



Analysis of measuring methods of the concentration of methylene blue in the sorption process in fixed-bed column

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Abstract

The wastewater from industry is contaminated with dyes, which should be removed in order to prevent environmental damage. This paper presents a method for removing methylene blue from an aqueous solution using a fixed-bed adsorption process onto sawdust. In the article, the method of continuous measurement of methylene blue concentration in the process of sorption on sawdust was used. The results were compared with the results obtained by the standard method of periodic testing of the dye concentration. Continuous measurement of the dye concentration after the sorption process provides the actual nature of the process and the method offers additional information about the process, as opposed to a periodic test in which the concentration of the ingredient gives only averaged results. The kinetics of the methylene blue sorption process at variable initial dye concentrations are presented. The initial concentration of methylene blue had a significant impact on the process breakthrough curve. The increase in concentration (range of 10–90 mg/dm³) caused the extension of the penetration zone of the dye mass, thus gradually adsorbing itself on the bed. In the study of kinetics, the Bohart–Adams model was the best fit.

Keywords Adsorption · Methylene blue · Fixed-bed · Kinetics · Sawdust

Introduction

Dye contamination in wastewater derived from many industries including textile, paper, printing, plastics, and cosmetics is difficult and expensive to treat. Dyes in sewage affect photosynthesis by blocking light penetration, even at low concentration (Saini 2017). Methylene blue (MB) is a model dye that is easily visible even at low concentrations (Fu et al. 2015). The compound can be eliminated easily using a variety of methods: ozonation, membrane filtration, electrokinetic coagulation, and adsorption, which have the greatest potential and good efficiency (Ahmad et al. 2015; Sivarajasekar and Baskar 2015). A sorbent that works well in this process is activated carbon, which is also responsible for the high costs involved. There are many researchers that are working on replacing activated carbon with cheaper

adsorbents, e.g. waste fruits (Chahm et al. 2018), plant waste (Vyavahare et al. 2019), ashes as eggshell-treated palm oil fuel ash (Hasan et al. 2019), natural and modified zeolites (Bedeenezhad et al. 2019).

The sorption process may be researched by two methods: batch or fixed-bed adsorption (Jain and Gogate 2018). In technology, the continuous method has better prospects due to better performance and an easier optimization process. In dynamic adsorption, it is very important to determine the breakthrough curve, because this gives information that is necessary for the design of the column adsorption system. We can use the breakthrough curves to obtain the practical scale of the column adsorption system (Mohammed et al. 2016). It is possible to find this information in two ways. The first is experimental, which is most likely to be expensive and time-consuming. The second is to develop a mathematical model of the process. Because the processes involved in adsorption between solid and liquid phases are hard to describe, and there are many parameters that can affect them, it is not possible to create just one general mathematical model that describes the breakthrough curve; thus, there have been some models created that are better or worse depending on the specification of process.

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The aim of the study was to investigate the process of removing MB (a model organic contamination of wastewater) from the water solution by sorption on sawdust. The sorption process was carried out in a continuous system. The efficiency of the sorption method with a mathematical description of the adsorption process dynamics was determined. The purpose of the study was also to describe the sorption process by providing continuous MB concentration measurements.

For both batch and fixed-bed sorption processes, there are difficulties with continuous data collection. In the batch method, in order to measure the sample, the process needs to be stopped. In other words, a few parallel processes are needed to measure one variable, over time. In the column adsorption on fixed-bed method, the effluent is collected in a small vessel for a few minutes and then a sample from the vessel is measured, so the values present an average result from the collecting time (Hu et al. 2015; Kumar et al. 2016). As stated, both of these methods lack continuous measurements, which makes it impossible to describe fully the nature and character of the process. The continuous MB concentration analysis method used in this research allows an average MB concentration value as a real value to be obtained, even if there is a significant dispersion of the data collected. The middle level of the sorption process (defined as the average concentration value) used in this test method refers to the actual process value.

Materials and methods

Materials

In the sorption process, a methylene blue (MB) solution (Avantor Performance Materials Poland S.A. POCH, CAS: 61–73–4, > 82%) with an initial concentration of 10, 30, 50, 70, and 90 mg/dm³ was used. Wood sawdust was chosen as the adsorbent. The wood sawdust was purchased from a commercial supplier from Poland. The material was of natural origin, as a by-product from the processing of deciduous trees. The average size of the material was approximately 12.5 × 6.5 mm. All chemicals were used without any further purification.

Methods

The concentration of MB solution was measured by UV–Vis spectroscopy method (Rayleigh UV–Vis 1800 apparatus) using a flow cuvette (measurements were made every 20 s). The concentration of MB was determined by a wavelength of 664 nm, at which a characteristic maximum of MB occurs.

Sawdust was analysed by Fourier transform infrared (FTIR) and scanning electron microscopy with

energy-dispersive spectroscopy (SEM–EDS). Based on FTIR, the presence of functional groups of raw material and material after MB sorption was determined. Materials were analysed using an FTIR spectroscope (Nicolet 380), where the spectra were recorded from 3900 to 400 cm^{−1}. Surface morphology and element composition were established using SEM (Vegall-Tescan Company). The analysis of the micro-area was complemented with the use of an EDS detector.

Batch sorption process

In order to determine the equation coefficients and to investigate the nature of the sorption process, a series of studies on the removal of MB on sawdust in a “batch” contact test were carried out. Three equilibrium models were used to describe the sorption equilibrium parameters: Langmuir, Freundlich, and Temkin. The experiment consisted of mixing 0.2 g of sawdust with 100 cm³ of MB solution ($C_0 = 10\text{--}90\text{ mg/dm}^3$). The samples were mixed with a magnetic stirrer at 300 rpm at 20 °C. The sorption time ranged from 5 to 60 min. Next, the solution was separated from the adsorbent by vacuum filtration. The concentration of the dye in the solution was tested by UV–Vis analysis. The sorption capacity of the material was calculated from the dependence:

$$q = \frac{V \cdot (C_0 - C_t)}{m}$$

where C_0 is the initial MB concentration (mg/dm³), C_t is the retained MB concentration (mg/dm³) in solution at time t , V is the solution volume (dm³), and m is the weight of the adsorbent (g).

In the Langmuir model, the substance is adsorbed to form a monolayer. The number of active sites at which processes can take place is steady, and each molecule has a constant enthalpy and sorption activation energy. As a result, the maximum quantity of adsorbed particles is equal to the number of active sites on the surface of the adsorbent, and further adsorption of the component is impossible. The model is presented in the equation:

$$q = q_{\max} \frac{k_L \cdot C_t}{1 + k_L C_t}$$

where q_{\max} is the maximum sorption capacity of the material (mg/g), k_L is the Langmuir constant (dm³/mg).

The Freundlich model explains the process of multilayer adsorption. An increase in solution concentration causes continuous adsorption of the molecules. Compared to the Langmuir model, Freundlich’s model does not assume inhibition of adsorption once the critical concentration is exceeded, as a result of which no further adsorption would occur. Therefore, the Freundlich equation corresponds to

processes in a narrow concentration range in which process conditions are physical:

$$q = k_F \cdot C_t^{1/n}$$

where k_F and n are constants specific to the system and temperature.

The Temkin model presents a two-parameter equation describing the adsorption process. In the model is assumed an even distribution of the energy of the adsorbent–adsorbate bond, in which with the distance from the surface of the molecules the adsorption energy decreases linearly. The model is presented in the equation:

$$q = \frac{R \cdot T}{B} \ln(A \cdot C_t)$$

where A, B are characteristic parameters for the process.

Continuous column system

The adsorption of MB solution on sawdust was studied using the continuous column method. Sawdust weighing 2.0 g was packed into a glass column with a height of 102 mm and a diameter of 11 mm, the height of the bed was 100 mm. Before starting the process, the column was fed with deionised water for 30 min in an up-flow mode. The flow rate was 0.100 cm³/s. Figure 1 shows the scheme of the process. The MB (1) was fed into the column (3) from the bottom by piston pump (2). After the sorption process, the MB solution was diluted (4), and absorbance of the dye solution was measured by the UV–Vis spectrophotometer. The solution was diluted using deionised water provided by a pump (5) which was working at different parameters: 0.100, 0.500, 0.900, and 1.300 cm³/s (depending on the initial MB concentration).

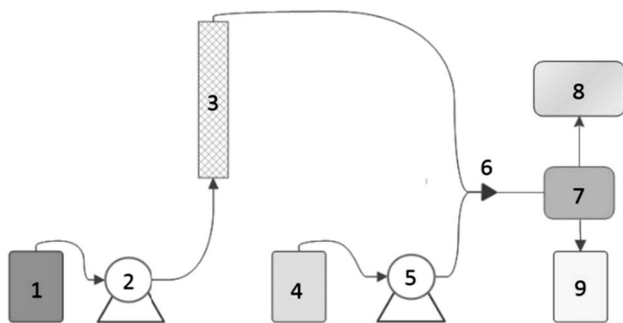


Fig. 1 Scheme of experimental column adsorption system: (1) dye solution, (2) pump, (3) column, (4) deionised water, (5) pump, (6) tee, (7) UV–Vis spectrophotometer, (8) computer, (9) diluted dye solution

Sorption dynamic

Two models were tried in order to establish breakthrough curves and other kinetic parameters that describe the process of the adsorption of MB onto sawdust in a fixed-bed column. The MB adsorption process in fixed-bed column for the following models is expressed in terms of C/C_0 as a function of time. The Bohart–Adams Bed Depth Service Time (BDST) model assumes that the adsorption rate is proportional to the concentration of the adsorbing individual as well as to the residual capacity of the adsorbent. This model is used to describe the initial part of the breakthrough curve (Bohart and Adams 1920). The nonlinear form is as follows:

$$\frac{C_t}{C_0} = \frac{\exp(k_{BA} \cdot C_0 \cdot t)}{\exp\left(k_{BA} \cdot N_0 \cdot \frac{H}{v}\right) - 1 + \exp(k_{BA} \cdot C_0 \cdot t)}$$

The Clark model is based on the mass transfer coefficient concept in combination with the Freundlich isotherm (Clark 1987). The nonlinear form is as follows:

$$\frac{C_t}{C_0} = \left(\frac{1}{1 + A_C \cdot \exp(-r \cdot t)} \right)^{\frac{1}{n_F}}$$

where C_t (mg/dm³) is the effluent concentration of MB at time (min), C_0 (mg/dm³) is the initial MB concentration, k_{BA} [dm³/(min mg)] is the Bohart–Adams kinetic constant, N_0 (mg/dm³) is the maximum volumetric adsorption capacity, H (cm) is the bed depth in the column, v (cm/min) is the flow rate, A_C and r (min⁻¹) are both Clark constants, n_F is the Freundlich constant, and t is the time (min).

Results and discussion

Discussion

Characteristics of sawdust

In the literature, the sawdust was successfully used to remove contaminants, e.g. dyes: Black T Dye (Akhouairi et al. 2019), metal ions including heavy metals, e.g. zinc, copper(II), iron(II), chrome(VI) (Gupta and Babu 2009; Božić et al. 2009), other organic compounds, e.g. phenol (Larous and Meniai 2012). The benefits of the chosen adsorbent are availability, the possibility of using sawdust-based waste material, as well as a high degree of biodegradation, low weight of the material, and extremely low cost.

The SEM microphotographs confirmed a fibrous structure of the material (Fig. 2). The EDS analysis revealed a significant proportion of carbon and oxygen, which, in combination with FTIR analysis, confirms the presence of cellulose, lignin, and hemicellulose. The 3412 cm⁻¹ band proves there



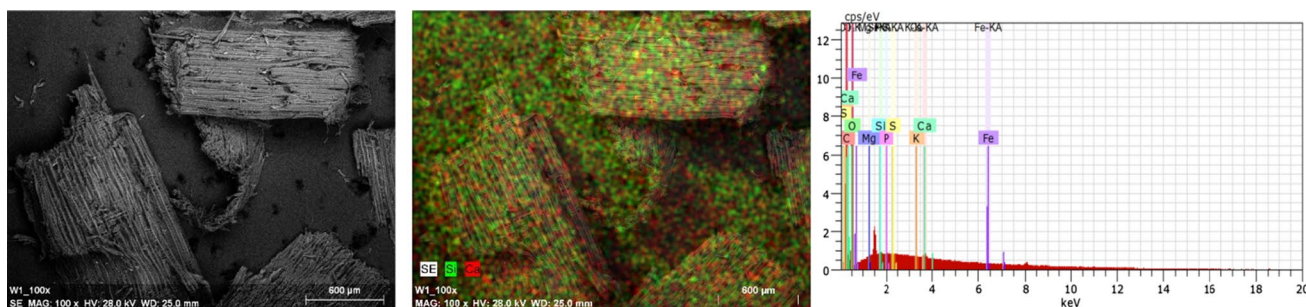


Fig. 2 SEM microphotographs of sawdust (100×), with EDS analysis

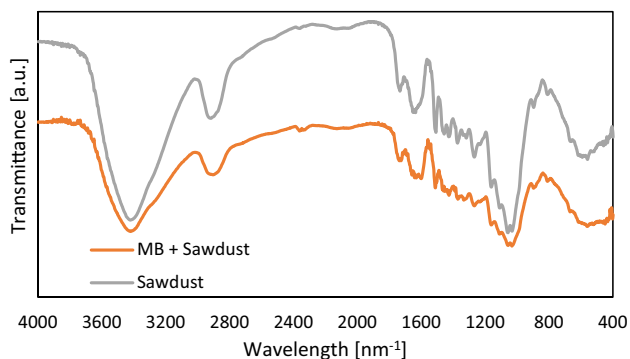


Fig. 3 Image of FTIR analysis for sawdust and sawdust with methylene blue after sorption process

were stretching vibrations of OH groups, stretching vibrations of alkyl C–H at 2924 cm^{-1} and of glycoside groups at 897 cm^{-1} , confirming the presence of cellulose. Bands at 1269 and 1059 cm^{-1} might be in response to stretching C–O group vibrations. The band at 1033 cm^{-1} may result in stretching C–O, C=O, C–C–O bonds, which are characteristic for cellulose, lignin, and hemicellulose. An aromatic structure can be defined by peaks at 1512 and 1427 cm^{-1} , which are responsible for C=C stretching bonds, while the 1464 cm^{-1} peak comes from bond vibrations of the C–H group, and bands under 900 cm^{-1} result from deformation vibrations.

The figure shows FTIR diagrams that enabled the confirmation of MB sorption on sawdust (Fig. 3). Comparison with the bed before sorption showed an increase in peak power at wavelengths 3412, 2924, 1059, and 1033 cm^{-1} . The FTIR spectrum of the sorbent with MB showed that most of the characteristic bonds for the sorbent are occupied by MB because their peaks were extinguished. Two new peaks

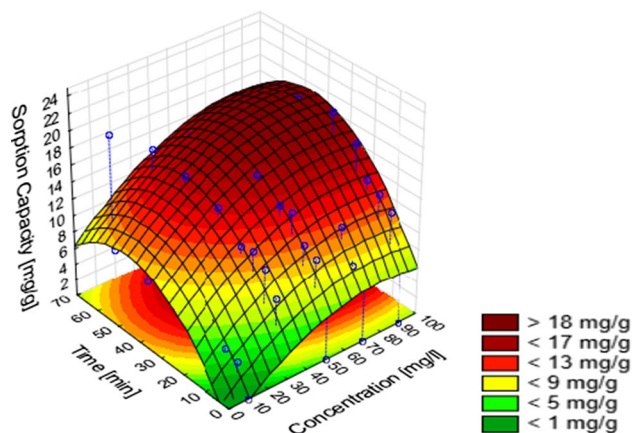


Fig. 4 Sorption capacity of sawdust depending on time and initial MB concentration

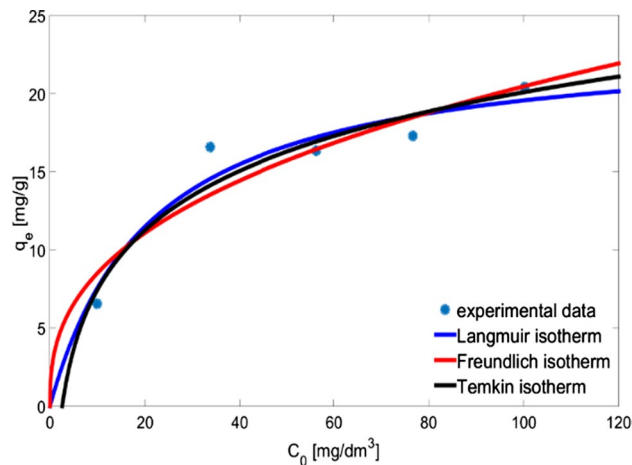


Fig. 5 Sorption isotherms plot of MB onto sawdust

Table 1 Langmuir, Freundlich, and Temkin isotherm constants and correlation coefficients for MB sorption on sawdust

Isotherm model	Parameter	Value
Langmuir $q = \frac{q_{\max} \cdot k_L \cdot C_t}{1 + k_L \cdot C_t}$	q_{\max} (mg/g)	23.73
	k_L (dm ³ /mg)	0.0469
	R^2	0.9252
Freundlich $q = k_F \cdot C_t^{1/n}$	k_F [mg ^{1-(1/n)} L ^{1/n} g ⁻¹]	3.542
	$1/n$	0.3808
	R^2	0.8677
Temkin $q = \frac{RT}{B} \ln(AC_t)$	A (dm ³ /g)	0.3884
	B	519.7
	R^2	0.9182

at 1600 and 1658 cm⁻¹ are visible that confirm the sorption of MB onto sawdust (Ovchinnikov et al. 2016).

Sorption dynamic

Figure 4 presents the simultaneous impact of time and MB initial concentration on the sorption capacity of the sawdust in batch processes.

In order to characterise the adsorption equilibrium of MB on sawdust, it is essential to establish the isotherm models for the equilibrium curve (Fig. 5). Three models have been tested: Langmuir, Freundlich, and Temkin (Table 1). The Langmuir model was characterised by the highest determination coefficient. Similar results of the equilibrium nature of MB sorption were achieved by, among others, Onyeocha et al. (2016) and Chowdhury et al. (2016), who adsorbed MB on sugarcane bark and chaff biomass and by rubber leaf powder, respectively. In the next stage, the determined

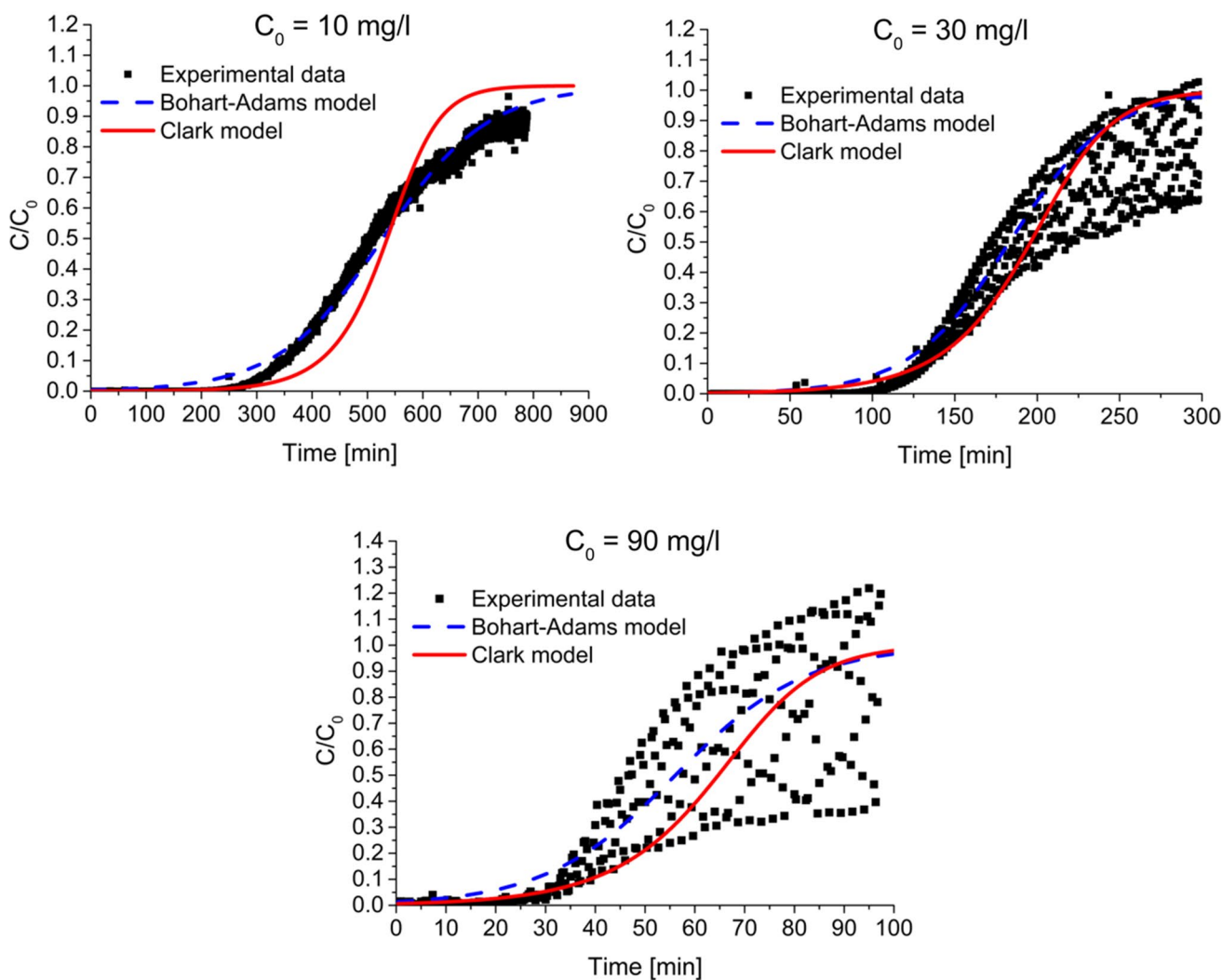


Fig. 6 Graphical presentation of experimental data and fitted models for different initial MB concentrations: 10, 30, and 90 mg/dm³

coefficients were used in the calculation of breakdown curve models in the fixed-bed adsorption process.

The experimental data obtained in this experiment are not linear. The data are in the shape of cloud of points—a wide range of dispersed values as shown in Fig. 6. Because of this, it is not possible to measure the description of their effective breakthrough curve using a model with R^2 as it is in data with linear form. To be able to choose the best model and to compare them the percentage of explained variance needs to be used.

The mass transfer coefficient k_{BA} is the highest at lowest initial concentrations and gradually decreases with an increase in the initial concentration, which may suggest the dominance of external mass transfer actions in the column adsorption process during the initial stages. Auta and Hameed (2014) observed the same correlation between MB concentration and k_{BA} which suggests a successful model prediction. The value of Clark constants, A_C constant decreases and r constant increases with the initial MB concentration. The reason for that dependence may be in the specification of the Clark model, because this model is compatible for low values of C/C_0 , and in this specific concentration, the data with low C/C_0 factor values have a wide dispersion, which may effect in that break of correlation with higher initial MB concentrations. Song et al. (2011) presented similar behaviour of the parameter A_C for changes of initial MB concentrations as in this paper. Because of the specific method of measurement, the results presenting a lower percentage of explained variance for each of the models analysed (Table 2), which evidently decreased with an increase in the initial MB concentrations. Among the models that were analysed, the best results were given by the Bohart–Adams model. As the initial MB concentration increases and the other parameters remain steady (flow rate and amount of sorbent), the sorption process becomes faster, the data exhibit a wider range of values, and the breakthrough curves are sharper as is shown in Fig. 7.

Higher compatibility of the Adams–Bohart model with experimental data results from the assumptions of the model. The Adams–Bohart model assumes that the rate of

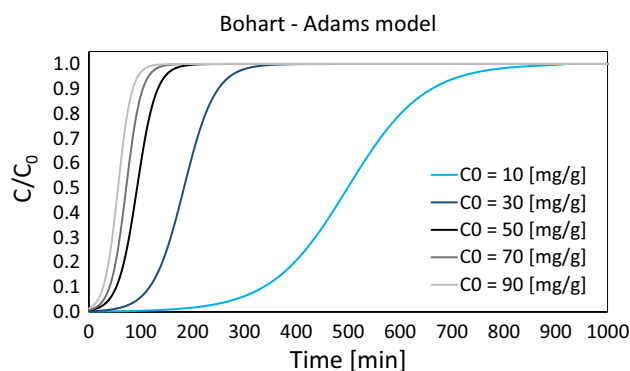


Fig. 7 Bohart–Adams model breakthrough curves for different initial MB concentration (from left to right, 90, 70, 50, 30, 10 mg/dm³)

adsorption is proportional to the adsorption capacity of the sorbent, and the balance of the process is not momentary, but is an averaged value of the whole process. This model describes the initial shape of the deposit's breakthrough curve particularly well, i.e. when the adsorbent still has most of the free active sites. Over time, the line describing the model is approximately an average result of the measurement points. Clark's model, contrary to the first one, assumes that the shape of the mass permeation zone is constant. In the MB sorption process, the mass transfer zone increases with the length of the column, which can be seen from the increasing distribution of experimental points in Fig. 6. This causes the limited fit of the model to process points.

Figure 7 presents the Bohart–Adams breakthrough curves for MB sorption for different initial MB concentrations. The breakthrough curves are sharper as the initial MB concentrations increase, which was expected as the fixed-bed height is constant. This figure also shows that the differences between the shape of the curves for the low concentrations of 10 and 30 mg/dm³ are much greater than between the high initial MB concentrations 50, 70, and 90 mg/dm³. These breakthrough curves also

Table 2 BDST—Bohart Adams and Clark model parameters obtained using non-linear regression analysis

C (mg/dm ³)	Bohart–Adams BDST model parameters			Clark model parameters		
	Adjusted R^2	k_{BA} (dm ³ mg ⁻¹ min ⁻¹)	N_0 (mg dm ⁻³)	Adjusted R^2	A_C	r (min ⁻¹)
10	0.9649	0.001353	6.2260	0.9363	96.70×10^4	0.02436
30	0.9141	0.001105	6.8616	0.8783	1.91×10^4	0.04654
50	0.8507	0.001095	5.7980	0.8638	1.02×10^4	0.07912
70	0.8262	0.000977	6.2852	0.8505	0.77×10^4	0.09105
90	0.7582	0.000851	6.3185	0.7944	0.37×10^4	0.11579



show that a wide dispersion of values does not have a negative impact on the calculated model. Ponnusami et al. (2008) researched the process of MB sorption onto guava leaf powder using a constant flow rate and initial dye concentration and measured the effect of bed depth for five different values. Measurements were made in the standard way: collecting samples of effluent for some time then analysing them. The breakthrough curves calculated using the Thomas model in this case are sharper with a decrease in the bed depth, which shows similar behaviour to the results obtained in the present work. Hamdaoui researched dynamic sorption of MB by cedar sawdust and crushed brick in a fixed-bed column. He used various models to calculate the breakthrough curves with different flow rate parameters. The samples were collected in the standard way, but in this case the R value was similar for each of the flow rates. The breakthrough curves predicted by the Clark model show that the experimental data suits the model very well at C/C_0 ratios of cedar sawdust above 0.07 (Hamdaoui 2006).

Conclusion

MB can be removed from an aqueous solution using a fixed-bed adsorption process onto sawdust. (The deposit breakthrough for the highest applied concentration of $C_{0,MB} = 90 \text{ mg/dm}^3$ was approximately 30 min.) The Langmuir model best characterises the MB sorption balance on sawdust. The highest fit of the model confirms the chemical nature of sorption.

The kinetics of the sorption of MB onto sawdust and breakthrough curves were best described by the Bohart–Adams model. This model assumes that the rate of adsorption is proportional to the adsorption capacity of the sorbent, and the balance of the process is not momentary, but is an averaged value of the whole process. The shapes of the breakthrough curves are sharper with increasing initial MB concentration. The differences in shape between the highest initial MB concentrations were slight, but the differences between the low initial MB concentrations were incomparably greater.

Continuous measurement of the dye concentration after the sorption process provides the actual nature of the process (not only averaged results), and the method offers additional information about the process. In the MB sorption process, the mass transfer zone increases with the length of the column, which can be seen from the increasing distribution of experimental points.

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Compliance with ethical standards

Conflict of interest The authors report no declarations of interest.

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