



Optimization and modeling of a fixed-bed biosorption of textile dye using agricultural biomass from the Moroccan Sahara

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Received 29 May 2021; Accepted 7 August 2021

ABSTRACT

Textile manufacturing is one of the major industries that discharge an enormous quantity of dyes over the printing and dyeing process. Accordingly, the increasing demand for efficient and inexpensive treatment has enabled the innovation of low-cost alternative adsorbents. In this study, agricultural biomass from the Moroccan Sahara (ABMS) was investigated as an ecofriendly and low-cost biosorbent of textile dye. The effect of independent variables affecting the process fixed-bed adsorption such as inlet textile dye concentration (40, 80, and 120 mg L⁻¹), flow rate (2, 4, and 6 mL min⁻¹) and bed height (5, 10, and 15 mm), were modeled and evaluated by response surface methodology based on the Box–Behnken design. The kinetic models, Thomas and Yoon and Nelson model were applied to experimental data to predict the breakthrough curves using linear regression and to determine the characteristic parameters of the packed bed column. The data were in good agreement for both models with $R^2 > 0.95$. The maximum Methylene blue dye removal capacity was found to be 30.15 mg g⁻¹. These findings suggested that ABMS biosorbent without any activation in the column structure presents great potential in the removal of dyes from textile wastewater.

Keywords: Biosorption; Textile dye; Continuous system; Fixed-bed; Agricultural biomass; Moroccan Sahara

1. Introduction

The development of the Moroccan textile industry, which represents 31% of all Moroccan industries, is accompanied by high water consumption and increasing wastewater discharge [1]. Indeed, effluents of the textile industry are very complex mixtures of organic and mineral matter [2,3].

Thus, the discharge of these wastewaters into the receiving environment without any treatment causes potential damage to the environment in general and to human health in particular [4]. This is why the reduction of pollution at the source and the treatment of these effluents are considered an absolute necessity in many countries, knowing that around 80 countries suffer from water scarcity.

Several research works have shown that the discoloration of textile effluents is possible using different techniques

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such as biological treatment [5], advanced oxidation processes [6], photocatalytic degradation [7], membrane technology [8] and coagulation–flocculation [9]. Indeed, these textile wastewater treatment processes have not proven to be cost-effective and some of these treatment processes also involve the use of excessive chemicals which subsequently cause additional environmental problems.

The adsorption process [10] is a relatively simple technique and is often used in the treatment of industrial wastewater. It has the advantage of adapting to a wide range of effluents, with low energy consumption and without the formation of dangerous by-products. Activated carbon is the most widely used adsorbent for color reduction, but it is very costly and requires regeneration [11].

Recently, various studies have focused on the use of renewable biomass as an alternative solution to solve these problems such as palm waste [12], peanut husk [13], activated artichoke leaves [14], activated fallen leaves of *Ficus racemosa* [15], wheat straw [16], potato plant wastes [17], activated coconut shell [18], agriculture waste corncob [19], chitosan composites [20], alginates [21], functionalized cellulose nanocrystals [22], activated *Prunus dulcis* leaves [23] have been used to remove dyes from wastewater.

The literature analysis established that researchers have not used agricultural biomass based on a prickly pear from the Moroccan Sahara, for biosorbent preparation in raw form without any activation and subsequent application for the removal of cationic dye, Methylene blue (MB) under continuous mode has also not been investigated. However, Methylene blue is a cationic dye widely used for dyeing paper, leather, plastic, wood and particularly textiles, in particular denim, a cotton fabric used for making jeans. Moreover, the advantage of our biosorbent over other biosorbents is it's available free of charge and in large quantities, especially in the southwestern region of Morocco. However, the cactus sector in Moroccan Sahara has undergone a remarkable transformation by relying on biotechnology through the valorization of this agricultural product in canned products and cosmetic products [24]. Indeed, this agro-industry produces a lot of biowastes which must be managed by sustainable methods [25].

The objective of this work is to enhance this agricultural biomass from the Moroccan Sahara (ABMS) in order to prepare an eco-friendly and low-cost biosorbent for the removal of MB dye from wastewater under continuous mode. The effect of continuous column operations was also investigated to establish breakthrough conditions using response surface methodology.

2. Materials and methods

2.1. Adsorbent and adsorbate

The adsorbent used in this study was prepared from prickly pear waste. This agricultural biomass was collected from agricultural cooperatives in the provinces of the Moroccan Sahara and more particularly the coastal areas of the region of Guelmim–Oued Noun.

The general procedure for the preparation of our biosorbent was as follows. First of all, agricultural waste was washed several times with boiling distilled water. Secondly,

they were dried in the open air and then in an oven at 50°C until a constant mass was obtained. Third, they were cut into small pieces, crushed and sieved. Finally, the dry adsorbent named ABMS was stored in a desiccator for further study without any chemical or physical activity that causes loss of energy and mass and moreover the use of a large amount of water for rinsing.

In this study, we have chosen Methylene blue (MB) as the model pollutant of the textile industry. Indeed, MB is widely used in dyeing and printing processes during textile manufacturing. Methylene blue was supplied by Sigma-Aldrich Company (USA) and was used as received without any prior purification.

The Methylene blue stock solution was prepared by dissolving 1 g of this dye without further purification in double-distilled water. The experimental solutions were obtained by diluting this stock solution in accurate proportions to needed inlet concentrations.

2.2. Fixed-bed experiments

Methylene blue biosorption experiments by agricultural biomass from the Moroccan Sahara in the continuous mode were carried out using a column with an internal diameter of 20 mm having a layer of glass wool at the bottom. The colored Methylene blue solution was pumped using a peristaltic pump as shown in Fig. 1.

Once the colored solution of initial concentration C_i is pumped, the dye is adsorbed on a packed bed filled with ABMS biosorbent. After each time, samples of Methylene blue at the outlet of the column were taken and analyzed using a UV-Vis spectrophotometer type Shimadzu (Japan) at λ equal to 664 nm.

The sorption front moves gradually down towards the saturation zone, arriving at the lower end of the packed bed. At that point, the effluent concentration at the outlet of the stack gets closer and closer to the initial concentration, which signifies the saturation point of the stack. In fixed-bed adsorption, the break-through time (t_b) is the time

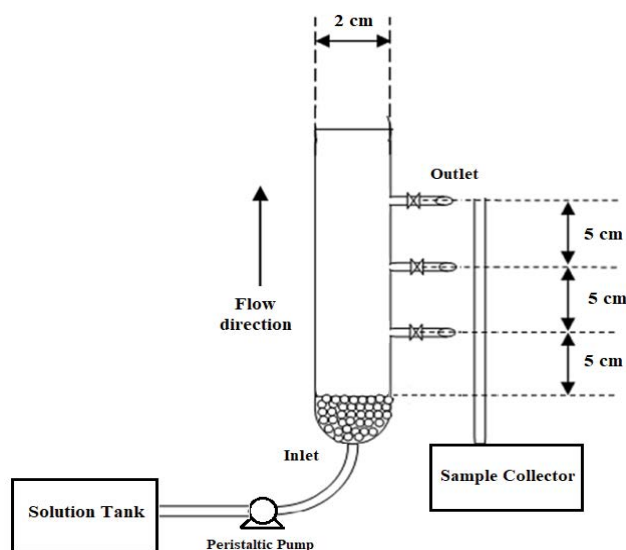


Fig. 1. Fixed-bed experiments.

when the effluent concentration at the column outlet is 5% of the initial concentration. While the exhaustion time (t_e) is established when the effluent concentration is above a level of between 90% and 95% of the initial concentration.

2.3. Response surface methodology

The most widely used and most general model for adjustment in the methodology of the response surfaces is the Box–Behnken design [26]. In this design, a few points of the design matrix are needed for experimental modeling with efficient estimation of first and second-order coefficients. The expected Box–Behnken design answers were computed using the second-order polynomial formula:

$$Y = \beta_0 + \sum_{i=1}^n \beta_i X_i + \sum_{i=1}^n \beta_{ii} X_i^2 + \sum_{i=1}^n \sum_{j=1}^n \beta_{ij} X_i X_j + \epsilon \tag{1}$$

where the predicted response is represented by Y ; the coefficients of the intercept, linear, quadratic and interaction terms among independent factors (X) are indicated respectively by β_0 , β_i , β_{ii} and β_{ij} ; and the model error is denoted by ϵ .

3. Results and discussion

3.1. Box–Behnken design

In this study, we used the Box–Behnken design with three levels per factor to find the optimal fixed-bed adsorption conditions of Methylene blue using agricultural biomass from the Moroccan Sahara. Based on data from the literature review [27–29] and on preliminary tests, three factors were chosen as independent variables to predict the dye removal efficiency (Y), are the inlet dye concentration (X_1), the feed flow rate (X_2) and the bed height (X_3). The experimental ranges and levels of independent variables are reported in Table 1.

During non-exceptional rainfall events, the optimization of wastewater treatment is a complex matter that will probably only be completed in the long or very long term. The aim is to minimize the impact of discharges on the natural environment, which is currently often very roughly assessed by a few parameters. The design matrix with fourteen experiments for 3 factors and 3 levels that were carried out and their responses notified, shown in Table 2.

According to the data in this table, it is observed that the three-level Box–Behnken design necessitates exact values for each variable at the low (–1), medium (0), and high (+1) levels, namely: 40, 80, and 120 mg L^{–1} for the initial concentrations of Methylene blue aqueous solution; 2, 4, and

6 mL min^{–1} for the influent flow rate; and 5, 10, and 15 mm for the adsorbent bed height.

The software Statgraphics Plus was used to create a Box–Behnken design matrix. The second-order polynomial equation for the predicted responses in terms of coded independent factors was deduced by fitting the experimental data. In this work, the relationship between independent variables and response was drawn by a second-order polynomial equation.

The coefficients of the regression equation (β_0 , β_i , β_{ii} and β_{ij}) were calculated easily calculated by the least-squares method and data were fitted to second-order polynomial equations for dye removal efficiency [30].

The final empirical model of the dye removal efficiency rate by fixed-bed biosorption using ABMS biosorbent, in terms of coded factors after exclusion of non-significant terms, is presented in Eq. (2):

$$Y = 72.15 + 14.94X_1 - 16.53X_2 + 12.35X_3 - 16.95X_1X_1 - 8.88X_3X_3 \tag{2}$$

Based on the second-order polynomial equation of the response surface methodology, the effect of independent variables affecting the process of fixed-bed adsorption on the response (Y) was analyzed.

Table 2
Box–Behnken design matrix

Runs	Coded units of variables X_i			Response Y
	X_1	X_2	X_3	
1	–1	–1	0	44.67
2	1	–1	0	81.42
3	–1	1	0	23.46
4	1	1	0	47.18
5	–1	0	–1	21.08
6	1	0	–1	44.16
7	–1	0	1	42.02
8	1	0	1	78.02
9	0	–1	–1	68.26
10	0	1	–1	24.28
11	0	–1	1	84.67
12	0	1	1	51.83
13	0	0	0	72.14
14	0	0	0	72.06

Table 1
Study field and coded factors

Factors	Unit	Levels of coded variables X_i		
		Low (–1)	Center (0)	High (+1)
$X_1 = C_i =$ Inlet dye concentration	mg L ^{–1}	40	80	120
$X_2 = Q =$ Feed flow rate	mL min ^{–1}	2	4	6
$X_3 = Z =$ Biosorbent bed height	cm	5	10	15

The significance of effects can be estimated by comparing the *F* distribution of the experimental values to a critical value ($F_{0.05}(1;4) = 7.71$) according to the results shown in Table 3.

In this case, the linear terms (X_1 , X_2 and X_3) and the squared terms (X_1X_1 and X_3X_3) were significant model terms whereas the squared terms (X_2X_2) and the interaction terms (X_1X_2 , X_1X_3 and X_2X_3) were insignificant to the response.

The analysis of variance (ANOVA) for a model of the dye removal efficiency rate by fixed-bed biosorption using agricultural biomass is reported in Table 4.

From the results of ANOVA, we notice that the model *F*-value is greater than the critical value $F_{0.01}(9;4)$ by 1%,

which implies that the regression is globally significant at a confidence level of 99% for this model. The influence of each independent variable on the dye removal efficiency is shown in Fig. 2.

From this figure, it can be seen that the flow rate (X_2) has a negative effect on the dye removal efficiency (*Y*). However, the initial concentration of Methylene blue (X_1) and the height of the bed (X_3) have a positive effect on the response, but looking at this figure, we see that they have a negative effect in the positive area, which can be explained by the negative effect of the interaction of the squared term.

Response surface plots were prepared for the purpose of evaluating the effect of each two parameters influencing

Table 3
Experimental design and data analysis

Terms	Coefficient	Sum of squares	Df	Mean square	F_{exp}	<i>p</i> -value	Significance test
b_1	14.944	1,786.53	1	1,786.53	99.23	0.0006	***
b_2	-16.534	2,186.92	1	2,186.92	121.47	0.0004	***
b_3	12.345	1,219.19	1	1,219.19	67.72	0.0012	***
b_{11}	-16.929	919.775	1	919.775	51.09	0.0020	***
b_{22}	-5.989	115.729	1	115.729	6.43	0.0643	NS
b_{33}	-8.851	252.121	1	252.121	14.00	0.0201	**
b_{12}	-3.258	42.4452	1	42.4452	2.36	0.1995	NS
b_{13}	3.230	41.7316	1	41.7316	2.32	0.2025	NS
b_{23}	2.785	31.0249	1	31.0249	1.72	0.2595	NS

****p* ≤ 0.01; ***p* ≤ 0.025; **p* ≤ 0.05; NS: No significant.

Table 4
Analysis of variance

Source of variation	Sum of squares	Df	Mean square	F_{exp}	$F_{0.01}(9;4)$	Significance test
Regression	6,337.9255	9	704.2139	39.12	14.66	***
Residue	72.0145	4	18.0036			
Total	6,409.94	13				

****p* ≤ 0.01; ***p* ≤ 0.025; **p* ≤ 0.05; NS: No significant.

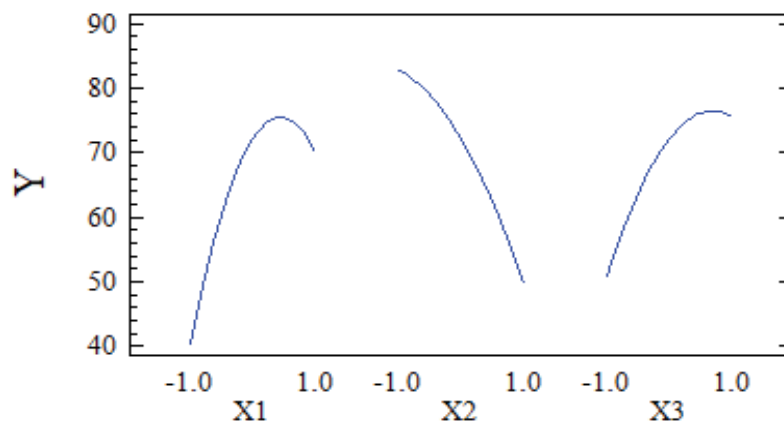


Fig. 2. Main effects plot for dye removal efficiency.

the phenomenon of fixed bed absorption of Methylene blue by keeping the third operating variable constant, as shown in Fig. 3.

Fig. 3A shows the interaction between the initial concentration of Methylene blue (X_1) and feed flow rate (X_2) at a constant value of the bed height ($X_3 = 0$). Interaction between the initial concentration of Methylene blue (X_1) and bed height (X_3) at a constant value of the feed flow rate ($X_2 = 0$) is shown in Fig. 3B. Fig. 3C shows the interaction between feed flow rate (X_2) and bed height (X_3) at a constant value of the Inlet dye concentration ($X_1 = 0$).

From these response surface plots, we can conclude that when the initial concentration of Methylene blue and the agricultural biomass bed height increase simultaneously or when the initial concentration of adsorbate increases and the bed height remains unchanged, the discoloration efficiency rate increases until an optimum which remains unchangeable in the experimental study area. On the contrary, for interactions involving the feed flow rate, the discoloration efficiency rate decreases until a minimum is obtained.

3.2. Column studies

In this work, the study of the operating conditions that control the sorption capacity of MB dye on ABMS biosorbent in continuous mode was carried out by passing an influent dye concentration (C_i) at a flow rate (F) through a biosorbent bed height (Z). Indeed, the breakthrough time is a very important factor in evaluating the operation of the column. Generally, a higher breakthrough time value indicates a higher biosorption capacity of the column.

3.2.1. Effect of biosorbent bed height (Z)

To study the effect of biosorbent bed height on the fixed bed adsorption column operation parameters, column

experiments were carried out for different heights at 5, 10 and 15 cm by passing an MB dye solution with an initial concentration of 80 mg L^{-1} and at 4 mL min^{-1} flow rate. The experimental data for the various breakthrough parameters such as breakthrough time (t_b), exhaustion time (t_e) and maximum uptake capacity (q_m) are described in Table 5.

From this table, it is noted that the increase in the bed height (5 to 15 cm) causes both the increase in the breakthrough time (120 to 250 min) and the biosorption capacity of ABMS biosorbent (22.48 to 26.81 mg g^{-1}). This result could be due to the fact that by increasing the height of the bed, the contact time of the dye inside the column will be increased. Moreover, the higher bed heights have a greater amount of biosorbent, which gives more contact time for the adsorption of the dye molecules to the biosorbent. A similar effect of biosorbent bed height has been reported for the adsorption of acid violet 17 dye using biosorbent obtained from NaOH and H_2SO_4 activation of fallen leaves of *Ficus racemosa* [31].

3.2.2. Effect of dye concentration (C_i)

The effect of dye concentration was achieved via column experiments by passing influent dye at 4 mL min^{-1} flow rate on ABMS biosorbent at 10 cm bed height with varying initial concentrations MB dye (40, 80, and 120 mg L^{-1}). The experimental data for the various breakthrough parameters are also reported in Table 5.

From this table, it can be seen that the increase in the dye concentration ($40\text{--}120 \text{ mg L}^{-1}$) causes the increase in the biosorption capacity of the ABMS biosorbent ($22.12\text{--}30.22 \text{ mg g}^{-1}$), on the other hand, the decrease in the breakthrough time (210–130 min). This result could be due to the fact that solutions of higher initial dye concentration have a greater driving force for mass transfer subsequently

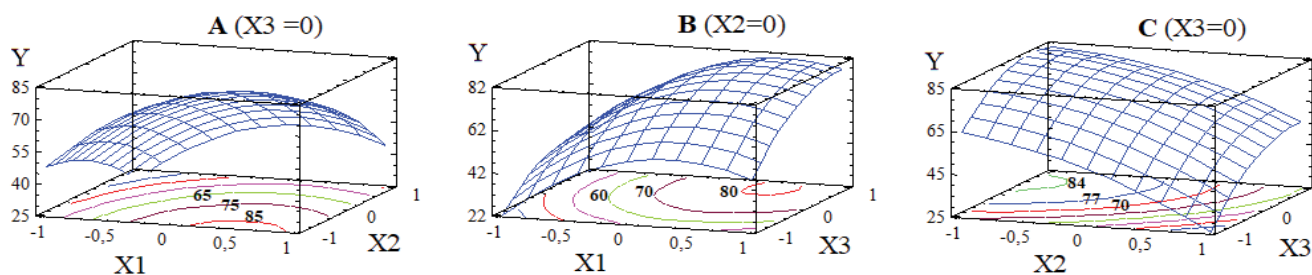


Fig. 3. Response surfaces for discoloration efficiency.

Table 5
Breakthrough parameters

C_i (mg L^{-1})	Q (mL min^{-1})	Z (cm)	t_b (min)	$t_{50\%}$ (min)	t_e (min)	q_m (mg g^{-1})
40	4	10	210	480	590	22.12
80	4	10	190	440	550	23.81
120	4	10	130	300	390	30.22
80	2	10	230	500	620	24.96
80	6	10	140	320	410	23.77
80	4	5	120	280	360	22.48
80	4	15	250	540	640	26.81

resulting in increased biosorption capacity. A similar effect of dye concentration has been observed for continuous fixed-bed adsorption of direct deep blue dye using glucose carbon composite-based biosorbents [32].

3.2.3. Effect of flow rate (Q)

The study of the effect of inlet influent flow rate was carried out via column experiments by passing an MB dye solution at an initial concentration of 80 mg L⁻¹ on ABMS biosorbent at 10 cm bed height with varying inlet MB dye flow rate (2, 4, and 6 mL min⁻¹). The experimental data for the maximum uptake capacity, breakthrough time and exhaustion time are also mentioned in Table 5.

From this table, it is noted that the increase in the inlet influent flow rate (2–6 mL min⁻¹) causes both the decrease in the breakthrough time (230–140 min) and the biosorption capacity of ABMS biosorbent (24.96–23.77 mg g⁻¹). This result could be due to the fact that the contact time of the dye molecules in the column at higher flow rates, is not sufficient and the dye leaves the column before equilibrium is established, resulting in subsequently a decrease in the breakthrough time and therefore in the biosorption capacity. A similar effect of inlet influent flow rate has been reported for the adsorption of Acid Green 25 dye using activated *Prunus dulcis* as biosorbent [33].

3.3. Modeling of column data

In continuous packed bed adsorption processes, the liquid phase and solid phase concentration vary both spatially and with time, so the design and optimization of fixed bed columns are particularly difficult if we do not have a quantitative approximation model.

Various theoretical models have been developed to describe the equilibrium and breakthrough curves of fixed bed adsorption [34]. The Thomas model and the Yoon and Nelson model have been widely used to fit experimental continuous adsorption data.

The Thomas adsorption model is the most widely used theoretical model to describe column performance. The Thomas model [35] can be written as follows:

$$\frac{C_t}{C_i} = \frac{1}{1 + \exp\left(\frac{k_T q_T W}{Q} - k_T C_i t\right)} \quad (3)$$

The Thomas linear equation is described by the following equation:

$$\ln\left(\frac{C_i}{C_t} - 1\right) = \frac{k_T q_T W}{Q} - k_T C_i t \quad (4)$$

where C_i is the influent concentrations (mg L⁻¹); C_t is the effluent concentrations (mg L⁻¹); k_T is the Thomas rate constant (mL mg⁻¹ min⁻¹); q_T is the maximum capacity of adsorption (mg g⁻¹); W is the weight of adsorbent (g); Q is the feed flow (mL min⁻¹); t is the flow time (min).

Yoon and Nelson have developed a relatively simple model. This model assumes that the rate of decrease in likely adsorption for each adsorbate molecule is proportional to the breakthrough of the adsorbent. The Yoon–Nelson model [36] is written as follows:

$$\frac{C_t}{C_i - C_t} = \exp(k_Y t - \tau k_Y) \quad (5)$$

The Yoon–Nelson linear equation is described by the following equation:

$$\ln \frac{C_t}{C_i - C_t} = k_Y t - k_Y \tau \quad (6)$$

where k_Y is the rate constant (min⁻¹) and τ is the time required for 50% adsorbate breakthrough (min).

Linear Eqs. (4) and (6) were used to estimate the parameters for the Thomas model (k_T and q_T) and the Yoon–Nelson model (k_Y and τ.). The values of these parameters and the correlation coefficients (R²) are presented in Table 6.

From Table 6, it can be seen that the values of biosorption capacity calculated by the Thomas model are on the whole very close to those obtained experimentally.

Also, it is noted that the time required for 50% adsorbate breakthrough calculated by Yoon–Nelson are comparable to those obtained experimentally.

It was also seen from Table 6 that correlation coefficient, R² values for the Thomas and Yoon–Nelson models were closer to 1 with a confidence level greater than 95% for both models.

All of these results confirmed a better fit of the Thomas and Yoon–Nelson models to the obtained column data.

Table 6
Thomas and Yoon–Nelson model parameters

C _i (mg L ⁻¹)	Q (mL min ⁻¹)	Z (cm)	Thomas			Yoon–Nelson		
			k _T (mL mg ⁻¹ min ⁻¹)	q _T (mg g ⁻¹)	R ²	k _Y (min ⁻¹)	τ (min)	R ²
40	4	10	0.45	22.58	0.9837	0.0239	481.37	0.9722
80	4	10	0.32	24.12	0.9776	0.0251	442.13	0.9681
120	4	10	0.17	30.15	0.9822	0.0266	294.28	0.9745
80	2	10	0.26	25.36	0.9806	0.0235	496.22	0.9682
80	6	10	0.38	23.41	0.9793	0.0275	317.73	0.9706
80	4	5	0.36	22.84	0.9754	0.0268	278.25	0.9667
80	4	15	0.21	26.03	0.9843	0.0197	543.19	0.9812

3.4. Comparison with other adsorbents

The sorption capacity results with agricultural biomass from the Moroccan Sahara were compared with those of other adsorbents cited in the literature. Indeed, the maximum MB dye removal capacity under a continuous model by ABMS biosorbent was found to be 30.15 mg g⁻¹. Thus, the biosorption capacity of our biosorbent was higher than kaolin (20.06 mg g⁻¹) [27], also comparable to alginate-sugarcane bagasse-based composite (30.13 mg g⁻¹) [37] and slightly lower than citrus peel-alginate composite beads (31.45 mg g⁻¹) [38]. Moreover, the advantage of ABMS biosorbent which is easily regenerable using methanol as eluent. And also, reusable several times with a very slight decrease in adsorption capacity which can be compensated by some operational modifications. These findings suggested that this agricultural biomass without any activation in the column structure presents great potential in the removal of cationic dyes from textile wastewater.

4. Conclusion

In this study, the response surface methodology was used to optimize textile dye removal under continuous mode by agricultural biomass based on a prickly pear from the Moroccan Sahara. The interactions between operational process parameters for adsorption optimization, such as inlet dye concentration, feed flow rate and bed height were analyzed. Moreover, the interaction of model performance has been examined for statistical analysis. The resulting *p*-value of this model was less than 0.01, demonstrating that the surface response model established by the Box–Behnken matrix is globally significant at a 99% confidence level.

The optimal conditions for adsorption testing were established as shown previously. These results were then analyzed using a number of different kinetic models. The maximum MB dye removal capacity was found to be 30.15 mg g⁻¹. The correlation coefficient, *R*² values for the Thomas and Yoon–Nelson models were closer to 1. These two kinetics models were found to be suitably fitted to obtained column data. These findings suggested that ABMS biosorbent without any activation in the column structure presents a great potential in the removal of cationic dyes from textile wastewater.

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