1 and X_4), pH and initial

Table 2

For % biosorption of methylene blue with banana leaves, a CCD matrix showing coded and actual values, as well as observed and anticipated values, is shown

Run No.	Coded values				Real values				% Biosorption of methylene blue	
	x_1	x_2	x_3	x_4	X_1	X_2	X_3	X_4	Observed	Predicted
1	-1	-1	-1	-1	35	5	0.2	40	69.2	69.50166
2	-1	-1	-1	1	35	5	0.2	80	58.38	58.39208
3	-1	-1	1	-1	35	5	0.4	40	66.13	66.44041
4	-1	-1	1	1	35	5	0.4	80	59.66	59.52833
5	-1	1	-1	-1	35	7	0.2	40	62.22	62.48542
6	-1	1	-1	1	35	7	0.2	80	60.12	60.19333
7	-1	1	1	-1	35	7	0.4	40	59.01	59.06167
8	-1	1	1	1	35	7	0.4	80	60.23	60.96708
9	1	-1	-1	-1	45	5	0.2	40	65.92	65.58542
10	1	-1	-1	1	45	5	0.2	80	60.21	60.40833
11	1	-1	1	-1	45	5	0.4	40	63.2	63.37667
12	1	-1	1	1	45	5	0.4	80	62.26	62.39708
13	1	1	-1	-1	45	7	0.2	40	59.29	59.67167
14	1	1	-1	1	45	7	0.2	80	63.22	63.31208
15	1	1	1	-1	45	7	0.4	40	56.71	57.10042
16	1	1	1	1	45	7	0.4	80	64.99	64.93833
17	-2	0	0	0	30	6	0.3	60	65.98	65.49625
18	2	0	0	0	50	6	0.3	60	65.72	65.55125
19	0	-2	0	0	40	4	0.3	60	49.12	49.11125
20	0	2	0	0	40	8	0.3	60	45.28	44.63625
21	0	0	-2	0	40	6	0.1	60	69.98	69.81125
22	0	0	2	0	40	6	0.5	60	68.86	68.37625
23	0	0	0	-2	40	6	0.3	20	68.93	68.48458
24	0	0	0	2	40	6	0.3	100	65.42	65.21291
25	0	0	0	0	40	6	0.3	60	75.82	75.82
26	0	0	0	0	40	6	0.3	60	75.82	75.82
27	0	0	0	0	40	6	0.3	60	75.82	75.82
28	0	0	0	0	40	6	0.3	60	75.82	75.82
29	0	0	0	0	40	6	0.3	60	75.82	75.82
30	0	0	0	0	40	6	0.3	60	75.82	75.82

Table 3

ANOVA for the whole quadratic model of methylene blue biosorption onto banana leaves

Source of variation	Sum of squares (SS)	Degrees of freedom (DF)	Mean squares (MS)	F-value	Probe. > F
Model	1,689.912	14	120.708	718.5	0.000000
Error	2.518	15	0.168		
Total	1,692.430	29			

$R^2 = 0.9985$; Adjusted $R^2 = 0.9971$;
 $F_{0.01(14,15)} = S_r^2/S_e^2 = 718.5 > F_{0.01(14,15)}$ Tabular = 2.46;
 $P_{model} > F = 0.000000$.

dye concentration (X_2 and X_4), and biosorbent dosage and initial dye concentration (X_3 and X_4) all had a direct proportional relationship on dye uptake [13]. The fact that the interaction between them was quite significant ($p < 0.05$) [14] and was found to be solely responsible for generating

a pretty high dye uptake as anticipated by the model and the response contour plots confirmed these interactions (Figs. 3–5).

The curved contour lines indicated a relationship between temperature and initial dye concentration (X_1 and X_4),

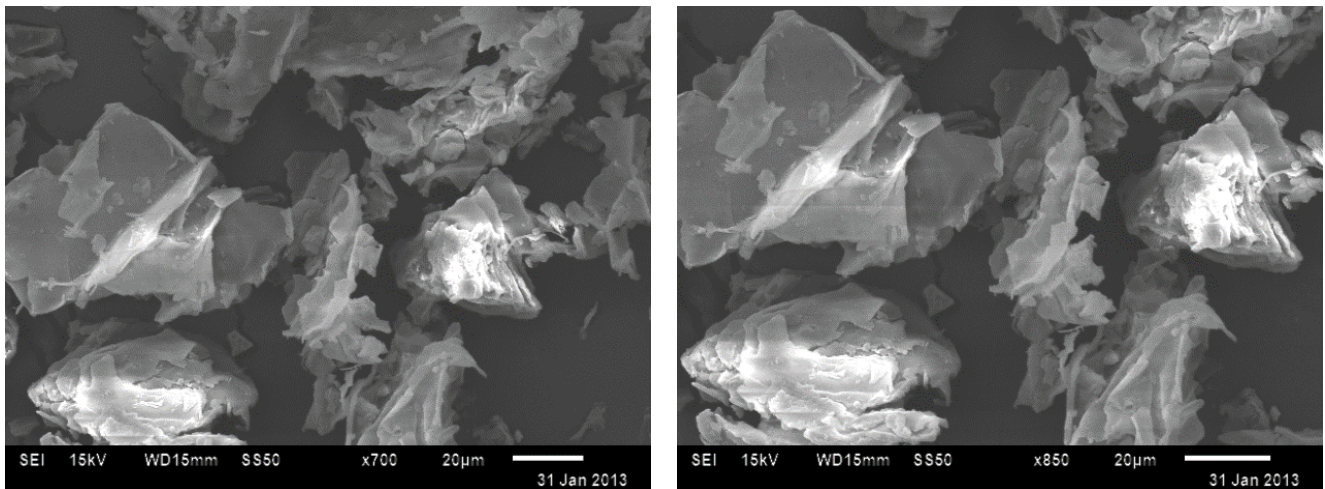


Fig. 5. SEM micrograph of *Musa acuminata* leaves biosorbent before biosorption.

Table 4

Model for biosorption of methylene blue onto banana leaves has coefficients, *t*-statistics, and a significant probability

Term	Coefficient	Value	Standard error of coefficient	<i>t</i> -value	<i>p</i> -value
Constant	b_0	-267.206	13.30745	-20.0795	0.000000 ^a
X_1	b_1	6.723	0.40690	16.5229	0.000000 ^a
X_1^2	b_{11}	-0.101	0.00458	-22.0191	0.000000 ^a
X_2	b_2	76.544	1.75245	43.6781	0.000000 ^a
X_2^2	b_{22}	-7.184	0.11452	-62.7337	0.000000 ^a
X_3	b_3	51.063	14.28647	3.5742	0.004363 ^a
X_3^2	b_{33}	-162.906	11.45168	-14.2255	0.000000 ^a
X_4	b_4	-0.796	0.07143	-11.1402	0.000000 ^a
X_4^2	b_{44}	-0.005	0.00029	-19.1266	0.000000 ^a
X_1X_2	b_{12}	0.055	0.02392	2.3044	0.041709 ^a
X_1X_3	b_{13}	0.426	0.23922	1.7818	0.102366
X_1X_4	b_{14}	0.015	0.00120	12.3998	0.000000 ^a
X_2X_3	b_{23}	-0.906	1.19609	-0.7577	0.464566
X_2X_4	b_{24}	0.110	0.00598	18.4299	0.000000 ^a
X_3X_4	b_{34}	0.525	0.05980	8.7734	0.000003 ^a

X_1 stands for temperature, X_2 for pH, X_3 for biosorbent dosage, and X_4 for initial concentration.

^aSignificant (*p* less than or equal to 0.05).

Table 5

Optimum variable values derived from regression models for the removal of methylene blue from banana leaves

Variables	Value of optimum for zinc
Temperature, °C	39.42077
pH	5.86821
Biosorbent dose, g	0.27744
Zinc initial concentration, mg/L	53.07435
% Predicted biosorption	75.865
% Observed biosorption	76.92

pH and initial dye concentration (X_2 and X_4), and biosorbent dosage and initial dye concentration (X_3 and X_4). In addition, Figs. 7–9 [14] reveal a moderate interaction between temperature and pH (X_1 and X_2), temperature and biosorbent dosage (X_1 and X_3), and pH and biosorbent dosage (X_2 and X_3). The highest anticipated biosorption capacity for the best biosorption variables was calculated using the point prediction method, and the surface response plots are shown in Table 5. The biosorption experiment was then carried out under optimal process conditions, and it was discovered that the experimental data was well represented by Eq. (3) of the current model.

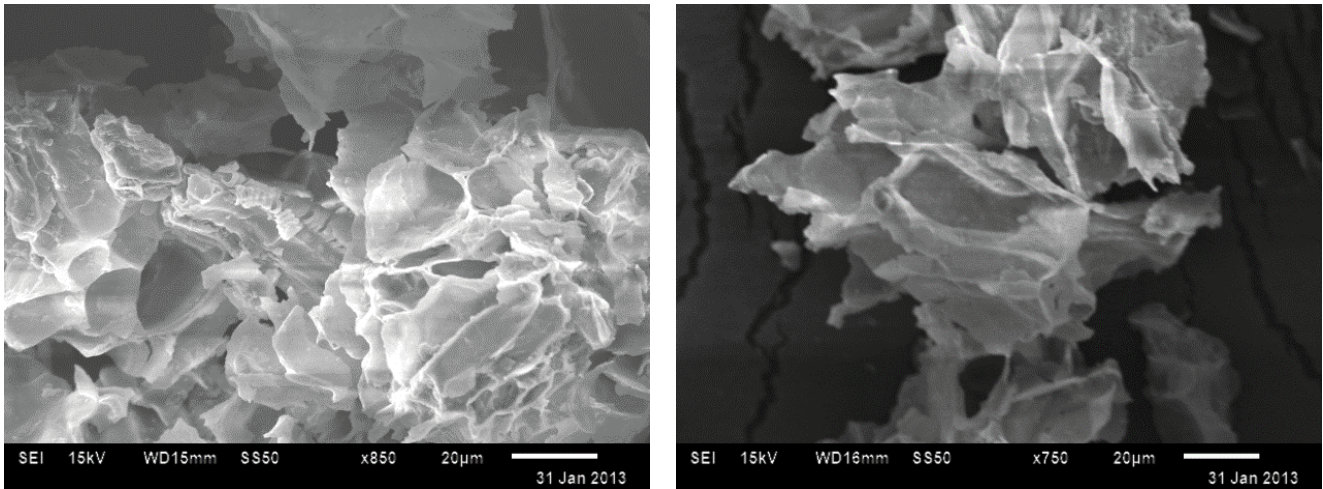


Fig. 6. SEM micrograph of *Musa acuminata* leaves biosorbent after biosorption of MB.

Table 6
Surface area and pore volume of adsorbent

Adsorbent	BET surface area, m ² /g	BET pore volume, cm ³ /g
<i>Musa acuminata</i>	40	0.265

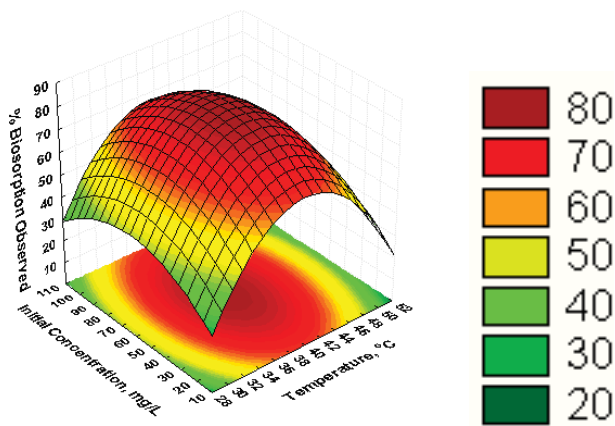


Fig. 7. Response surface plot of the effects of initial dye concentration and temperature on percentage biosorption of methylene blue with banana leaves.

3.4. Artificial neural networks

The created neural network employed as input data, the experimental variables (initial pH, sorbent dosage, and starting concentration) were used to determine the dye adsorbed quantities. Fig. 13 shows the verification, training and test data for the model ANN that made the better prediction. Furthermore, the models' statistical performance was evaluated using the statistical parameters of (SE) standard error, (μ) mean, standard deviation (σ), and (R^2) regression coefficient. Table 7 shows the statistical performance.

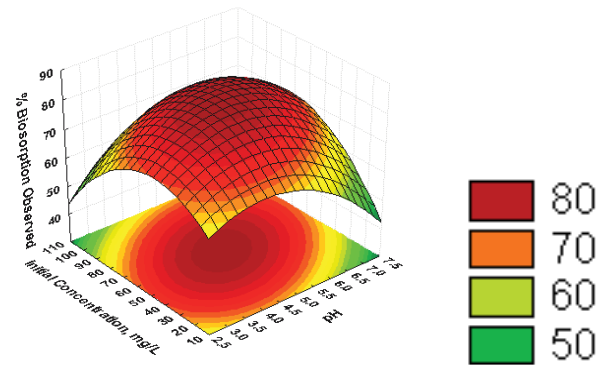


Fig. 8. Response surface plot of the effects of initial dye concentration and pH on percentage biosorption of methylene blue with banana leaves.

These findings show a substantial association between the observed values in the generated models, as shown in table. The relationship between the developed ANN model's prediction outputs and experimental data was structured in order to assess the efficacy of the ANN modeling as a useful tool. Fig. 14 shows a comparison of the experimental and expected results (starting pH, sorbent dosage "x" and initial concentration "C₀"). The experimental and projected findings are in good agreement, as seen in the graph.

As evidenced by strong R^2 values ($R^2 = (0.97)$ test, (0.99) training, and (0.99) validation), ANN demonstrated to be an excellent method for modeling biosorption. The efficiency of the ANN model was determined by maximum of R^2 and reduction in the testing set's MSE value (1–20 neurons correspond to the hidden layer).

There is no noticeable change after 27 epochs of the method's performance, according to the graph for ideal ANN models, the least mean squared error (MSE) vs. the number of epochs (Fig. 15). At epochs 27 and 28, 0.0011846 is the best validation performance. Fig. 13 shows how the network was successfully trained using the resilient back-propagation technique.

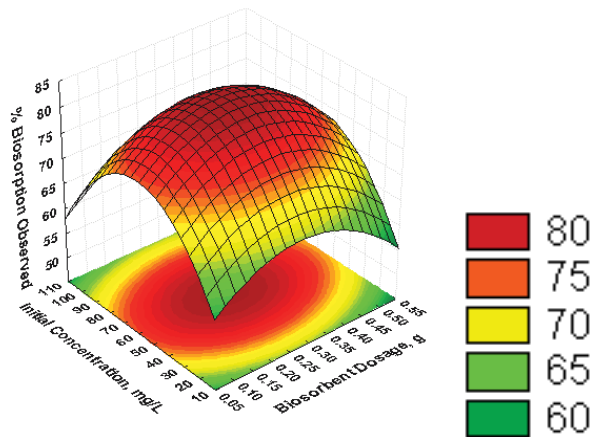


Fig. 9. Response surface plot of the effects of initial dye concentration and biosorbent dosage on percentage biosorption of methylene blue with banana leaves.

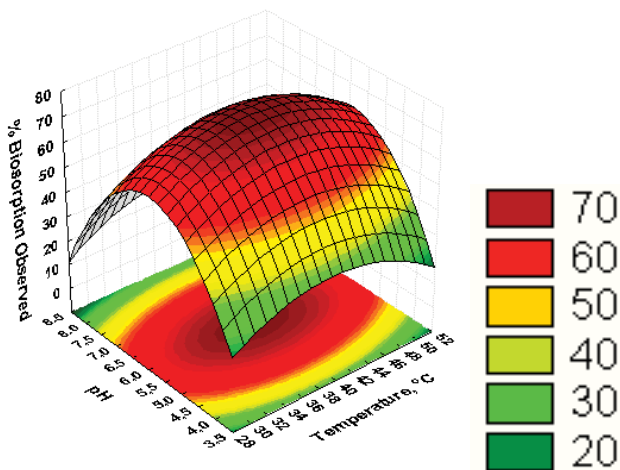


Fig. 10. Response surface plot of the effects of pH and temperature on percentage biosorption of methylene blue with banana leaves.

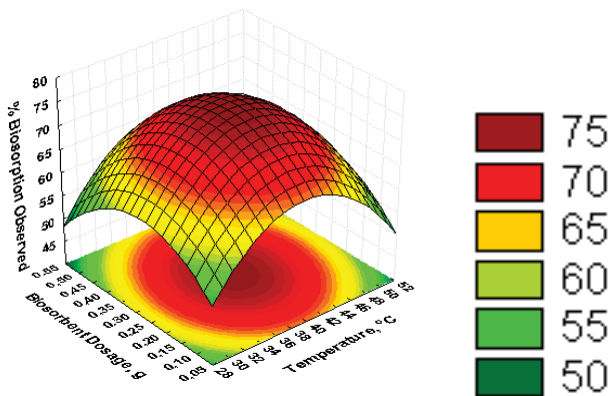


Fig. 11. Response surface plot of the effects of biosorbent dosage and temperature on percentage biosorption of methylene blue with banana leaves.

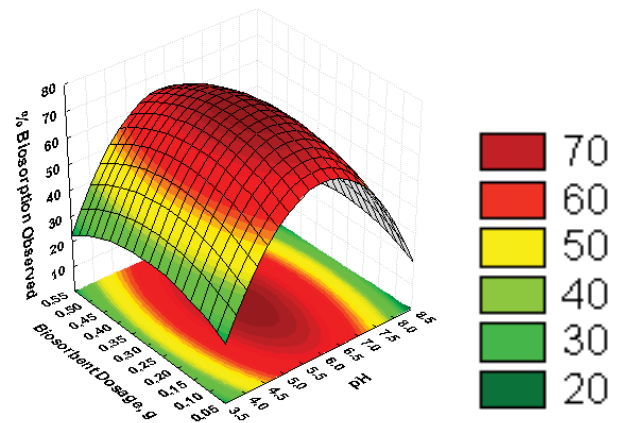


Fig. 12. Response surface plot of the effects of biosorbent dosage and pH on percentage biosorption of methylene blue with banana leaves.

Table 7
ANNs model’s statistical performance

Model	Structure	R ²	σ	SE	μ
I	3-6-1-1	0.99	8.4	2.16	1.01
II	3-5-1-1	0.97	8.4	2.24	0.99
III	3-4-1-1	0.96	8.3	1.64	1.00
IV	3-3-1-1	0.93	8.9	1.29	0.99
V	3-2-1-1	0.94	8.9	1.34	1.01

4. Conclusion

For methylene blue removal utilizing banana leaves as a biosorbent, the RSM was utilized to find the best process parameters. In the elimination of methylene blue dye, the combined effects of pH, temperature, biosorbent dosage, and methylene blue initial concentration resulted in a significant interaction. At pH 5.8, the best conditions for removing 75.86% of 53.07 mg/L of methylene blue from banana leaves were 39.4°C temperature and 0.27 g biosorbent dosage. The surface area of 40 m²/g was provided by *M. acuminata* and the pore volume of *M. acuminata* was observed as 0.265 cm³/g.

The buried layer of a three-layered neural network has six neurons, the biosorption performance of banana leaves during the removal of methylene blue from aqueous solutions was successfully predicted. The experimental data and the predicted results of the developed ANN model were evaluated and found to be with lot of agreement.

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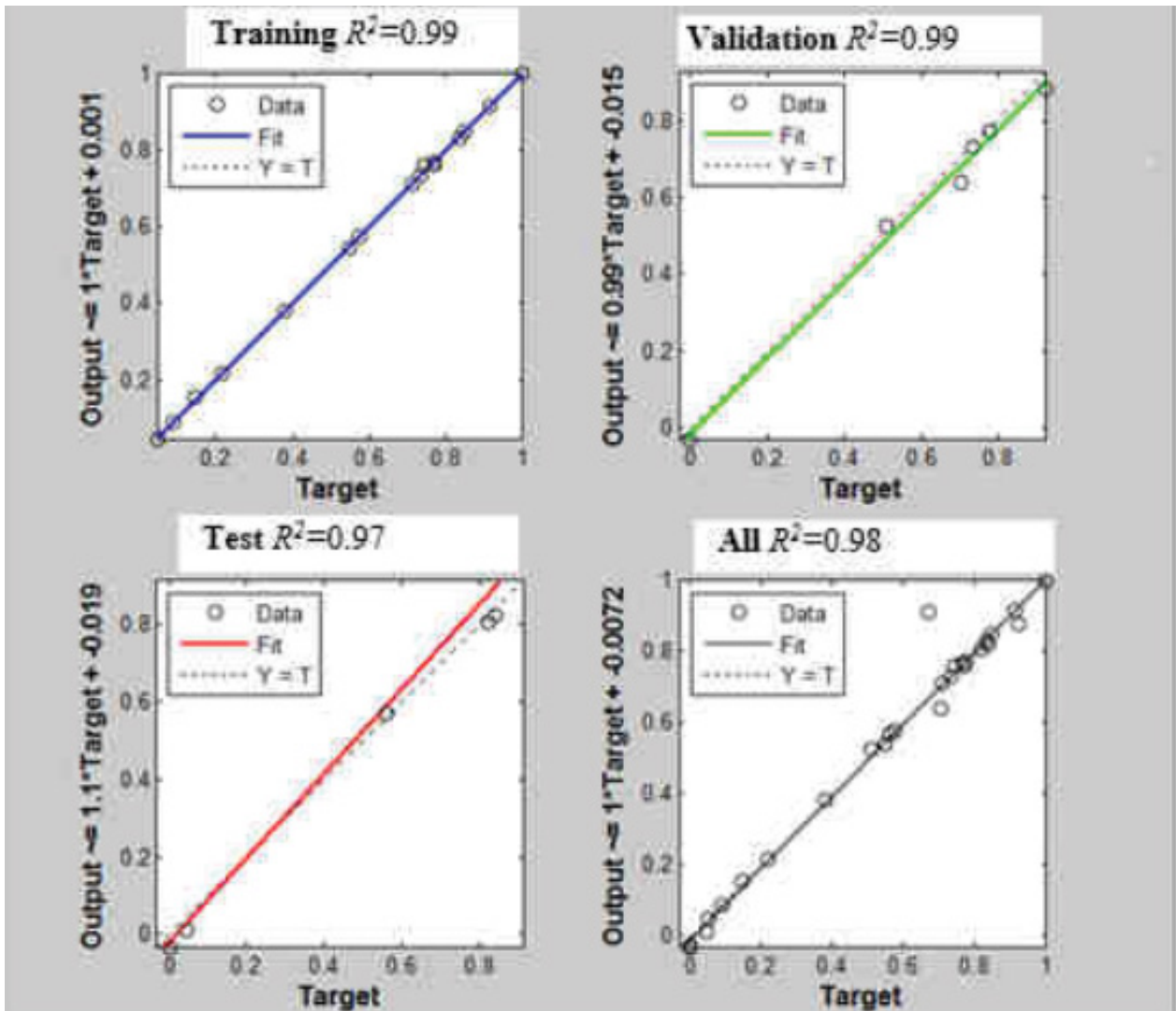


Fig. 13. In terms of dye elimination %, the predicted and target values are compared.

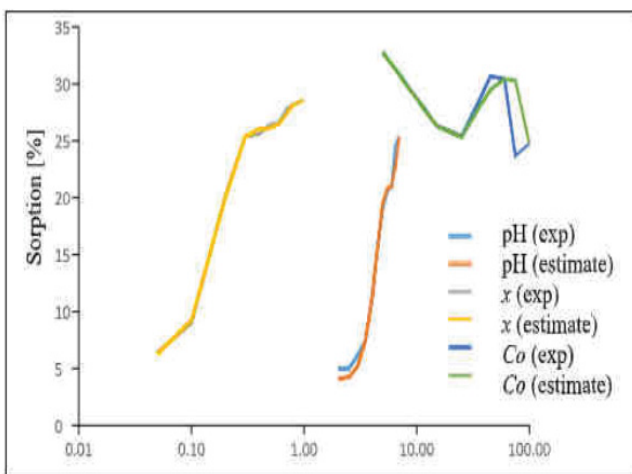


Fig. 14. Findings of the experiment are compared to the model's predictions.

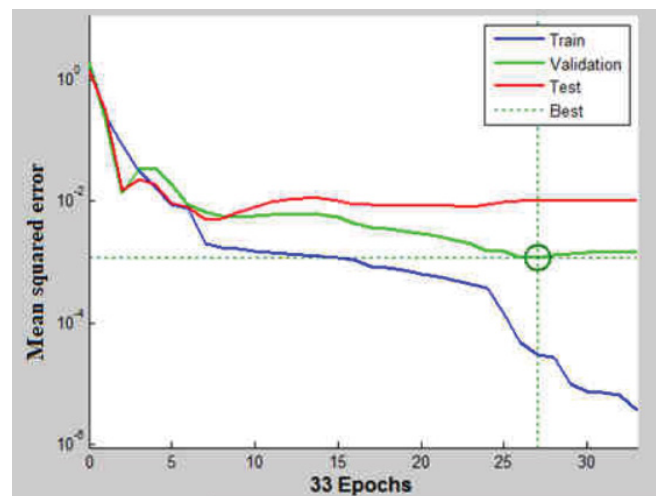


Fig. 15. Methylene blue dye MSE vs. the number of epochs.

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