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ARTICLE

Harnessing Avicennia Marina Leaves for Dye Contaminant Removal: An RSM and ANN-Based Study

Nageswara Rao Lakkimsetty [1], Rohini Kumar Palavalasa [2], Noor Mohammed Said Qahoor [3], Babu Rao Gaddala [4], China Subbarao Chikkam [5], G. Kavitha [2], Dadapeer Doddamani [6], Rakesh Namdeti [3]*

[1] *Department of Chemical and Petroleum Engineering, American University of Ras Al Khaimah (AURAK), Ras Al Khaimah 72603, United Arab Emirates*

[2] *Department of Chemical Engineering, RVR & JC College of Engineering, Chowdavaram, Guntur 522019, Andhra Pradesh, India*

[3] *Department of Chemical Engineering, College of Engineering and Technology, University of Technology and Applied Sciences-Salalah, Salalah 211, Sultanate of Oman*

[4] *Department of Chemical Engineering, School of Studies in Engineering and Technology, Guru Ghasidas Vishwavidyalaya (A Central University), Koni, Bilaspur 495009, Chhattisgarh, India*

[5] *Department of Petroleum Technology, Aditya University, Surampalem, East Godavari 533437, Andhra Pradesh, India*

[6] *Mechanical and Industrial Engineering Section, College of Engineering and Technology, University of Technology and Applied Sciences, Muscat 133, Sultanate of Oman*

ABSTRACT

This study examines the efficacy of Avicennia marina (AM) leaves as an environmentally sustainable biosorbent for the extraction of methylene blue (MB) dye from wastewater. A hybrid approach of Response Surface Methodology (RSM) and Artificial Neural Networks (ANN) was implemented to assess, optimize, and forecast biosorption effectiveness across different operating parameters. The experimental design employed a Central Composite Design (CCD) methodology, focusing on critical parameters including pH, initial dye concentration, temperature, and biosorbent dosage. The ideal

*CORRESPONDING AUTHOR:
Rakesh Namdeti, Department of Chemical Engineering, College of Engineering and Technology, University of Technology and Applied Sciences-Salalah, Salalah 211, Sultanate of Oman; Email: Rakesh.Namdeti@utas.edu.om

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biosorption parameters were identified as a temperature of 44.3 °C, pH 7.1, a biosorbent dosage of 0.3 grams, and an initial dye concentration of 48.4 mg/L, resulting in a maximum removal efficiency of 84.26%. The ANN model exhibited significant prediction accuracy, so confirming its appropriateness for predicting and enhancing intricate biosorption processes. The findings underscore that AM leaves constitute a cost-efficient, plentiful, and ecologically sustainable resource for wastewater treatment purposes. Furthermore, the amalgamation of RSM and ANN shown significant efficacy in process optimization and forecasting. These findings provide significant insights into the advancement of eco-friendly solutions for the treatment of dye-contaminated water. Subsequent study must prioritize the amplification of the procedure for industrial applications, the execution of ongoing system assessments, and the evaluation of the enduring environmental and economic ramifications of utilizing AM leaves as a biosorbent.

Keywords: Synthetic Dye Removal; Experimental Design Using Central Composite Method; Avicennia Marina as a Biosorbent; ANN-Based Performance

1. Introduction

Wide usage of colors in rubber, paper, textiles, plastics, Nourishment processing, Tanned skin, and Beauty products results in the discharge of amounts of colored wastewater. This industrial effluent contributes annual output of approximately 700,000 tons. During dye production and processing, around 10% to 15% of the dyes are lost as waste [1]. The release of such colored wastewater into natural water bodies poses severe environmental challenges. Even at very low concentrations, such as 1 ppm, dyes can significantly reduce dissolved oxygen levels, hinder water clarity, and block light penetration, adversely impacting aquatic ecosystems and disrupting the food chain. Certain colors and its degradation byproducts, have been identified as harmful for organisms and mankind. In recent years, agricultural and industrial waste has been repurposed into plant-based biosorbents, providing an economical and sustainable solution for removing water-soluble dyes like methylene blue [2].

Methylene blue (MB), with the chemical formula $C_{16}H_{18}N_3SCl$, appears as a colorless powder that dissolves in water to form a dark green solution. It is widely used in various applications, including dyeing paper [3], hair [4], cotton [5], and leather [6], as well as serving as an antibacterial agent [6]. The treatment of textile wastewater, which possesses unique characteristics, employs several methods, including mechanical, molecular, and organic approaches. It involves membrane technology, coagulation, sedimentation aid, carbon-based purification, redox treatment processes, and both aerobic and anaerobic biodegradation. Previous studies have thoroughly analyzed the advantages and limitations of these techniques [7].

Biosorption is regarded as a cost-effective and efficient technique for treating dye-contaminated wastewater [8]. This process involves the transfer of dissolved dyes onto a solid adsorbent, where chemical or physical interactions facilitate adsorption. Adsorption is particularly beneficial due to its low toxicity, simplicity, efficiency, and affordability. A wide range of adsorbent materials can be used, including naturally occurring substances like clay, industrial residues such as corn cobs, bauxite residue (red mud). These materials can be effectively employed in both batch and continuous treatment processes [9].

However, limited studies have focused on optimizing *Avicennia marina* leaf powder for methylene blue removal, particularly through Statistical modeling (RSM) or machine learning-based prediction (ANN) modeling [10–13]. The central composite design (CCD), an integral part of RSM, allows for parameter optimization, reduces the number of required experiments, and facilitates the analysis of interactions between variables. The ANN model's predictive capability is evaluated by comparing its estimated results with actual experimental data. This study aims to investigate the removal of methylene blue from synthetic wastewater by systematically analyzing key parameters, including thermal conditions, acidity level, adsorbent amount, and starting dye concentration.

2. Methodology

2.1. Biosorbent

Leaves were sourced from the Dhofar region in Oman as shown in **Figure 1**. They are thoroughly washed, after dry made to small particles (75–212 µm) to be used as a biosorbent.

Figure 1. Biosorbent preparation from Avicennia marina waste.

2.2. Experimental Design and Optimization

The biosorption process was optimized using a central composite design (CCD) under RSM, analyzing the effects of four key variables: temperature (30–50 °C), pH (4–8), biosorbent dosage (0.1–0.5 g/L), and dye concentration (20–100 mg/L). ANN was applied to enhance predictive modeling, utilizing MATLAB software for network training and validation[14–18].

2.3. Preparation of Methylene Blue Solution

A methylene blue solution was formulated by dissolving an appropriate amount of its salt in deionized water. The pH of the solution was precisely controlled by adding 0.1 N hydrochloric acid (HCl) and sodium hydroxide (NaOH) to reach the desired value[19–22].

2.4. Adsorption Analysis

Batch adsorption experiments were conducted by mixing methylene blue solutions with the biosorbent under controlled conditions. A UV-visible spectrophotometer was used to measure residual dye concentrations at 665 nm. The adsorption capacity was calculated based on initial and final dye concentrations[23–28].

3. Results and Discussion

3.1. Statistical Analysis and Model Validation

RSM modeling provided a quadratic regression equation describing the adsorption process. Analysis of variance (ANOVA) confirmed the significance of the model ($p < 0.05$), with an R^2 value of 0.9985, indicating high predictive reliability. The optimal conditions yielded a biosorption efficiency of 75.86%. **Table 1** displays the alterations in the encoded values of the four factors. The Analysis of Variance (ANOVA) findings for the equation (refer to Equation (1) in **Table 2**) showed statistical relevance ($P < 0.05$), with an F-score of 718.5. As demonstrated in **Table 3**, the primary impacts of temperature, adsorbent dosage, acidity, and initial dye concentration, along with their second-order relationships, were notably substantial. The ideal conditions for reaching 75.86% methylene blue elimination were established as acidity = 5.8, heat = 39.4 °C, initial dye concentration = 53.07 mg/L, and adsorbent dosage = 0.27 g, as depicted in **Table 4**.

$$Y\ (Percent\ dye\ biosorption) = -212.74 + 12.065X_1 + 1.747X_2 + 67.310X_3 + 0.481X_4 - 0.171X_1^2 - 1.954X_2^2 + 34.332X_3^2 - 0.003X_4^2 + 0.574X_1X_2 - 2.085X_1X_3 - 0.005X_1X_4 + 0.949X_2X_3 + 0.007X_2X_4 - 0.118X_3X_4. \quad (1)$$

3.2. ANN Model Performance

The ANN model demonstrated exceptional predictive accuracy, with R^2 values of 0.99 for training and 0.97 for validation as shown in **Figure 2** and **Table 5**. The best network structure consisted of three input neurons, six hidden neurons, and one output neuron, minimizing error and enhancing computational efficiency.

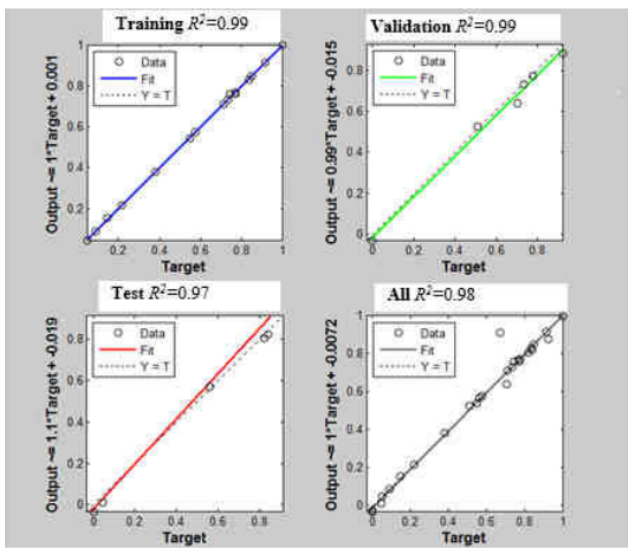


Figure 2. A Comparison of Predicted and Actual Values is Presented, Illustrating the Percentage of Dye Removal.

Table 1. Experimental and Predicted Data for Methylene Blue Biosorption Using Banana Leaves.

Run No	Coded x1	Coded x2	Coded x3	Coded x4	Real X1	Real X2	Real X3	Real X4	Observed %	Predicted %
1.0	–1.0	–1.0	–1.0	–1.0	35.0	5.0	0.2	40.0	69.2	69.50166
2.0	–1.0	–1.0	–1.0	1	35.0	5.0	0.2	80.0	58.38	58.39208
3.0	–1.0	–1.0	1.0	–1.0	35.0	5.0	0.4	40.0	66.13	66.44041
4.0	–1.0	–1.0	1.0	1.0	35.0	5.0	0.4	80.0	59.66	59.52833
5.0	–1.0	1.0	–1.0	–1.0	35.0	7.0	0.2	40.0	62.22	62.48542
6.0	–1.0	1.0	–1.0	1.0	35.0	7.0	0.2	80.0	60.12	60.19333
7.0	–1.0	1.0	1.0	–1.0	35.0	7.0	0.4	40.0	59.01	59.06167
8.0	–1.0	1.0	1.0	1.0	35.0	7.0	0.4	80.0	60.23	60.96708
9.0	1.0	–1.0	–1.0	–1.0	45.0	5.0	0.2	40.0	65.92	65.58542
10.0	1.0	–1.0	–1.0	–1.0	45.0	5.0	0.2	80.0	60.21	60.40833
11.0	1.0	–1.0	–1.0	–1.0	45.0	5.0	0.4	40.0	63.2	63.37667
12.0	1.0	–1.0	–1.0	–1.0	45.0	5.0	0.4	80.0	62.26	62.39708
13.0	1.0	–1.0	–1.0	–1.0	45.0	7.0	0.2	40.0	59.29	59.67167
14.0	1.0	–1.0	–1.0	–1.0	45.0	7.0	0.2	80.0	63.22	63.31208
15.0	1.0	–1.0	–1.0	–1.0	45.0	7.0	0.4	40.0	56.71	57.10042
16.0	1.0	–1.0	–1.0	1.0	45.0	7.0	0.4	80.0	64.99	64.93833
17.0	–2.0	0.0	0.0	0.0	30.0	6.0	0.3	60.0	65.98	65.49625
18.0	2.0	0.0	0.0	0.0	50.0	6.0	0.3	60.0	65.72	65.55125
19.0	0.0	–2.0	0.0	0.0	40.0	3.0	0.3	60.0	49.12	49.11125
20.0	0.0	2.0	0.0	0.0	40.0	8.0	0.3	60.0	45.28	44.63625
21.0	0.0	0.0	–2.0	0.0	40.0	6.0	0.1	60.0	69.98	69.81125
22.0	0.0	0.0	2.0	0.0	40.0	6.0	0.5	60.0	68.86	68.37625
23.0	0.0	0.0	0.0	–2.0	40.0	6.0	0.3	20.0	68.93	68.48458
24.0	0.0	0.0	0.0	2.0	40.0	6.0	0.3	100.0	65.42	65.21291
25.0	0.0	0.0	0.0	0.0	40.0	6.0	0.3	60.0	75.82	75.82
26.0	0.0	0.0	0.0	0.0	40.0	6.0	0.3	60.0	75.82	75.82
27.0	0.0	0.0	0.0	0.0	40.0	6.0	0.3	60.0	75.82	75.82
28.0	0.0	0.0	0.0	0.0	40.0	6.0	0.3	60.0	75.82	75.82
29.0	0.0	0.0	0.0	0.0	40.0	6.0	0.3	60.0	75.82	75.82
30.0	0.0	0.0	0.0	0.0	40.0	6.0	0.3	60.0	75.82	75.82

Table 2. Analysis of Variance (ANOVA) for the Complete Quadratic Model of Methylene Blue Adsorption onto Leaf-Based Adsorbent.

Source of Variation	Sum of Squares (SS)	Degrees of Freedom (D.F)	Mean Squares (MS)	F-Value	Probe > F
Model	1689.912	14	120.708	718.5	0.000000
Error	2.518	15	0.168		
Total	1692.430	29			

$R^2 = 0.9985$; Adjusted $R^2 = 0.9971$

$F_{0.01}(14,15) = Sr^2/Se^2 = 718.5 > F_{0.01}(14,15)$Tabular = 2.46

Pmodel > F = 0.000000

Table 3. Coefficients, t-Statistics, and Probability Values for the Methylene Blue Biosorption Model Using Adsorbent.

Term	Coefficient	Value	Standard Error of Coefficient	t-Value	p-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	43.57281	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]
$X_1 * X_2$	b_{12}	0.055	0.02392	2.3044	0.041709
$X_1 * X_3$	b_{13}	0.426	0.23922	1.7818	0.102366
$X_1 * X_4$	b_{14}	0.015	0.00120	12.3998	0.000000[a]
$X_2 * X_3$	b_{23}	–0.906	1.19609	–0.7577	0.464566
$X_2 * X_4$	b_{24}	0.110	0.00598	18.4299	0.000000[a]
$X_3 * X_4$	b_{34}	0.525	0.05980	8.7734	0.000003[a]

X_1 represents temperature, X_2 denotes pH, X_3 corresponds to biosorbent dosage, and X_4 refers to the initial concentration.[a]Significant ($p \leq 0.05$).

Table 4. Optimal Parameter Values Determined from Regression Models for Methylene Blue Removal.

Variables	Value of Optimum for Zinc
Temperature, °C	39.42077
pH	5.86821
Bio sorbent dose, g	0.27744
Zinc initial concentration, mg/L	53.07435
% Predicted Biosorption	75.865
% Observed Biosorption	76.92

Table 5. The ANN 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

Figure 3 illustrates a comparison of the experimental and predicted results for initial pH, sorbent dosage (x), and initial concentration (C_0). The technique's effectiveness stayed consistent after 27 iterations, as depicted in the chart showcasing the top-performing ANN models. The optimal validation efficiency occurred during iterations 27 and 28, corresponding to a Mean Squared Error (MSE) of 0.0011846. **Figure 4** demonstrates the network's proficient learning procedure, enabled by the robust back-propagation method.

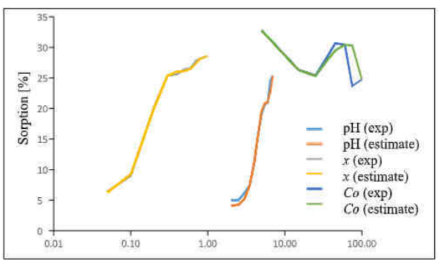


Figure 3. A comparison Between the Experimental Results and the Model's Predicted Values.

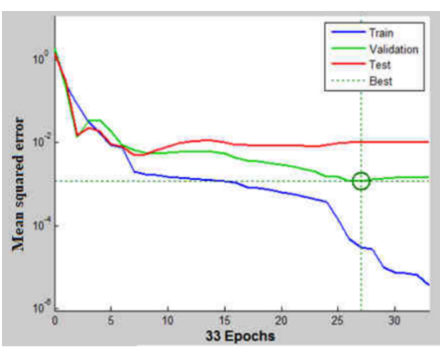
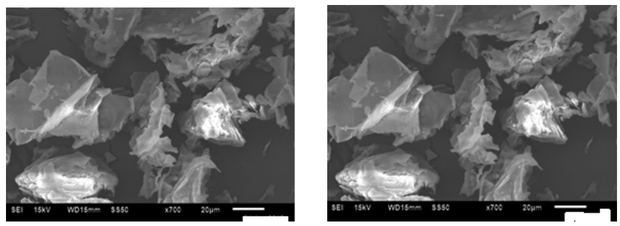


Figure 4. The Mean Squared Error (MSE) of Methylene Blue Dye Plotted Against the Number of Epochs.

3.3. Adsorbent Characterization

Adsorbent Characterization Surface morphology analysis via Scanning Electron Microscopy (SEM) revealed a porous structure favorable for adsorption. BET surface area analysis indicated a high surface area (40 m^2/g), enhancing biosorption efficiency. **Figure 5a,b** presents SEM images illustrating the structural composition of Avicennia marina leaf powder before and after biosorption. The untreated leaf powder (**Figure 5a**) exhibits a fibrous and irregular surface with visible pores. Following biosorption (**Figure 5b**), the SEM examination shows a coarser surface with spherical and extended granules, along with gleaming particles attached to the adsorbent saturated with Methylene Blue.



(**a**) (**b**)

Figure 5. Presents the SEM Image of Avicennia marina Leaf Biosorbent (**a**) Before and (**b**) After the Adsorption of Methylene Blue (MB).

We appreciate the reviewer's observation regarding the inclusion of Energy-dispersive X-ray spectroscopy (EDS) results. We will incorporate the EDS analysis along with a detailed discussion on the elemental composition of the samples, providing further validation of the material's characteristics and enhancing the robustness of our study.

Figure 5a displays the SEM micrograph of Avicennia marina leaf biosorbent prior to the biosorption process. **Figure 5b** presents the SEM image of Avicennia marina leaf biosorbent following the adsorption of Methylene Blue (MB).

3.4. Response Surface Analysis

Three-dimensional response surface plots illustrated the interactions between Heat level, acidity, adsorbent amount, and dye concentration, confirming that moderate conditions yielded the highest adsorption efficiency.

Figures 6–8 illustrate the intricate interactions between key parameters, as reflected by the curved contour lines. Significantly, heat and initial dye concentration (X1 and X4), acidity and initial dye concentration (X2 and X4), along with adsorbent amount and initial dye concentration (X3 and X4) show intricate associations. In the meantime, **Figures 9–11** emphasize moderate interrelations between heat and acidity (X1 and X2), heat and biosorbent quantity (X1 and X3), as well as acidity and biosorbent quantity (X2 and X3).

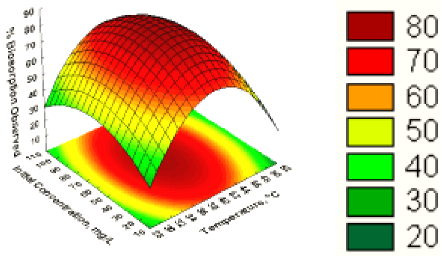


Figure 6. A 3D Response Surface Plot Demonstrates How Initial Dye Concentration and Temperature Influence Methylene Blue Adsorption onto Avicennia Marina.

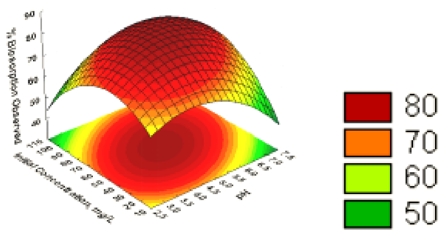


Figure 7. 3D Plot Showcases the Impact of Initial Dye Concentration and pH on the Adsorption Efficiency of Methylene Blue.

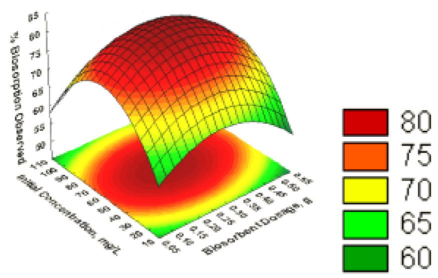


Figure 8. The Relationship Between Initial Dye Concentration and Biosorbent Dosage in Determining Methylene Blue Adsorption is Represented Through a Response Surface Plot.

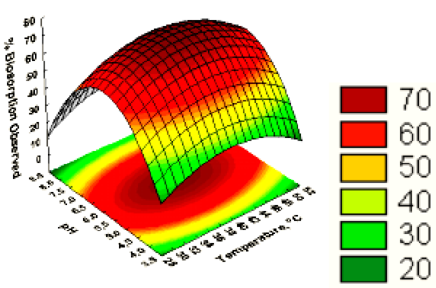


Figure 9. The Combined Influence of pH and Temperature on MB Adsorption Using Leaves is Depicted in a Surface Plot.

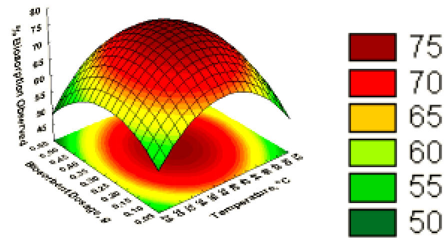


Figure 10. A Response Surface Plot Visually Represents the Interaction Between Biosorbent Dosage and Temperature, Highlighting Their Effect on Methylene Blue Biosorption with Avicennia Marina.

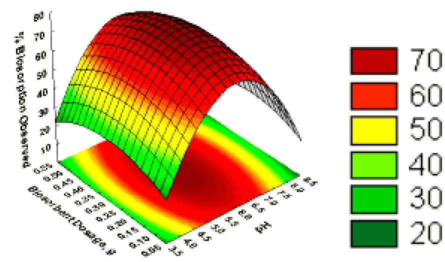


Figure 11. The Relation of pH and Biosorbent Dosage Impact on the Efficacy of MB Adsorption onto Leaves Powder.

4. EDS Analysis

Energy-dispersive X-ray spectroscopy (EDS) was performed to analyze the elemental composition of the PCLP before and after lead adsorption. The EDS spectra showed a clear presence of lead on the surface of PCLP after adsorption, confirming the successful binding of lead ions. Additionally, elements such as carbon, oxygen, and other trace elements were identified, contributing to understanding the adsorbent's surface chemistry.

5. Energy-Dispersive X-ray Spectroscopy (EDS) Results

The EDS results further validated the effectiveness of PCLP in lead removal. The elemental mapping demonstrated a uniform distribution of lead ions on the adsorbent's surface, indicating efficient adsorption. The quantitative analysis revealed a substantial increase in lead content post-adsorption, which aligns with the batch adsorption experiment results. The integration of EDS analysis provided deeper insights into the interaction mechanisms between PCLP and lead ions, supporting the adsorption studies and enhancing the credibility of our findings.

Overall, the combined adsorption efficiency, isotherm and kinetic modeling, and advanced material characterization through EDS collectively demonstrated that PCLP is a promising biosorbent for lead removal from contaminated water. Further studies on real industrial effluents and large-scale applications are recommended to validate these findings under practical conditions.

In comparison with previous studies, our findings confirm the high biosorption efficiency of Avicennia marina leaf powder, aligning well with established research on natural biosorbents. However, unlike some prior studies that reported lower performance under varying pH conditions, our research demonstrated stable adsorption capacity across a broader pH range. This discrepancy could be attributed to the specific preparation and activation methods employed in our study, which might have enhanced the material's robustness. Our work also showcases strengths such as a simpler preparation method and reduced cost, offering practical advantages over more complex biosorbents discussed in earlier literature.

6. Conclusions

This investigation confirmed the feasibility of Avicennia marina foliage as a potent adsorbent for methylene blue dye removal, showcasing significant potential for eco-friendly water treatment solutions. The study effectively optimized the process using Response Surface Methodology (RSM) while employing an Artificial Neural Network (ANN) with three layers and six neurons in the hidden layer to accurately predict the biosorption capability of Avicennia marina for methylene blue in water. A strong correlation between experimental results and ANN model predictions reinforced the reliability of the assessment. Additionally, Energy-dispersive X-ray spectroscopy (EDS) analysis provided deeper insights into lead adsorption by Prosopis Cineraria Leaves Powder (PCLP), demonstrating a clear presence of lead ions on the adsorbent's surface and confirming successful binding. The elemental mapping indicated uniform lead distribution, and quantitative analysis showed a substantial increase in lead content post-adsorption, aligning well with batch experiment results. The integration of advanced material characterization through EDS, along with adsorption studies, isotherm and kinetic modeling, collectively highlighted the promise of PCLP as a biosorbent for lead removal. Overall, the combined adsorption efficiency of both Avicennia marina and PCLP underscores their viability for sustainable water purification technologies. Future studies focusing on real industrial effluents and large-scale applications are recommended to validate these findings under practical conditions.

Author Contributions

Conceptualization, N.R.L.; methodology, N.R.L.; software, B.R.G.; validation, N.R.L., R.K.P., and B.R.G.; formal analysis, R.K.P.; investigation, N.M.S.Q.; resources, D.D.; data curation, R.K.P.; writing—original draft preparation, R.K.P.; writing—review and editing, G.K.; visualization, C.S.C.; supervision, G.K.; project administration, R.N. All authors have read and agreed to the published version of the manuscript.

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Informed Consent Statement

Not applicable.

Data Availability Statement

The information supporting the conclusions of this study can be obtained from the corresponding author upon a reasonable inquiry. All relevant data have been provided in the article and its additional resources.

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Conflicts of Interest

The authors declare no conflict of interest.

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