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Research Article

Fixed-Bed Column Adsorption of Methylene Blue onto Dried Activated Sludge: Modeling and Optimization with ANNs

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Abstract:

This study investigates the continuous adsorption of methylene blue (MB) onto dried activated sludge in a fixed-bed column. The effects of key operational parameters—initial MB concentration (50-300 mg/L), flow rate (2.5-7 mL/min), and bed height (2.5-5 cm)—on the breakthrough behavior were systematically examined. An Artificial Neural Network (ANN) model was developed to predict the dynamic adsorption process. The ANN demonstrated exceptional predictive power, achieving a near-perfect correlation (R = 0.998) with low error values (RMSE = 0.018). Sensitivity analysis further revealed the relative importance of each input parameter. The results conclusively establish the ANN as a powerful and accurate tool for modeling the complex dynamics of MB adsorption onto this waste-derived adsorbent, facilitating potential process optimization.

1. Introduction

Wastewater from textile dyeing and finishing factories represents a paramount environmental pollution source. Numerous businesses, particularly textile industry, disseminate tinted effluents containing dyes and pigments. According to the evaluation, it has been determined that approximately 10 to 15% of the dye content is lost in the effluent during the dyeing process. Additionally, it has been observed that color compounds, which include dyes and pigments discharged from various industries, pose significant risks to the aquatic life in rivers and lakes [1-3]. The market offers a wide range of over 100,000 dyes available for commercial purposes. Due to their complex structure and synthetic origin, these dyes pose significant challenges when it comes to the decolorization process [4]. Therefore, it becomes crucial to effectively eliminate these dyes from effluent streams, not only to address water coloration but also to safeguard the environment [2,5]. Several methods are accessible for the decontamination of dyes, including solvent extraction, photocatalytic degradation, membrane filtration, ion exchange, adsorption, chemical precipitation, and coagulation-flocculation. These procedures offer a variety of approaches to effectively treat and remove dyes from contaminated sources [6, 7]. Among these methods, adsorption is the most commonly employed technique due to its high efficiency and capability to effectively capture a wide range of chemical compounds. Adsorption has proven to be a versatile and effective approach in the treatment of various contaminants, making it a preferred choice in many applications [8]. Adsorption is a widely recognized and impactful technique employed in the treatment of domestic and industrial waste. Activated carbon serves as the most commonly utilized adsorbent for eliminating dyes and treating textile effluents. However, its extensive adoption is hindered by its relatively high cost, limiting its widespread application [9,10].Adsorption can generally be categorized into two main types based on the process method: static adsorption and dynamic adsorption. Static adsorption, also known as batch adsorption, occurs in a closed system where a specific amount of adsorbent comes into contact with a given quantity of adsorbate solution. On the other hand, dynamic adsorption takes place in an open system where the adsorbate solution continuously flows through a column packed with adsorbent.Dynamic adsorption, often referred to as fixed-bed adsorption, is a widely utilized technique for separation and purification processes. It is favored for its high effectiveness and ease of operation. In this method, the adsorbate solution continuously passes through the adsorbent bed, allowing for efficient removal of target substances. Dynamic adsorption has found extensive application in various industries due to its ability to achieve effective separation and purification of desired components [11].Various adsorbents have been documented in the literature for dye removal. These include maize cob carbon [12], cinnamon bark [13], hydrogels [14], fly ash, and zeolite [15], as well as chitosan [16]. Another promising alternative for detoxification is the utilization of microorganisms as adsorbents for dyes. The cell walls of microorganisms primarily consist of multiple organic components such as chitin, lipids, amino acids, and other cellular constituents, which facilitate the passive uptake of reactive dyes [17,18]. Recent studies have demonstrated that different types of biomass exhibit impressive adsorption capacities for heavy metal ions, organic pollutants, and dyes [19-23]. Notably, the tried activated sludge generated from wastewater treatment processes represents a particularly abundant source of microbial biomass.The utilization of artificial neural networks (ANN) as intelligent approaches for predicting and simulating complex systems has gained significant popularity in recent years [24,25]. ANN has emerged as an appealing alternative to traditional methods due to its high parallelism, robustness [26], and, notably, its ability to capture highly nonlinear and complex relationships between variables in a problem using experimental data [27], without requiring exhaustive knowledge of the system [28].In the context of dynamic adsorption phenomena, ANN has been increasingly employed as a "black box" modeling tool. For instance, *Cavas et al*. [29] developed an ANN to investigate the dynamic removal of methylene blue from an aqueous solution using *Posidonia* oceanic (L.) dead leaves as a beach waste adsorbent. Their study demonstrated the superiority of ANN in achieving favorable results. Similarly, *Tovar-Gómez et al*. [30] employed a hybrid neural network approach to model the fixed-bed adsorption of fluoride on bone char. They found that ANN yielded satisfactory outcomes in their research. In another study, *Dalhat et al*. [31] created an artificial neural network to predict the fixed-bed adsorption of phenolic chemicals onto activated date palm biochar, reporting improved results with the use of ANN. *Alakent et al*. [32] used statistic and machine learning toolbox to predict the methylene blue adsorption under various operating conditions on different clay minerals and alkali activated materials such as Koalinite and Sepiolite. They obtained important results and their study was a powerful guideline for methylene blue uptake in clay minerals and under different operating conditions. *Rebouh et al*. [33] developed a neuro fuzzy model to predict the adsorption of Cu(II) and Cr(VI) from waste water using wheat straw. Their modeling studies give satisfactory results. The dynamic adsorption of organic pollutants on active carbon under different operating conditions was predicted by *Msallem et al.* [34] using ANN model, support vector machine (SVM) and Adaptive Neuro- Fuzzy Interference System (ANFIS) model. Their results show the superiority of SVM-DA model against other tested model to estimate accurately the dynamic adsorption phenomena. *Moreno-Pérez et al.* [35] applied the ANN model to describe the dynamic adsorption of a multi-component of heavy metals using biochar. Their results present the robustness of ANN model with a height correlation coefficient. ANN was exploited by Yusuf et *al.* [36] to model the heavy metals dynamic on surfactant decorated graphene. ANN was also applied by *Sediri et al.* [37] to describe the dynamic adsorption of complex system from aqueous solution. Their performances results provide that ANN is a strong tool to predict the behavior of adsorption phenomena. The current study concentrates on investigating the dynamic adsorption of methylene blue (MB) onto dried activated sludge. MB is a widely employed substance in the dyeing of cotton, wood, and silk.

However, it possesses hazardous properties and can cause eye burns, resulting in potential permanent injury to both humans and animals [9,10]. Consequently, there is a significant interest in treating effluents containing MB dye due to its detrimental effects on receiving bodies of water. The ability of dried activated sludge biomass to remove methylene blue MB from aqueous solution was studied. The influence of some essential parameters, namely initial MB concentration, flow rate, and bed height, on the dynamic adsorption of MB on this dried activated sludge was investigated. The modeling of the experimental data was performed with ANN model in order to describe and to predict the dynamic adsorption phenomena of MB.

2. Materials and methods

2.1 Preparation of the biosorbent

The aerobic dried activated sludge used in this study was obtained from the STEP (Sewage Treatment Plant) of Oued Lahrèche, Médéa, Algeria. The cells of the dried activated sludge were subjected to centrifugation at 5,000 rpm for 5 minutes and thoroughly washed with sterile distilled water. Subsequently, the sludge was dried at 60°C for 24 hours before being utilized. To examine the surface morphology of the dried activated sludge, scanning electron microscopy (SEM) analysis was performed using a QUANTA 2000 scanning electron microscope. Fourier transform infrared (FT-IR) spectroscopy, specifically using a SHIMADZU FT-IR-8400 spectroscope, was employed to detect the functional groups present on the surface of the dried activated sludge. The spectra were recorded in the range of 4,000 to 400 cm^{-1}.It's worth noting that this particular aerobic activated sludge was previously used by Cherifi. et *al*. [38] in their previous article to investigate the batch adsorption of methylene blue under various operating conditions.

2.2 Chemicals

The model adsorbate utilized in the experiment was methylene blue, obtained from Aldrich with a purity of 99.9%. To create a stock solution of 1,000 mg/L, an adequate amount of methylene blue was dissolved in one liter of distilled water. The working solutions were then prepared by diluting the stock solution (1,000 mg/L) with distilled water, thereby achieving the desired concentration for each working solution.

2.3 Analysis

The methylene blue concentration in the supernatant solution was analyzed using a UV spectrophotometer (Shimadzu UV Mini-1240) by monitoring the absorbance changes at a wavelength of maximum absorbance of 665 nm [9,12].

2.4 Fixed-bed adsorption studies

Experiments were conducted in a laboratory-scale fixed bed reactor (Schema 1). The reactor comprises:

(1) A feed tank filled with an aqueous solution,
(2) A pump to control the flow rate and liquid velocity within the fixed bed reactor,
(3) A fixed bed of glass beads positioned at both the bottom and top of the column,
(4) A grid with a 2mm mesh,
(5) Dried activated sludge adsorbent with equivalent particle sizes ranging 220 µm,
(6) A cylindrical glass column measuring 60 cm in height and 1.5 cm in diameter,
(7) A collecting tank.

Activated sludge was packed into the column to achieve the desired bed height. The dynamic dye adsorption experiments were conducted at a temperature of 298.15±1 K. To investigate the impact of initial concentration, flow rate, and bed height on the breakthrough performance of the column, a set of experiments were conducted at different range of values presented in table 1. Samples were collected at regular time intervals from the outlet of the column, and the dye concentration in the effluent was analyzed using a UV/VIS spectrophotometer.

2.5 Artificial neural network model

Our study involves the development of an Artificial Neural Network (ANN) model to predict the dynamic adsorption behavior of methylene blue on dried activated carbon. ANNs are intelligent models inspired by biological systems and exhibit strong nonlinearity [39,40]. This concept is employed to execute software simulations for the large parallel processes that contain processing elements interconnected in network structure. In the human brain, learning achieves in a network of neurons that are interconnected between them by axons, synapse and dendrites. The resistance of variable synaptic has an impact on the flow of information between two biological neurons. The artificial neuron receives inputs that are similar to the electrochemical impulses that the dendrites of biological neurons receive from other neurons. Therefore, ANN can be seen as a network of neurons in elements and weighted connections form. In the human brain, the connections and weights are analogous to axons and synapses respectively. The ANN, simulating human brain analytical function, has a high ability to solve the relationships between input and output parameters of complex and non-linear systems [27]. The hyperparameters of ANN model as input parameters, output parameters, type of training algorithm, number of neurons in hidden layer, transfer function were chosen based on interactions between organic compounds properties, adsorbent characteristics and removal operating conditions. The best structure of ANN model corresponds to the best agreement between experimental and predicted data [41]. There are various ANN structures, with the Multilayer Perceptron (MLP-ANN) being the most commonly employed architecture [42-46]. The MLP-ANN comprises an input layer, hidden layer(s), and an output layer. Neurons within each layer are interconnected through weighted connections. For our research, we carefully selected input and output variables based on the interactions between organic compound properties, adsorbent characteristics, and dynamic adsorption conditions. The inputs considered were the initial concentration (C_0), mass of adsorbent (m), bed height (H), time (t), and volume flow (Q). The output variable was the reduced concentration (C / C_0). The ranges of the database utilized for training the ANN model are presented in Table 2. The dynamic adsorption of methylene blue on dried activated carbon was studied using MLP-ANN calculations performed with STATISTICA software (STATISTICA 8.0, Tulsa; StatSoft, Inc.). A database comprising 367 data points was utilized and divided into three phases: training, validation, and testing. The data was divided into 70% for the training phase (phase containing a big number of dataset in order to ensure a good learning of ANN model), 15% for the testing phase and 15% for the validation phase. Consequently from 367 experimental data, 257 data were used for training phase, 55 data for testing phase and 55 data for validation phase. This division was based on trial and error. In this investigation, the input layer consisted of a fixed number of five neurons, while the number of hidden neurons was optimized through a trial and error procedure during the training process. The output layer consisted of a single neuron representing the observed reduced concentration of methylene blue. For the hidden layer, the logistic function (Hyperbolic tangent) was employed as the transfer function, while a linear function was used for the output layer. The network was trained using the quasi-Newton BFGS (Broyden–Fletcher–Goldfarb–Shanno) algorithm [47]. To obtain the optimal structure of the ANN model, each topology was repeated five times. The number of epochs was set to 1000. The structure of the developed ANN is depicted in (Schama2).

2.6 Statistical Analysis

To assess the goodness of fit of the model to the experimental results, an error function evaluation is necessary. In this case, using MATLAB, error analyses including the Root Mean Square Error (RMSE), Sum of the absolute error (SAE), Average relative error (ARE), Chisquare statistic test (χ2) and the correlation coefficient (R^2) were conducted between the experimental and predicted values. The statistical analyses were performed according to the following equations [48, 49]:

$$RMSE = \sqrt{\frac{1}{p}\sum_{q=1}^{p}\left[\left(\frac{C}{C_0}\right)_q^{exp} - \left(\frac{C}{C_0}\right)_q^{predict}\right]^2} \quad (01)$$

$$SAE = \sum_{i=1}^{n}\left|\left(\frac{C}{C_0}\right)_{predict} - \left(\frac{C}{C_0}\right)_{exp}\right|_i \quad (02)$$

$$X^2 = \sum_{i=1}^{n}\left[\frac{\left(\left(\frac{C}{C_0}\right)_{predict} - \left(\frac{C}{C_0}\right)_{exp}\right)^2}{\left(\frac{C}{C_0}\right)_{predict}}\right]_i$$

$$R^2 = 1 - \frac{\sum_q^p\left[\left(\frac{C}{C_0}\right)_q^{exp} - \left(\frac{C}{C_0}\right)_q^{predict}\right]^2}{\sum_q^p\left[\left(\frac{C}{C_0}\right)_q^{exp} - \left(\frac{C}{C_0}\right)_n\right]^2}$$

Where, q is the number of data points, $\left[\frac{C}{C_0}^{exp}\right]$ is the experimental reduced concentration and, $\left[\frac{C}{C_0}^{predict}\right]$ is the reduced concentration predicted by the model.

3. Results and discussion

3.1 Textural characterization of the prepared dried activated sludge

The scanning electron micrograph in (Schema3) exemplifies the surface morphology of aerobic activated sludge. From the SEM micrograph, it is evident that the surface of dead biomass particles exhibited an irregular and rough structure with high heterogeneity. The sorption process is likely to involve various sorption mechanisms due to the complex nature of the surface morphology.Reminding that this dried activated sludge was previously used by Cherifi. et *al*. [38] in order to study the batch adsorption of methylene blue, and in this work it elaborated for dynamic adsorption. The FT-IR spectrum of biomass (Schema4) obtained by Cherifi et al. [38] exhibits notable peaks within the range of 3,753.6 to 3,440.9 cm^{-1}, indicating the presence of functional groups such as OH, NH, and NH2. An asymmetric vibration of CH is observed at 2,926.11 cm^{-1}, while a symmetric vibration of CH occurs at 2,860.53 cm^{-1}. The presence of carboxylic acids is indicated by the peak at 2,382.17 cm^{-1}. The stretching vibration of C=O and NH peptidic bond of proteins is observed at 1,641.48 and 1,552.75 cm^{-1}, respectively. The phenolic OH and CO stretching are identified at 1,433.16 cm^{-1}. The band at 1,037.74 cm^{-1} corresponds to the vibration of C–O–C in polysaccharides. [38].

3.2 Effect of the initial concentration of MB

The initial concentration plays a crucial role in overcoming the overall mass transfer resistance experienced by molecules as they traverse both the aqueous and solid phases [50]. In order to assess the impact of the initial concentration on the adsorption of MB in an aqueous solution in contact with dried activated sludge, various initial concentrations C_0 (50, 100, and 300 mg/L) were investigated at pH 7.5, with a bed height (H) of 3.5 cm, flow rate (Q) of 5 mL/min, adsorbent mass of 10 g, and at ambient temperature. The results of these experiments, as depicted in (Schema 5), demonstrate that the breakthrough curve became steeper as the initial concentration decreased. The highest removal efficiency was achieved at an initial concentration of 50 mg/L.

3.3 Effect of the bed height

To examine the influence of bed height on the breakthrough curve, the impact of this parameter was investigated at bed heights of 2.5 cm, 3.5 cm, and 5 cm corresponding to 8g, 10 g and 15g of adsorbent respectively, while keeping other factors constant, including an initial concentration of 50 mg/L, a flow rate of 5 mL/min, a pH of 7.8, and ambient temperature. The results of this investigation are presented in (Schema 6), illustrating the performance of the breakthrough curves. It is observed that the breakthrough time, bed exhaustion time, and dye removal efficiency all increased as the bed height increased. Notably, at a height of 5 cm, the saturation of the sludge occured at a slower rate compared to a height of 3.5 cm. From schema 6, the breakthrough time of BM increased from 230 to 380 minutes, while the exhaustion time increased from 620 to 1000 minutes. These results indicate that a bed depth of 5 cm provided an optimum breakthrough curve with longer use duration. The reason is likely that a greater bed height increases the specific surface area of the dried activated sludge [51]. Consequently, more liquid volume could be treated, leading to longer breakthrough and exhaustion times [52].

3.4 Effect of the flow rate

The flow rate is a crucial parameter when assessing the performance of an adsorption process, especially in the continuous treatment of wastewater on an industrial scale [53]. To investigate the impact of the flow rate on the adsorption of MB, the following values were examined: 2.5 mL/min, 5 mL/min, and 7 mL/min, while maintaining a constant initial dye concentration (50 mg/L), bed height (3.5 cm), and adsorbent mass of 10g. The effect of flow rate on breakthrough performance under the aforementioned operating conditions is depicted in (Figure 7). It can be observed that the length of the adsorption zone decreased as the flow rate increased. Additionally, the percentage removal of the dye decreased as the flow rate increased from 2.5 mL/min to 7 mL/min. The accelerated movement of compounds at higher flow rates leads to a shorter residence time of the dye solution in the column. Consequently, there is insufficient time for effective interaction between the dye and the adsorbent, impeding the binding of dye molecules. As a result, molecules tend to exit the column before equilibrium is attained. This trend aligns with findings reported by Uddin et al. [54] for fixed-bed adsorption of methylene blue using Artocarpusheterophyllus (jackfruit) leaf powder, as well as by Chowdhury et *al*. [49] for fixed-bed adsorption of methylene blue by NAOH-modified rice husk and by Julin Cao et *al* [52] for dynamic adsorption of anionic dyes by apricot shell activated carbon.

3.5 ANN predictive model

ANN is a valuable and potent modeling tool for constructing predictive models. In this particular study, the quasi-Newton BFGS algorithm was employed to create a nonlinear model. Three-layer neural networks (NNs) were utilized, with the input layer consisting of five neurons corresponding to the number of input parameters. A single hidden layer was employed, with the number of neurons ranging from 3 to 20, and the output layer contained one neuron representing the reduced concentration. The optimized ANN model exhibited a structure that approached an R value of 1 and an RMSE value approaching 0.The optimal architecture was determined to be an ANN structure with 12 neurons in the hidden layer. (Schema8), generated using Origin Pro 8 software, it illustrates the comparison between experimental values and predicted values obtained from the optimized ANN model for the entire database, training phase, validation phase and testing phase . For the total database, the results demonstrated a remarkable agreement between the experimental data and the predictions made by the ANN model, as evidenced by the high correlation coefficient (R = 0.99) and the low RMSE value (RMSE = 0.018). The parameters of the optimal ANN model can be found in Table 3, providing further insights into its configuration. [R], RMSE, SAE and X^2 statistic test were carried out between experimental data and predicted data for total database, training phase, testing and validation phase. The results are given in Table 4. The results proved the robustness of ANN developed model and the high ability of the ANN to predict the dynamic adsorption of methylene blue onto dried activated carbon with satisfactory correlation coefficients for all database and phases approaching to 1 and statistical errors close to 0.

3.6 Sensitivity analysis study

In order to study the importance of each parameter for the prediction of dynamic adsorption of methylene blue, a sensitivity analysis was conducted using STATISTICA software. This method, proposed by Garson [55] then taken by Goh [56], indicates a quantification of the relative importance of different inputs parameters on the output of the NN. The significance of descriptors in the ANN model is presented in Schema 9.

Table 1. *Range of values for the variables.*

Parameters	Values
Initial concentrations	**50-100-300 mg/L**
Bed heights	**2.5- 3.5- 5 Cm**
Flow rates	**2.5 -5-7 mL/min**

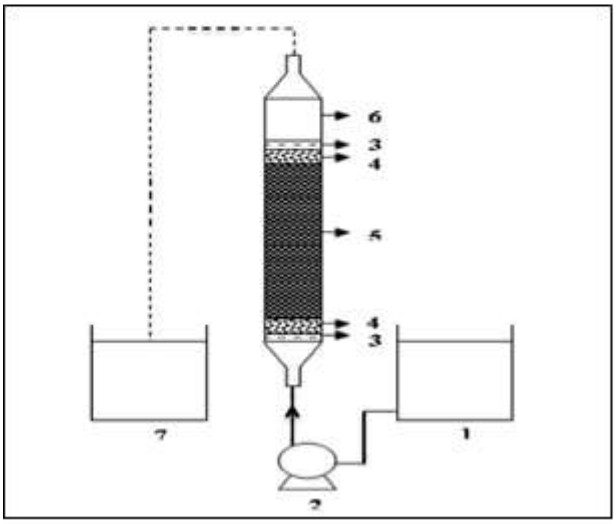

Figure 1. *Experimental dispositive*

Table 2. *ANN input parameters*

	Parameters	Unit	Minimum	Maximum
	Time (t)	mn	0	1210
	mass of adsorbent(m)	g	8	15
Inputs	Bed height(H)	Cm	2.5	5
	Initial concentration(C_0)	mg/L	50	300
	volume flow (Q)	mL/min	2.5	7
Output	reduced concentration (C/C_0)	-	0	1

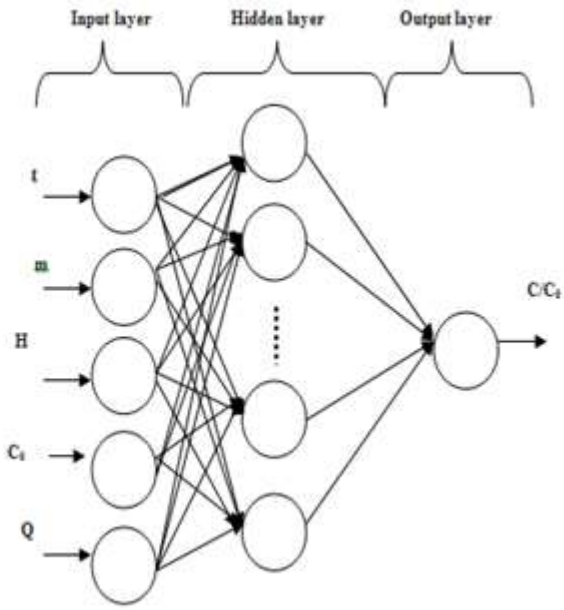

Figure 2. *ANN developed structure*

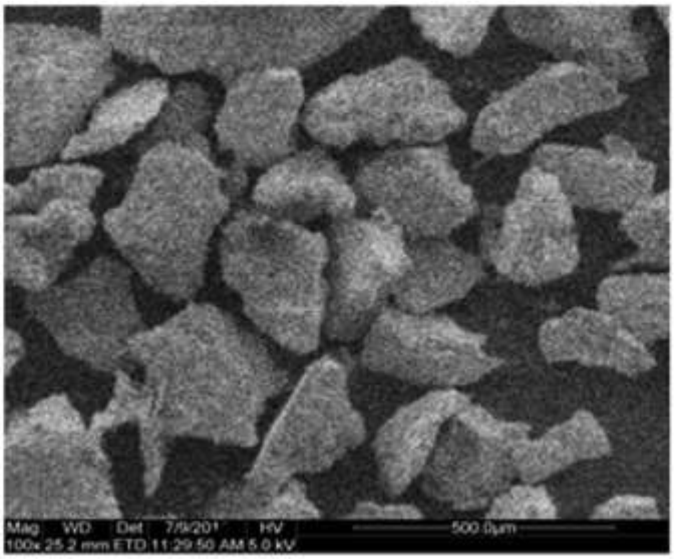

Figure 3. *The SEM image of dried activated sludge [38].*

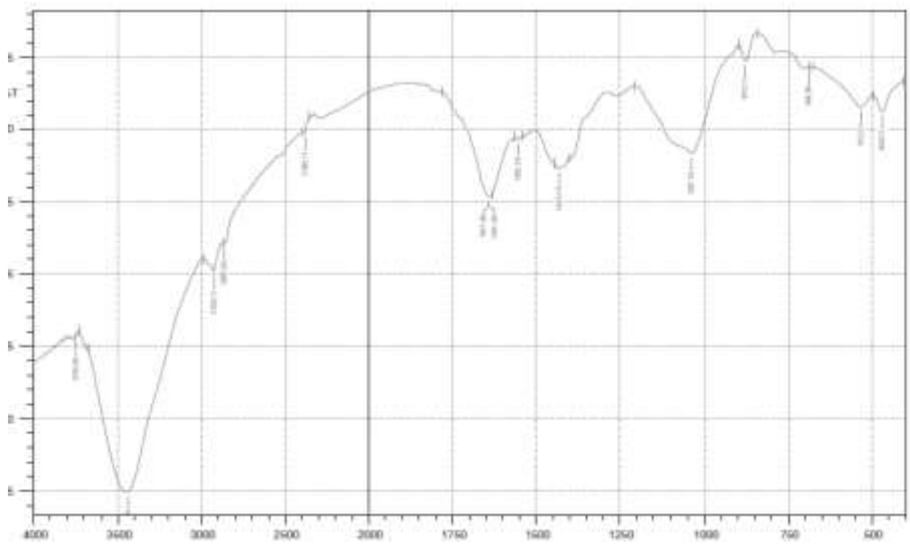

Figure 4. *FT-IR spectra of waste activated sludge [38]*

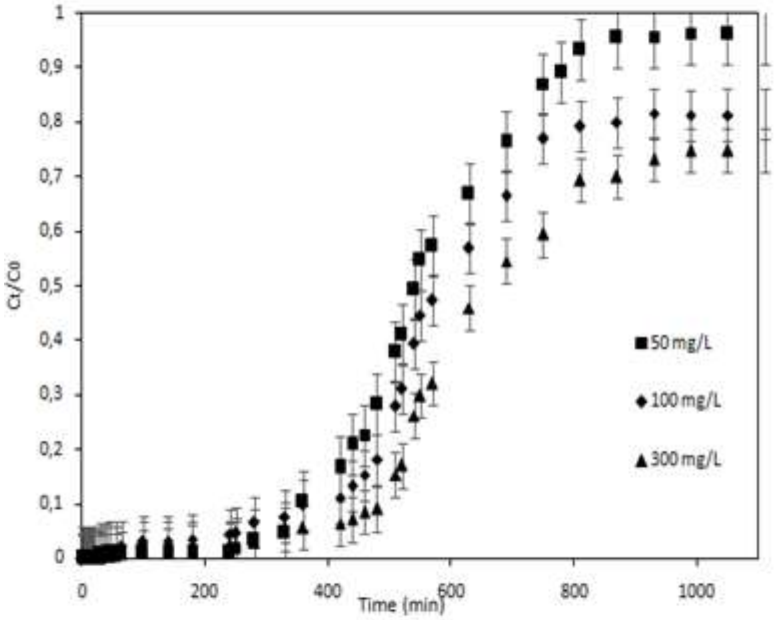

Figure 5. *Effect of initial concentration on breakthrough curve for fixed-bed adsorption of MB by dried activated sludge (pH= 7.98, H= 3.5 cm, Q= 5mL/mn, m=10g)*

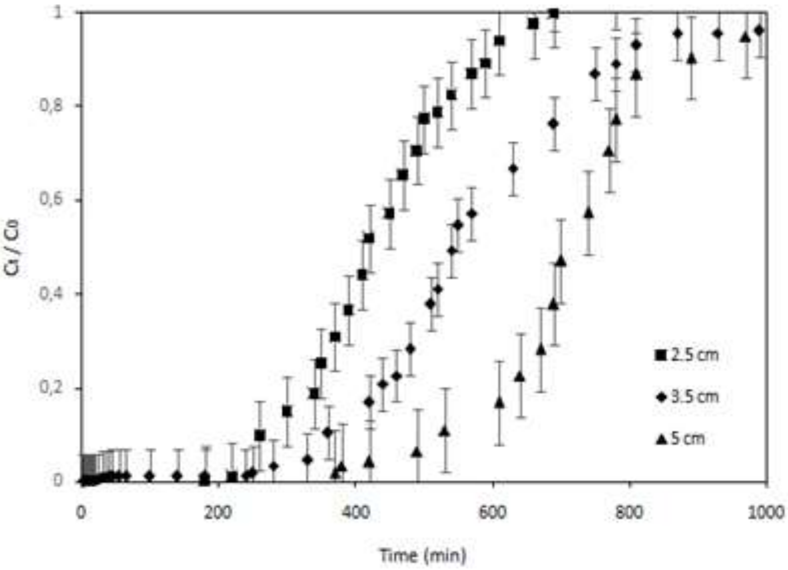

Figure 6. *Effect of bed height on the breakthrough curve for fixed-bed adsorption of MB by dried activated sludge (C_0= 50 mg/L, pH= 7.98, Q= 5mL/min)*

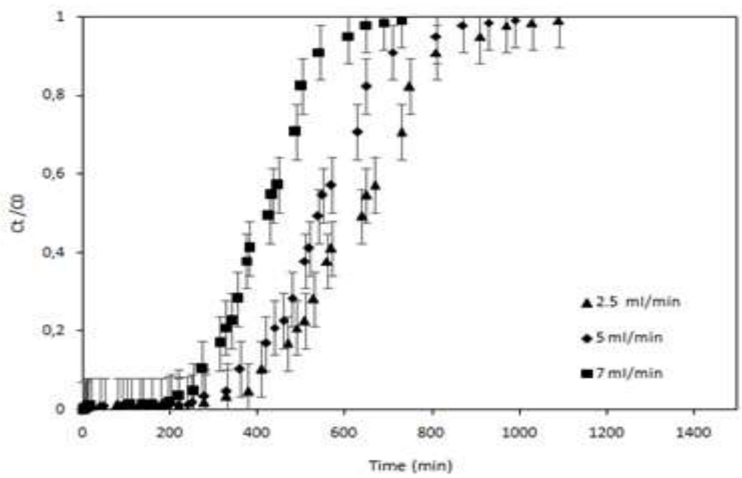

Figure 7. *Effect of flow rate on the breakthrough curve for fixed-bed adsorption of MB by dried activated sludge (C_0= 50 mg/L, pH= 7.98, H= 3.5 Cm, m=10g)*

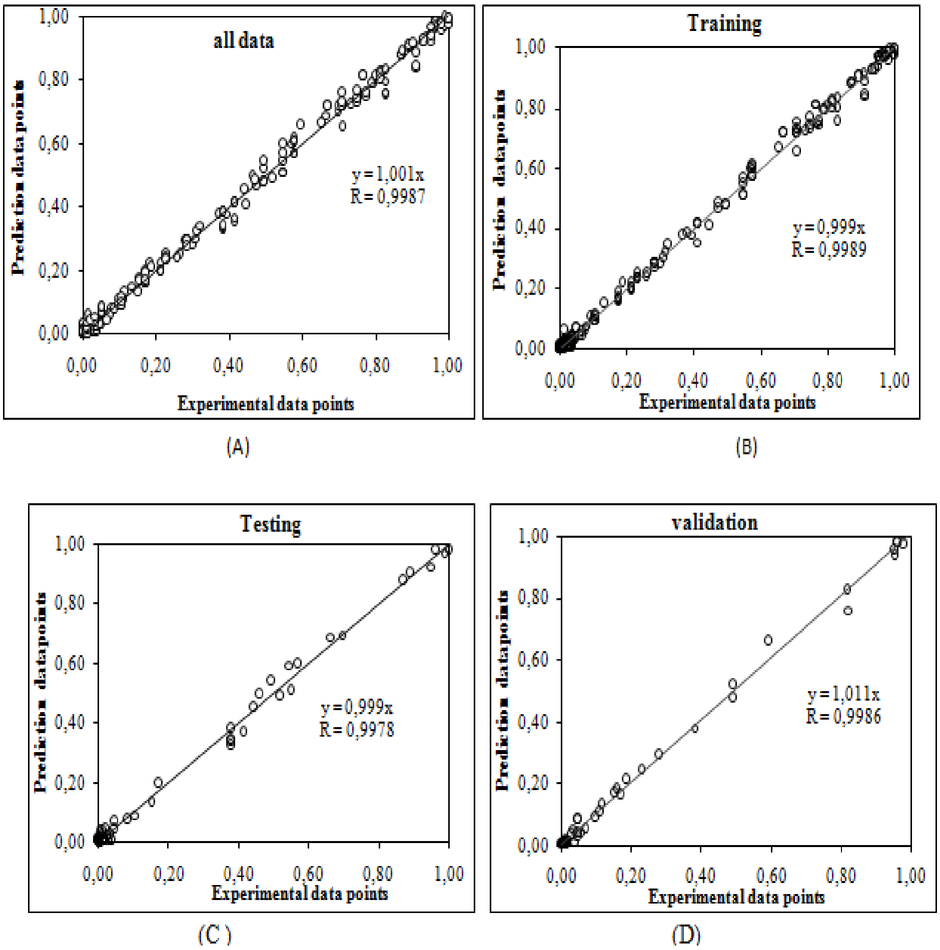

Figure 8. *Comparison between experimental and predicted data for: A: the total data set, B: the training phase, C: the testing phase, D: the validation phase.*

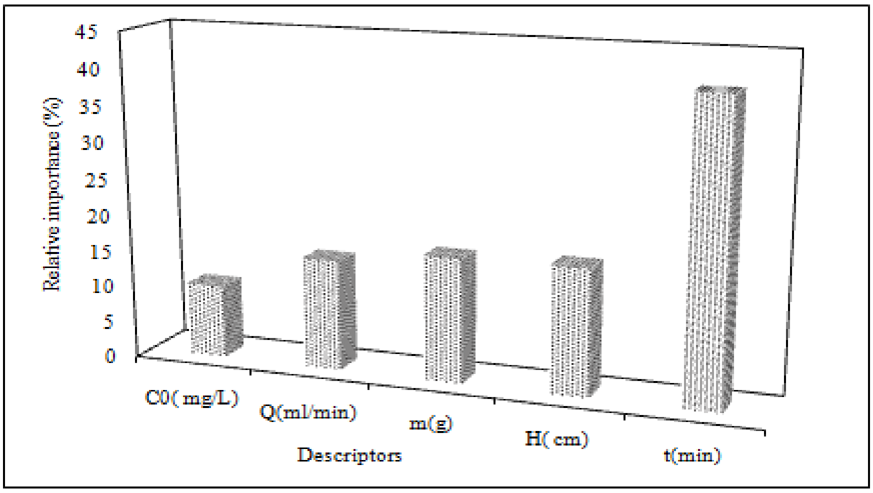

Figure 9. *Relative importance of the descriptors to the ANN model*

Table 3. *Optimal ANN optimal parameters*

Number of input layer	1
Number of hiddenlayer	1
Number of output layer	1
Number of input neurons	5
Number of hidden neurons	12
Number of output neurons	1
Transfer function of the	Tanh

hidden layer	
Transfer function of the Output layer	identity
Training algorithm	BFGS

Table 4. *Error function assessment for comparison between experimental and predicted data*

Error function	R	RMSE	SAE	X^2
Total database	0.9987	0.018	0.044	0.0121
Training phase	0.9989	0.0166	0.028	0.0064
Validation phase	0.9986	0.0196	0.0073	0.001
Testing phase	0.9978	0.021	0.008	0.0038

4. Conclusions

This study aimed at exploring the continuous biosorption of methylene blue (MB) on dried activated sludge. The research focused on investigating the impact of initial MB concentration, flow rate, and bed height. By reducing the flow rate and increasing the bed height, the breakthrough time was observed to be delayed at an initial concentration of 50 mg/L. To analyze the experimental data, an Artificial Neural Network (ANN) model was employed. Extensive error analysis was conducted to compare the experimental and predicted data. The findings demonstrated an excellent agreement between them, as evidenced by a high correlation coefficient (R = 0.99) and a root mean square error (RMSE), Average, Sum of the absolute error (SAE) and Chisquare statistic test (X2) close to 0. These results validate the reliability of the developed ANN model for effectively removing MB through dried activated sludge, highlighting its strong affinity and potential for treating aqueous solutions contaminated with MB dye. The use of other intelligent predictive models such as neuro-fuzzy (Fuzzy Neural Network Inference System adaptive 'ANFIS') or PSO, and the use of other adsorbent/adsorbate systems either simple or complex such as heavy metals, organic compounds and inorganic in different phases and under different conditions are the perspectives which can be envisaged in the future. AI and deep learning are applied to different fields and reported in the literature [57-71].

Author Statements:

- **Ethical approval:** The conducted research is not related to either human or animal use.
- **Conflict of interest:** The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper

- **Acknowledgement:** The authors declare that they have nobody or no-company to acknowledge.

- **Author contributions:** 1- Meriem Sediri : Conceptualization, Methodology, Software, Writing original draft. 2- Radhia Yous : Prepared figures 3- Hakima Cherifi : Reviewed the manuscript and project administration.4- Mounia Louanes : Visualisation 5-Abdeltif Amrane : Reviewed the manuscript

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- **Data availability statement:** The data that support the findings of this study are available on request from the corresponding author. The data are not publicly available due to privacy or ethical restrictions.

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