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Modeling of the adsorption isotherm of methylene blue onto activated carbons derived from cotton (*Gossypium hirsutum*) residues

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ABSTRACT: The objective of this paper is to propose an adsorption isotherm model for methylene blue on composite activated carbons derived from cotton (*Gossypium hirsutum*) hulls and capsules. The adsorption mechanism involved was described using a three-parameter model that is intermediate between the Langmuir and Frundlich models and is characterized by its ease of use and physical interpretability. The experimental adsorption data were first adjusted using nine classical isotherm models, whose performance was evaluated using five statistical criteria: the adjusted coefficient of determination, the residual sum of squares, the Akaike information criterion (AIC), and the Bayesian information criterion (BIC). The models were best fitted in the following order: Sips> Toth> Redlich-Peterson> Khan>Langmuir> Frundlich> Jovanovich> Dubinin-Radushkevich. The proposed model provides the best fit with the experimental data for methylene blue adsorption on activated carbons derived from cottonseed shells and cotton (*Gossypium hirsutum*) capsules, with a determination coefficient of 0.9910 and a theoretical maximum adsorption capacity q_m of 820.86 $mg.g^{-1,}$ approximately double that predicted by classical models, illustrating an expected improvement in the predicted performance of the studied activated carbon.

KEYWORDS: modeling, isotherm, adsorption, activated carbon, dye, cotton.

1 INTRODUCTION

Adsorption is widely recognized as an efficient and attractive technique in water treatment, particularly for the removal of dyes from effluents generated by pharmaceutical, textile, agro-food, and paper industries [1,2]. Dyes present in industrial wastewater are often non-biodegradable organic compounds that can cause serious environmental pollution, adversely affect aquatic ecosystems, and deteriorate the quality of drinking water. Therefore, their removal prior to the discharge of industrial effluents into the environment is of critical importance [3,4]. Among the range of available treatment methods, the adsorption of micropollutants and trace metal elements [5,6] by activated carbons stands out, particularly due to their porous structure, large specific surface area, and also because they generally exhibit good affinity for several compounds [7,8]. In recent years, numerous studies have focused on the production of activated carbons from locally available lignocellulosic biomass [9–11] in a circular economy approach through the use of industrial residues. Most of these investigations aim to optimize the adsorption performance of newly developed materials [12]. Such optimization requires a thorough understanding of the adsorption mechanisms occurring at the adsorbent surface, which explains the extensive use of adsorption isotherm models in adsorption studies in order to estimate the consumables, including the adsorbents needed in an industrial contaminant removal process. In conventional studies dealing with pollutant adsorption onto activated carbons, several isotherm models are commonly employed [13] to describe and predict adsorption behavior under real conditions, design adsorption columns, or compare the performance of newly synthesized materials with existing ones. Among these models, the Langmuir [14], Freundlich, and Temkin isotherms are the most frequently used. Each adsorption model is based on specific assumptions. The Langmuir model assumes monolayer adsorption on a homogeneous surface with no interaction between adsorbed molecules, whereas the Freundlich model considers a heterogeneous surface with adsorption sites of different energies. Other models, such as Sips, Redlich–Peterson, and Toth, have also been proposed to reduce deviations between experimental data and theoretical predictions. In practice, researchers often face significant challenges when selecting the most appropriate isotherm model for their experimental data. The classical approach consists of fitting several documented models and identifying the one that best represents the adsorption system. However, the performance of these models is highly variable and depends on several factors, including the nature of the adsorbent, the type of pollutant, and the complexity of the adsorption mechanism. It is frequently observed that no single model can adequately describe all the specific features of a given adsorption system. To overcome this increasingly evident limitation, a new research trend has emerged, focusing on the development of novel adsorption isotherm models capable of accurately describing complex adsorption mechanisms [15–17]. Several approaches have been proposed, one of which consists of building new models based on existing ones. This strategy allows improved fitting accuracy while preserving the possibility of meaningful physical and chemical interpretation of the results. Within this context, the present study aims to propose a three-parameter adsorption isotherm model capable of describing dye adsorption onto activated carbons. The proposed approach is based on existing adsorption models. First, experimental data were fitted using nine isotherm models reported in the literature. Subsequently, a new model was developed by combining the characteristics of selected existing models, while maintaining simplicity in the final mathematical expression. The statistical criteria used for model evaluation include the correlation coefficient, adjusted coefficient of determination, residual sum of squares, Akaike Information Criterion (AIC), and Bayesian Information Criterion (BIC). To the best of our knowledge, this study represents the first attempt to simultaneously integrate these five statistical criteria into the methodology for selecting the most appropriate adsorption isotherm model for activated carbon systems, thereby enhancing the rigor of model performance evaluation.

The hypotheses of this study are as follows:

- The Langmuir and Freundlich models are unable to capture all aspects of the adsorption mechanism of methylene blue onto composite activated carbons derived from cotton shells and capsules;
- The coefficient of determination alone is insufficient for selecting the most appropriate adsorption isotherm model;
- The adsorption of methylene blue onto activated carbons derived from cotton shells and capsules involves a surface where homogeneous and heterogeneous adsorption sites coexist simultaneously.

This work therefore has a dual significance. Scientifically, it contributes to the diversification of existing modeling tools used to investigate adsorption phenomena. Environmentally, it contributes to the development of new, high-performance adsorbents materials from lignocellulosic waste.

2 MATERIAL AND METHODS

2.1 EQUILIBRIUM ADSORPTION ISOTHERM

At a fixed temperature, the adsorption isotherm study provides information on the amount of pollutant retained within the pores of activated carbon as a function of the equilibrium pollutant concentration. To this end, methylene blue solutions with different initial concentrations ranging from 100 to 700 ppm were prepared by dilution of a 1000 ppm stock solution. For each concentration, 100 mL of solution was transferred into a flask, to which 100 mg of activated carbon was added. The resulting suspension was agitated for four hours.

Previous kinetic experiments demonstrated that this contact time was sufficient to reach adsorption equilibrium. After four hours, agitation was stopped, and the residual solution was filtered. The absorbance of the filtrate was then measured at the maximum absorption wavelength of methylene blue (λ_max = 662 nm), previously determined using a UV–visible spectrophotometer.

The amount of dye adsorbed at equilibrium, denoted as qe (mg·g^{-1}), was calculated using the following equation:

$$qe = \frac{(C_0 - C_e) * V}{m} \quad (1)$$

with C_0 the initial concentration in mg.L^{-1}, C_e the equilibrium concentration in mg.L^{-1},V the volume of the methylene blue solution in L in contact with the mass m of the activated carbon in g.

The equilibrium adsorption isotherm was obtained by plotting the adsorption capacity qe as a function of the equilibrium concentration C_e.

2.2 ADSORPTION ISOTHERM MODELING

For adsorption isotherm modeling, nine isotherm models were employed, including five two-parameters models and four three-parameters models, as summarized in Table 1.

Table 1. Commonly used adsorption isotherm models

Number of parameters	Models	Mathematical formula	Hypotheses	References
Two-parameters models	Langmuir (1918)	$q_e = \frac{q_m K C_e}{1 + K C_e}$	• Single layer • Homogeneous sites • No interaction between the molecules	[18]
	Frundlich (1909)	$q_e = K C_e^{1/n}$	Heterogeneous sites	[19]
	Tempkin (1941)	$q_e = B \ln(A C_e)$	• Interactions between adsorbed molecules • Active sites of different energies	[20]
	Dubinin Radushkevich (1947)	$q_e = q_m e^{-K(\ln\left(1+\left(\frac{1}{C_e}\right)\right))^2}$	Heterogeneous adsorption surface	[21]
	Jovanovich (1969)	$q_e = q_m (1 - e^{K*Ce})$	• Interactions between the adsorbed molecules • Heterogeneous adsorption surface	[22]
Three-parameters models	Redlich-Peterson	$q_e = \frac{q_m C_e}{1 + K C_e^n}$	Heterogeneous adsorption surface	[19]
	Toth (1962)	$q_e = \frac{q_m * \mathrm{Ce}}{((\frac{1}{K}) + (C_e^t))1/\mathrm{t}}$	• Heterogeneous adsorption surface • Interactions adsorbant-adsorbate	[23]
	Khan	$q_e = \frac{q_m * \mathrm{K*Ce}}{(1+K*Ce^n}$	Heterogeneous adsorption surface	[24]
	Sips	$q_e = \frac{q_m * (K*Ce)^n}{(1+K*Ce^n}$	Heterogeneous adsorption surface	[25]

<u>Selection of the Best Adsorption Isotherm Model</u>

Each of the nine isotherm models was fitted to the experimental data. A rigorous comparison of these models was conducted based on five complementary statistical criteria: the coefficient of determination (r^2), the adjusted r^2, the sum of squared residual errors (SSE), the Akaike Information Criterion (AIC), and the Bayesian Information Criterion (BIC). The first three criteria were initially used to compare the two-parameter models among themselves and the three-parameter models among themselves. Subsequently, a general comparison was performed using AIC and BIC, which enabled the selection of the most suitable model for the experimental data.

The coefficient of determination (r^2) measures the proportion of variance explained by the model, providing an assessment of the goodness of fit. The higher the value, the better the fit. However, r^2 systematically increases with the number of model parameters [26]. This limitation is addressed by the adjusted r^2, which accounts for the number of parameters in the model. The sum of squared residual errors (SSE) quantifies the deviation between the observed values and the values predicted by the model, thereby providing additional information about the accuracy of the fit. Lower SSE values indicate better model performance.

The AIC and BIC criteria not only reflect the goodness of fit but also include a penalty for the number of parameters, allowing for a fair comparison of models with different complexities [26]. The best model is considered to be the one with the lowest AIC and BIC values.

AIC = N ln $(SSE/N) + 2 * K$ (2)

BIC = N*ln $(SSE/N) + K * \ln(N)$ [27] (3)

Where N is the number of experimental data
SSE is the sum of the squares of the errors
K is the nomber of parameters in the model

For N < 50, it is advised to use the corected AIC

AICc = AIC + $\frac{2*K*(K+1)}{N-K-1}$ [28] (4)

2.3 DEVELOPMENT OF A THREE-PARAMETER INDEPENDENT MODEL

This section presents a novel methodology for studying the adsorption isotherm of methylene blue onto a composite activated carbon prepared from cotton shells and capsules. The proposed three-parameter independent model, referred to as the **constrained Langmuir–Freundlich model,** combines the features of the classical Langmuir and Freundlich isotherms with a physical constraint reflecting the simultaneous presence of homogeneous and heterogeneous adsorption sites on the surface of the adsorbent.

- **Adsorption–Desorption Phenomenon**

Consider an adsorbent/adsorbate system. The dynamic equilibrium between adsorption and desorption of the adsorbate X (solute) at the surface of the adsorbent M can be expressed as follows:

$$X + M_{surface} \rightarrow XM_{surface} \quad (5)$$

Let us designate by:

Θ: the coverage rate of the surface or the ratio between the number of occupied sites and the number of available sites;

Ka ($L.s^{-1}mg^{-1}$): the adsorption rate and

Kd (s^{-1}): the desorption rate.

The variation of the surface coverage rate during the adsorption is:

$$\frac{d\Theta}{dt} = Ka * fa\ (C,\Theta) \quad (6)$$

Where fa is a function of the adsorbate concentration C and Θ

Likewise, the variation of the surface coverage rate during the desorption is:

$$\frac{d\Theta}{dt} = Kd * fd\ (C,\Theta) \quad (7)$$

Where fd is a function of the adsorbate concentration C and Θ

At the dynamic equilibrium:

$Ka * fa\ (C,\Theta) = Kd * fd\ (C,\Theta)$ [29] (8)

- Application to the Langmuir model

For Langmuir,

$$fa\ (C,\Theta) = C(1-\Theta) \quad (9)$$

And

$$fd\ (C,\Theta) = \Theta \quad (10)$$

By posing $K = \frac{Ka}{Kd}$ with K in L.mg^{-1}

And $\Theta = \frac{q}{qm}$,

It comes:

$$\Theta = \frac{KC}{1+KC} \quad (11)$$

And

$$q = q_m \frac{KC}{1+KC} \quad (12)$$

Under the following hypotheses:

- Single layer coverage;
- Equivalence of active sites;
- Indépendance of active sites [18]

With q_m the maximum adsorption capacity of the gaz.

This model has certain limitations in practical applications. Indeed, although this isotherm was initially proposed for gases, it is not always suitable for describing the adsorption mechanisms of molecules in solution [29].

Furthermore, the adsorption of a species in solution is always accompanied by desorption. Surface heterogeneity is also commonly observed. These factors necessitate a careful selection of the adsorption and desorption functions $fa\ (C, \Theta)$ and $fd\ (C, \Theta)$ respectively. In the following section, we propose a model designed to address these limitations.

- **Constrained Langmuir–Freundlich Model**

This model assumes that the adsorbent surface is composed simultaneously of homogeneous and heterogeneous sites, with a continuous weighting between them.

Let us put:

$$fa\ (C, \Theta) = \frac{C^{\alpha}}{1+K\ C^{\beta}} \quad (13)$$

The term C^{α} governs the initial adsorption growth, which can represent an inhibition phase where adsorption is moderate and the homogeneous sites dominate in terms of energy. Adsorbent–adsorbate interactions only become significant above a certain threshold. The term in the denominator accounts for the effect of heterogeneous sites and becomes significant only at high concentrations, reflecting a saturation effect that limits the amount adsorbed.

The exponents α and β, ranging between 0 and 1, represent the fractions of homogeneous and heterogeneous sites on the adsorbent surface, respectively, and are constrained by the relation α+β= 1. This constraint ensures a consistent physicochemical interpretation of both contributions.

$$fd\ (C, \Theta) = \Theta \quad (14)$$

By putting at the dynamic equilibrium:

$$Ka * fa\ (C, \Theta) = Kd * fd\ (C, \Theta) \quad (15)$$

And by making substitutions, we have:

$$q_e = \frac{q_m * K * C_e^{\alpha}}{1 + K * C_e^{\beta}} \quad (16)$$

Which is the constrained Langmuir-Frundlich model.

In this formula:

q_m (mgg^{-1}) represents the theoretical maximum adsorption capacity of the activated carbon, i.e., the amount of dye adsorbed by the activated carbon at saturation.

K is a coefficient reflecting the affinity between the adsorbent and the adsorbate. Its unit depends on β, and is generally expressed as $(L.mg^{-1})^{\beta}$

α and β represent, respectively, the fractions of homogeneous and heterogeneous sites on the surface, with the constraint α + β=1

- If α = 1 et β=0, we have:

$$q_e = \frac{q_m * K * C_e}{1+K} \quad (17)$$

The amount adsorbed is proportional to the equilibrium concentration, which corresponds to a Langmuir-type isotherm at low surface coverage, where the adsorbent exhibits a nearly uniform surface.

- If α = 0 et β=1, we have:

$$q_e = \frac{q_m * K}{1+K * C_e} \quad (18)$$

The adsorbent surface is totally heterogeneous.

- If 0< α<1 and 0< β<1, the activated carbon surface contains both homogeneous micropores and energetically dispersed sites, which is commonly observed in real activated carbons. This feature makes the model more representative of practical systems than the Langmuir or Freundlich models considered individually.
- In particular, if α = β = 0,5, we approach the Sips model:

$$q_e = \frac{q_m * K * C_e^{0,5}}{1+K * C_e^{0,5}} \quad (19)$$

3 RESULTS AND DISCUSSION

3.1 ADSORPTION ISOTHERM MODELING USING EXISTING MODELS

Table 2 presents the fitting results of the Langmuir, Freundlich, Dubinin–Radushkevich, Temkin, Jovanovich, Redlich–Peterson, Toth, Khan, and Sips models to the experimental data, based on five statistical criteria.

- **Comparison of two-parameters models**

The coefficients of determination (r^2) obtained for the two-parameter models ranged from 0.7922 (Dubinin–Radushkevich model) to 0.9414 (Langmuir model). The adjusted r^2 values followed the same trend, with the lowest value for Dubinin–Radushkevich (0.7691) and the highest for Langmuir (0.9349). Conversely, the sum of squared residual errors (SSE) displayed the opposite trend, with the highest value observed for Dubinin–Radushkevich (24,854.78) and the lowest for Langmuir (7,006.89). This observation is consistent with expectations, as a higher r^2 generally corresponds to a lower residual error [27].

Based on these criteria, the Langmuir model emerges as the best among the two-parameter models in terms of SSE and coefficients of determination and adjusted determination. Regarding the AICc and BIC values, they ranged from 107.74 (Langmuir) to 121.67 (Dubinin–Radushkevich) and from 107.04 (Langmuir) to 123.86 (Dubinin–Radushkevich), respectively. Accordingly, the Langmuir model is considered the best two-parameters model based on the lowest AICc and BIC values [27]

- **Comparison of three-parameters models**

For the three-parameter models, the coefficients of determination (r^2) ranged from 0.9763 (Khan) to 0.9873 (Sips). The adjusted r^2 values followed the same trend, with the lowest value for Khan (0.9703) and the highest for Sips (0.9873). The SSE values exhibited an opposite trend, with the highest value for Khan (2,834.78) and the lowest for Sips (1,509.71). This is consistent with the expectation that a higher r^2 corresponds to a smaller residual error [27].

Based on these criteria, the Sips model is the best among the three-parameter models, as it exhibits the lowest SSE and the highest $r^2$and adjusted r^2 values. The AICc and BIC values ranged from 94.78 (Sips) to 101.71 (Khan) and from 92.55 (Sips) to 99.48 (Khan), respectively. Therefore, Sips is also the preferred model among the three-parameter models due to its lowest AICc and BIC values [27].

- **Overall comparison of the nine models**

When considering $r^2$and adjusted r^2 alone, it is apparent that these metrics, along with SSE, become less relevant when comparing models with different numbers of parameters [30], as noted in several studies [31].

Table 2. Calculation of parameters and error functions for the nonlinear forms of the studied isotherm models

Numbers	Nonlinear models	Calculated Parameters								Errors functions					
		Qm	K	n	A	B	t	α	β	r^2	$adjusted r^2$	SSE	AIC	AICc	BIC
1	Langmuir	383,41	0.79	-	-	-	-			0,9414	0,9349	7006,89	75,02	107,74	107,04
2	Frundlich	-	198,41	7,04	-	-	-			0,8611	0,8456	16262,26	84,28	117,00	116,30
3	Dubinin-Radushkevich	354,85	0,97	-	-	-	-			0,7922	0,7691	24854,78	88,95	121,67	123,36
4	Tempkin	-	-	-	75,06	42,90	-			0,9388	0,9320	7311,79	75,4928	108,2139	109,9076
5	Jovanovich	-	-	-	361,38	0,63	-			0,8239	0,8044	21056,9279	87,1279	119,8490	121,5427
6	Redlich-Peterson	490,99	1,68	0,94	-	-	-			0,9775	0,9719	2685,50	66,47	101,12	98,88
7	Khan	278,10	1,65	0,93	-	-	-			0,9763	0,9703	2834,78	67,07	101,71	99,48
8	Toth	428,26	1,74	-	-	-	0,51			0,9866	0,9833	1592,88	60,72	95,37	93,14
9	Sips	420,61	0,57	0,60	-	-	-			0,9873	0,9873	1509,71	60,13	94,78	92,55
10	Constrained Langmuir-Frundlich	820,86	0,26	-	-	-	-	0,45	0,55	0,9910	0 ,9900	1076,56	56,42	91,07	88,83

The Sips model yields lower AICc and BIC values (94.78 and 92.55, respectively) compared to the Langmuir model (107.74 and 107.04, respectively), indicating that it is the best model among those studied for describing the adsorption of methylene blue onto composite activated carbons derived from seed shells and cotton capsules. These results are consistent with previous studies [32,33], which demonstrated that methylene blue adsorption onto coconut shell activated carbons was better described by the Sips model than by the Langmuir, Freundlich, Temkin, or Toth models.

The AIC criterion, which penalizes model complexity, suggests that the Sips model captures the underlying structure of the data while avoiding overfitting. Similarly, the BIC, which applies a stricter penalty for the number of parameters [34], reinforces this conclusion, indicating that the Sips model is not only accurate but also robust.

- **Type of adsorption**

The parameters obtained from the Sips model better reflect the actual adsorption process. In particular, the heterogeneity factor n calculated for this model is 0.60, which is less than 1, indicating heterogeneous adsorption [35]. Accordingly, methylene blue adsorption onto the composite activated carbons derived from seed shells and cotton capsules occurs on a heterogeneous surface, with a maximum adsorption capacity estimated at 420.61 mg·g^{-1}.

3.2 Performance Evaluation Of The Constrained Langmuir-Frundlich Model

Commonly used two-parameter isotherm models, such as Langmuir, Freundlich, Temkin, Dubinin–Radushkevich, and Jovanovich, were unable to fully capture all aspects of the adsorption system due to its complexity. This is reflected in their relatively higher AICc and BIC values. To achieve better fitting, it was necessary to consider three-parameter models, including Sips, Toth, Khan, and Redlich–Peterson. Among these, the Sips model provided the best overall performance. However, its coefficient of determination (r^2=0.9873) suggested there was still room for improvement, motivating the development and application of the **constrained Langmuir–Freundlich model** to the experimental data.

The constrained Langmuir–Freundlich model achieved a high coefficient of determination (r^2=0.9910), indicating excellent fitting to the experimental data. Although it is a three-parameter independent model, like the Sips (0.9873), Toth (0.9866), Khan (0.9763), and Redlich–Peterson (0.9775) models, it more effectively describes the adsorption of methylene blue on composite activated carbons derived from seed shells and cotton capsules. This suggests that certain physical aspects of the adsorption system are not fully captured by conventional models, whereas the constrained Langmuir–Freundlich model does. Moreover, it exhibits smaller error metrics (AICc = 91.07 and BIC = 88.83) than those obtained with the Langmuir (107.74 and 107.04, respectively) and Sips (94.78 and 92.55, respectively) models.

4 CONCLUSION

In this study, a new adsorption isotherm model was proposed, based on the simultaneous existence of homogeneous and heterogeneous sites on the adsorbent surface. This coexistence is physically represented by two parameters, α and β, corresponding to the fractions of homogeneous and heterogeneous sites, with the constraint α+β=1.

Although this model belongs to the family of hybrid isotherms, it is structurally novel due to its explicit physicochemical interpretation. It fits the experimental data well, with a coefficient of determination of 0.9910 and lower AICc (91.07) and BIC (88.83) values than the classical models studied, including Langmuir, Freundlich, and Sips.

The model can be used to design adsorption columns or to optimize dye adsorption processes on activated carbons. Future perspectives include applying this model to other adsorbents (e.g., zeolites, pozzolans) and adsorbates (e.g., dyes, metals) and integrating thermodynamic parameters (entropy, Gibbs free energy) to further characterize the types of interactions governing the adsorption process.

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