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Research Article

DESIGN OF A FIXED BED ADSORPTION COLUMN AND MODELLING OF OPERATING PARAMETERS FOR THE REMOVAL OF METHYLENE BLUE IN DYNAMIC MODE

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ABSTRACT

This study investigates the potential of using the mixture of titaniferous sand and attapulgite as adsorbents in a fixed bed adsorption process to remove a synthetic dye such as methylene blue in aqueous media. The different adsorbents were characterised by X-ray fluorescence spectroscopy and infrared spectroscopy. The different physico-chemical parameters such as pH, zero charge potential, bulk and absolute density, porosity and specific surface area were determined. The sizing algorithm used resulted in a number of transfer units (NUT) equal to 20.109, a height of transfer unit (HUT) equal to 0.515, a material transfer coefficient (Kya) equal to 3.159 and a height of the column (Z) equal to 1.05m. The influence of different experimental parameters such as initial dye concentration, adsorbent bed height and feed rate on the breakthrough curve was investigated. Various simple mathematical models such as Adams-Bohart and Thomas were applied in order to study the dynamic behaviour of the column and to estimate some kinetic coefficients through the experimental data obtained from the dynamic studies performed on the fixed bed. The results showed that the Thomas and Adams-Bohart models perfectly describe the behaviour of the breakthrough curves with values of coefficients of determination R2 that are higher than 0.90 except for the concentration of the dye equal to 50mg/L which has a coefficient R2 equal to 0.88.

KEYWORDS

Adsorption, methylene blue, fixed bed column, attapulgite, titaniferous sand.

INTRODUCTION

Dyes are organic compounds that are widely used in many industries, including textile, paper, food, cosmetics and paint, resulting in large amounts of water-soluble dye molecules in wastewater (Al Arni et al., 2021; Laskar & Kumar, 2018). In recent decades, as a result of industrial increasing and technological advances, coloured effluents have caused damage to the aquatic ecosystem and posed a significant threat to human health due to their toxicity (Karaoğlu et al., 2010; Wazir et al., 2020). To date, several techniques have been identified for the removal of dyes, including solvent extraction, photocatalytic degradation, coagulation, membrane filtration, chemical and biological oxidation and adsorption (Léonce Kouadio et al., 2022; Wang et al., 2022). Compared to other treatment systems, adsorption is an inexpensive, accessible and feasible method for the decontamination of wastewater with dyes (Al-Mahbashi et al., 2022; Blanco-Flores et al., 2016). Activated carbon is the most widely used adsorbent due to its high adsorption capacity for organic materials (Bouchemal & Achour, 2007; SEHILI, 2008). However, this adsorbent has a high cost and is difficult to regenerate (Dutta & Basu, 2014; Shah et al., 2021). The search for another effective and less expensive adsorbent is therefore interesting. Fixed bed column adsorption also has many advantages due to its ease of use and high removal efficiency. In addition, it can be easily scaled up to laboratory or industrial scale (Sylvia et al., 2018; Vasques et al., 2009). Continuous mode adsorption is generally characterized by the so-called breakthrough curves, representation of the effluent concentration at the column outlet as a function of the time profile in a fixed bed column (Patel, 2019; Puthiya Veetil Nidheesh, Rajan Gandhimathi, 2012). The design and optimization of the fixed bed column generally involves mathematical models which are used for both definition and prediction of the experimental data and are an interesting tool for scaling up and design Nwabanne & Igbokwe, 2012; Sazali et al., 2020). These mathematical models such as Bohart-Adams, Thomas, Yoon-Nelson, Wolborska, Clark etc. have been used by

M.D. Yahya et al. for the optimization of iron (II) and lead (II) removal on a fixed bed adsorption column (Yahya et al, 2020) and Fatemeh Maleki et al. for the removal of cobalt (II) from an aqueous solution by modified polymeric adsorbents prepared with induced grafts (Maleki et al., 2021). On the other hand, the studies of Patel, H showed that fixed bed column adsorption techniques are considered as the best methods due to their practical advantages and a set of columns is used to have a proper industrial perspective and get better results(Patel, 2019). GHINWA MELODIE NAJA et al. showed that columns can be operated in series to better control adsorption or in parallel to increase the capacity of the system. The advantages of this system include the simple form, the ability to perform continuous flow operations, the lack of limitation in increasing its scale, the lack of need for solid-liquid separation and the ability to perform regeneration and washing (Naja et al., 2010).

In this work, firstly, a fixed bed adsorption column was designed and then a mixture of titaniferous sand and attapulgite was used as an effective adsorbent to remove methylene blue in aqueous media. In addition, this study examined the effects of operating conditions such as bed height, initial dye concentration and flow rate. The experimental data obtained from the dye adsorption was modelled using the Thomas and Adam-Bohart models.

MATERIALS AND METHODS

Adsorbat

The pollutant was methylene blue, also called tetramethylthionine hydrochloride. It is a cationic dye which belonging to the group of quinone imides belonging to the Thiazine section. It is a sulphur dye in which two benzene rings are joined by a closed ring of one nitrogen atom, one sulphur atom and four carbon atoms.

Preparation of adsorbents

Titaniferous sand and attapulgite were used as adsorbents. The titaniferous sand is a residue of a mining industry located in Senegal. The treatment was carried out by bringing it into contact with a sulphuric acid solution of concentration 4mol/L and a volume of 100 mL. The mixture was stirred for 4 hours to ensure maximum contact with the sulphuric acid. At the end of the operation, the sand is recovered and washed several times with distilled water until a pH close to neutrality is obtained. It is then dried in an oven for 24 hours and placed in flasks for later use. The other material, namely attapulgite, has not undergone any prior treatment and is used in a raw state in the work. It was collected in Mbour, one of the departments of Senegal.

Sizing of the adsorption column

The sizing of the adsorption column was based on results from the removal of methylene blue in batch mode (Kalidou BA et al., 2021). After optimizing the operating parameters that influence the pollutant removal efficiency, the adsorption isotherms were studied in order to calculate the material transfer zone (MTZ) using the most suitable isotherm, the initial concentration and the maximum tolerated concentration (Cb). The various calculations were carried out in Excel.

Considering that the adsorption takes place on a fixed bed, the sizing will therefore consist in finding the length of the material transfer zone as well as that of the column. For this, the following assumptions will be used:

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- The density of the fluid is assumed to be that of water and is ρf =1000g/dm3 ;
- The density of the adsorbent is assimilated to that of titanium sand and is ρa =2209g/dm3 ;

The algorithm used to size the fixed bed column is given below :

The required adsorbent mass Ma is calculated using Eq. (1).

$$M_a = y * M_{a_{exp}} \text{ avec } y = \frac{Q}{Q_{exp}} \quad (1)$$

Where :

Y is a multiplicative factor to move from experimental to real values ;

$M_{a_{exp}}$: the optimal mass found experimentally ;

Q : the actual volume flow rate of the column ;

Q_{exp} : the experimental volume flow rate.

The fraction L'/V' is determined from Eq. (2) :

$$\frac{L'}{V'} = \frac{q_1}{C_i} \quad (2)$$

with :

L' : mass flow rate of the adsorbent ;

V' : Volume flow of the effluent.

q_1 is calculated by replacing C_e with C_i in the equation for the equilibrium curve and this leads to Eq. (3).

$$q_1 = \frac{K_L q_m C_i}{1 + K_L C_i} \quad (3)$$

The saturation concentration C_S is calculated using Eq. (4).

$$C_s = \gamma \, Ci \text{ avec } \gamma = 0{,}95 \quad (4)$$

The number of transfer units NUT is obtained from the Eq. (5).

$$NUT = \int_{C_b}^{C_S} \frac{1}{C - C_e} dC \quad (5)$$

with :

C_b : the maximum tolerated concentration ;

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C_e : the concentration at equilibrium.

The transfer unit height HUT is calculated through Eq. (6).

$$\mathrm{HUT} = \frac{Q}{Ky.S} \tag{6}$$

with :

K_y : the material transfer coefficient ;

S : the section of the column.

The height of the material transfer zone Z_a is obtained using Eq. (7).

$$Z_a = \mathrm{NUT} \times \mathrm{HUT} \tag{7}$$

The unsaturated fraction f of the adsorbent was also calculated using Eq. (8).

$$f = \int_0^1 \frac{1}{C - Ci} d\alpha \tag{8}$$

Where α is a change of variable such that :

$$\alpha = \frac{\int_{C_b}^{C} \frac{1}{C - C_e} dC}{\int_{C_b}^{C_s} \frac{1}{C - C_e} dC} \tag{9}$$

The calculation of ZF is done using Eq. (10).

$$ZF = t_c \times \frac{Q}{S} \times \frac{L'}{V'} \times \frac{1}{\rho_a} \tag{10}$$

with :

V' Flow rate of the inert fluid ;

F : the saturated fraction of the column ;

Z : the height of the column ;

ρ_a : the density of the adsorbent.

The height Z of the adsorption column is obtained from Eq. (11).

$$Z = ZF + f.Za \tag{11}$$

The cycle time is deduced from Eq. (12)

$$t_c = \frac{SZ\rho_a X_1 F}{V'Y_1} \tag{12}$$

Description of the adsorption pilot

The pilot consisted of a glass column (1m height and 3cm in diameter), inside which there was a support to allow attachment of the adsorbent bed. The effluent with a known initial concentration was sent to the top of the column by a peristaltic pump calibrated beforehand. The dye was passed through the adsorbent bed and recovered at the

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bottom of the column using a tank. The change in methylene blue concentration was monitored as a function of time. The samples were collected in 2h intervals and were put inside glass tubes to avoid any kind of light modification of the solution or any kind of bacterial contamination. The residual concentration of methylene blue was determined by a spectrophotometer at a wavelength of 664nm. The different parameters as a function of time were the feed flowrate to the column, the initial concentration of methylene blue and the height of the adsorbent bed. Finally, different mathematical models such as Thomas, Adams and Board were used to study the behaviour of methylene blue on adsorbent beds. Figure 1 shows the process used to remove methylene blue in dynamic mode.

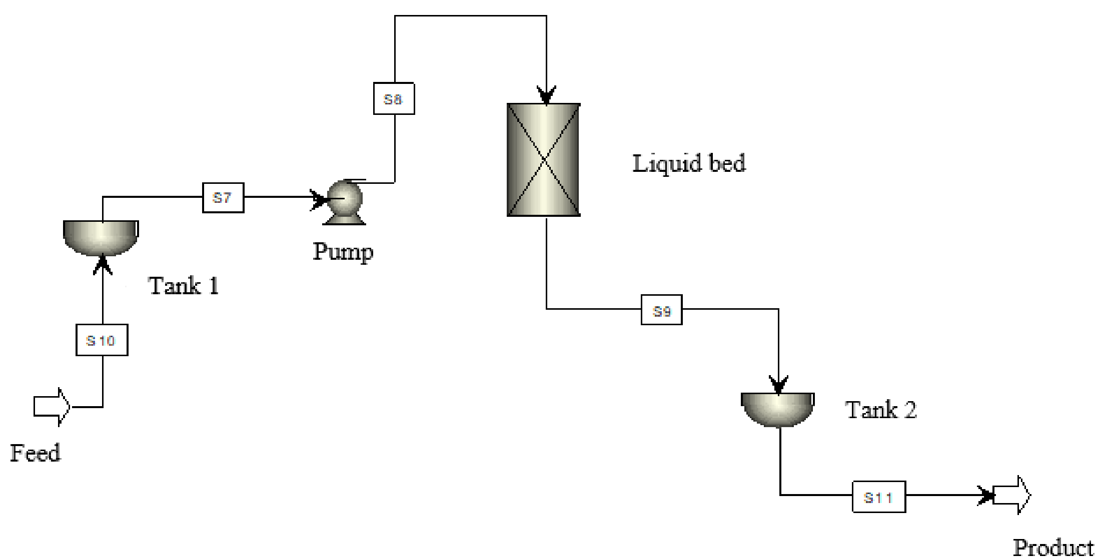

Figure 1. Methylene blue removal process in dynamic mode

Modelling of breakthrough curves

It consists of using numerical models to access the concentration profile of the dye at the outlet of the adsorption column. Various simple mathematical models such as the Bohart-Adams and Thomas models have been developed to predict the dynamic behaviour of the column and to estimate some kinetic coefficients through the experimental data obtained from the dynamic studies carried out on fixed bed to predict the breakthrough curves.

The Thomas Model

This is the most widely used model in the literature to describe the adsorption of organic, inorganic and heavy metal compounds. It is obtained by plotting the variation of ln $((C_0 / C_t) - 1)$ as a function of time t. It is given by Eq. (13) (Alardhi et al., 2020).

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$$\ln\left(\frac{C_0}{C_t}-1\right)=\frac{K_{Th}*q_0*m}{F}-K_{Th}C_0t \quad (13)$$

C_0 : Initial concentration of the pollutant (mg. L^{-1}) ;
C_t : Effluent concentration at time (t) at the column outlet (mg. L^{-1}) ;
m : Mass of the bed (g) ;
K_{Th} : Thomas constant (L.mg^{-1}. min^{-1}) ;

q_0 : Maximum adsorption capacity of the solute on the adsorbent (mg.g^{-1});
F : Solution feed rate to the column (L.min^{-1}) ;
t : Breakthrough time relative to the concentration C_t at the column outlet (min).

The Adams Bohart model

It is one of the first models to describe the breakthrough curves of an adsorbent bed. The breakthrough time is related to the operating parameters of the adsorber by Eq. (14) (Alardhi et al., 2020).

$$\ln\left(\frac{C_0}{C_b}-1\right)=\frac{K_{AB}*N_0*Z}{U}-K_{AB}C_0t_b \quad (14)$$

t_b : the breakthrough time;
C_0 : initial concentration (mg. L^{-1}) ;
U : velocity in the reactor assumed to be empty (cm.min^{-1}) ;
N_0 : dynamic adsorption capacity (mg. L^{-1}) ;
Z : Trim height (cm) ;
K_{AB} : Adams-Bohart constant.

RESULTS AND DISCUSSION

Physico-chemical characterization

Before the two materials were used as adsorbents, they were characterized in order to determine their physico-chemical properties. The different values are given in Table 1.

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Table 1 : Physico-chemical properties of adsorbents

Physical properties	Titaniferous sand	Attapulgite
pH	6.4	8.21
pH_{pzc}	8,75	8.75
Absolute density (kg/m^3)	4054	1660
Bulk density (kg/m^3)	2500	455
Porosity (%)	38.33	72.5
External specific surface (m^2 /g)	13.4	70.68

The values obtained show on the one hand that titaniferous sand treated with sulphuric acid is very dense, somewhat acidic and has a relatively low specific surface area, whereas attapulgite is basic, less dense than titaniferous sand and has a higher specific surface area. The combination of these two materials will go a long way to promoting good interaction with the cationic dyes.

X-ray fluorescence characterisation

X-ray fluorescence spectroscopy was used to characterise the composition of the titaniferous sand and attapulgite (figure.2a and 2b).

The analysis of titaniferous sand treated with sulphuric acid shows that titanium, iron and aluminium are the predominant elements which are present at concentrations of 300000 ppm, 63423.27 ppm and 18676.65 ppm respectively. This confirms the presence of oxides such as titanium dioxide, iron oxide and alumina, which are essential elements in the adsorption of refractory organic and inorganic compounds. However, for attapulgite, the major elements are silicon, iron, calcium with respective concentrations of 408028.69 ppm, 17829.26 ppm and 11952.21ppm. The literature has revealed a great importance given to these elements in the context of surface adsorption of organic compounds and heavy metals (Bhattacharyya & Gupta, 2007).

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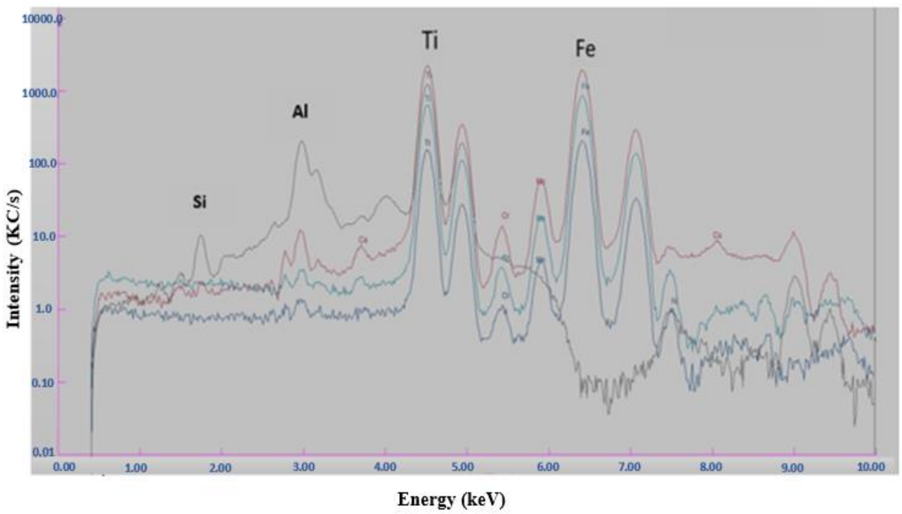
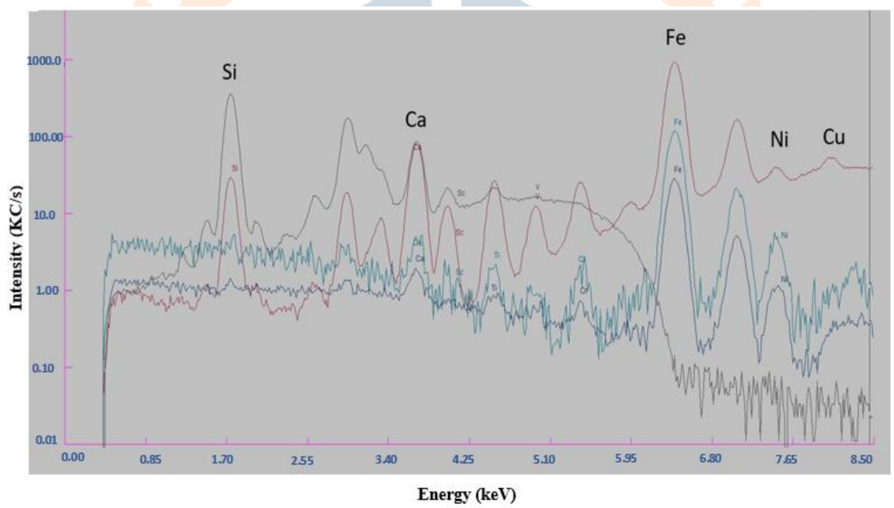


(a)



(b)

Figure 2. X-ray spectrum of titaniferous sand (a) and attapulgite (b)

Characterizations with IR spectroscopy

IR characterisation was carried out in order to determine the different surface functions of attapulgite and titaniferous sand (figure.3a and 3b). We note the existence of a band located at 3550.19 cm-1 corresponds to the elongation vibrations of the OH groups of the water molecules absorbed within the attapulgite structure. These water molecules are characterised by coordination water, zeolite water and hydroxyl groups bound to the last metal cations in the octahedral layer. A band between 1600-1700 cm-1 can

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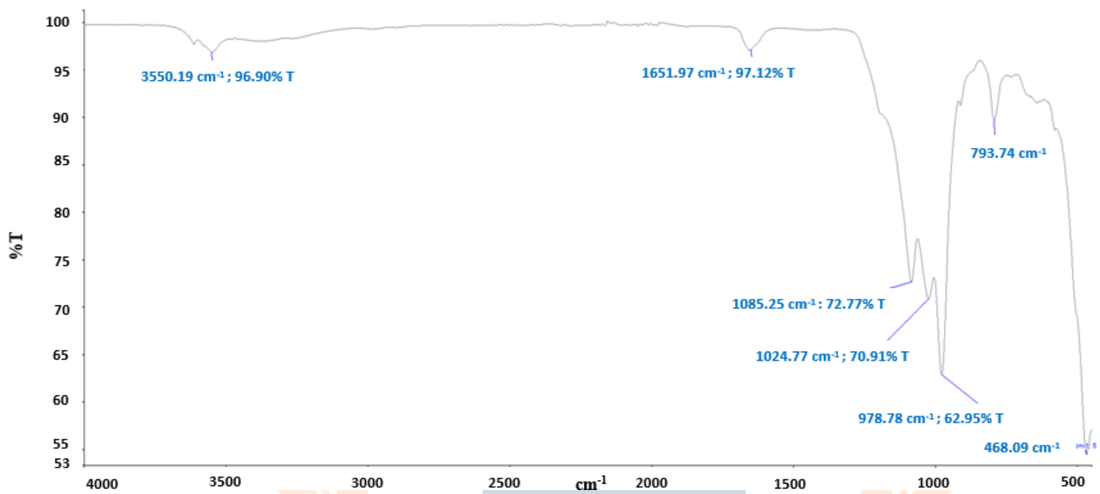
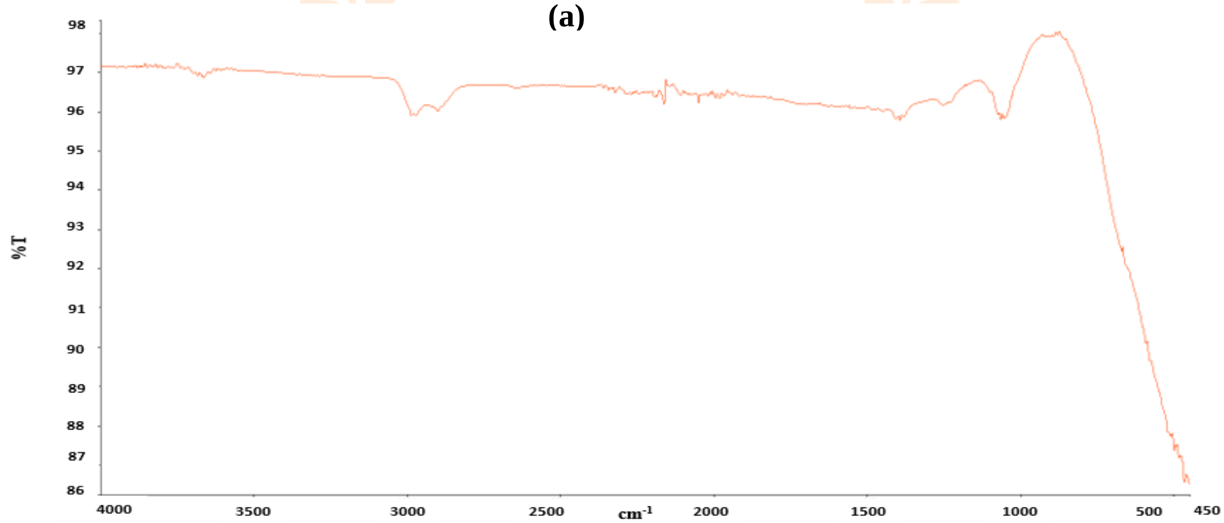
be attributed to the valence vibrations of the OH group of the water of constitution, in addition to the binding vibrations of the adsorbed water located at 1651.97cm-1(Y. Kismir, 2011). An intense band located between 900-1200 cm-1 and centred around 1085.25 cm-1 corresponds to the valence vibrations of the Si-O bond. The band around 978.6 cm-1 corresponds to the presence of calcium carbonate $CaCO_3$. The Si-O-Al bond gives rise to a band around 468.09 cm-1(Prost, 2018). However, for titaniferous sand, no absorption bands were detected but the pattern of the curve shows that the constituents in its structure are linked together by ionic bonds (crystal structure).



(a)



(b)

Figure 3. IR spectrum of attapulgite (a) and titaniferous sand (b)

Sizing of the adsorption column

The values obtained from the adopted sizing algorithm are presented in Table 2. These were used for the design of the fixed bed adsorption column. The calculation of the column sizing was done using the Excel spreadsheet.

Table 2 : Design parameters of the fixed bed column

HUT	NUT	K_{ya}	M_a (g/s)	Z (m)	Z_a (cm)	T_c (s)	f
0.515	20.109	3.159	5.6	1.05	10.37	14400	0.93

Study of the effect of operating parameters

Effect of initial dye concentration

The breakthrough curves of methylene blue adsorption by the adsorbent mixture studied under the conditions of bed height equal to 3cm, flow rate equal to 18mL/min, show that the increase in concentration from 50mg/L to 100mg/L affects the bed saturation and breakthrough time. This observation can be explained by the fact that the concentration gradient is a driving force for the proper functioning of the mass transfer from the liquid phase to the adsorbent surface. Thus, an increase in the concentration of methylene blue causes a decrease in the breakthrough time of the dye. This can be verified from figure 4a, at increasing dye concentration, the adsorption sites are occupied very quickly and the adsorbent bed is saturated in a shorter period of time. Identical results were reported by Sarici-Ozdemir (Sarici-Ozdemir & Onay, 2018) and Aydın et al. (Aydın et al., 2021).

Effect of adsorbent bed height

The breakthrough curves for the adsorbent mixture with two different bed heights (2 and 3 cm), with an initial concentration of 100 mg. L-1 and a flow rate of 18 mL.min-1 are presented in figure 4b. For the same concentration and fixed feed rate, increasing the bed height from 2cm to 3cm leads to an increase in the amount of BM adsorbed and the time required to reach saturation of the column increases. Thus, the variation in height is proportional to the breakthrough time, the higher the height, the better the breakthrough time. By increasing the bed height, the height of the material transfer zone increases, the exchange rate between solute and solid decreases. Similar results were found by M.D. Yahya (Yahya et al., 2020) and Dereje Tadesse Mekonnen (Mekonnen et al., 2021).

Effect of dye feed rate on breakthrough curve

Breakthrough curves are obtained by maintaining the initial concentration at 100 mg.L-1, the bed height at 3cm and varying the feed rate from 18 to 20 mL.min-1. The results obtained are shown in figure 4c. These variations show that when the feed rate is increased, when the residence time of the solute molecules in the column is decreased, the height of the matter transfer zone decreases, and thus a loss of efficiency of the adsorbent to immobilise the dye molecules. This leads to an increase in the exchange rate between the dye molecules and the adsorbent, allowing the saturation rate of the bed to increase rapidly, resulting in a decrease in breakthrough time and retention of the

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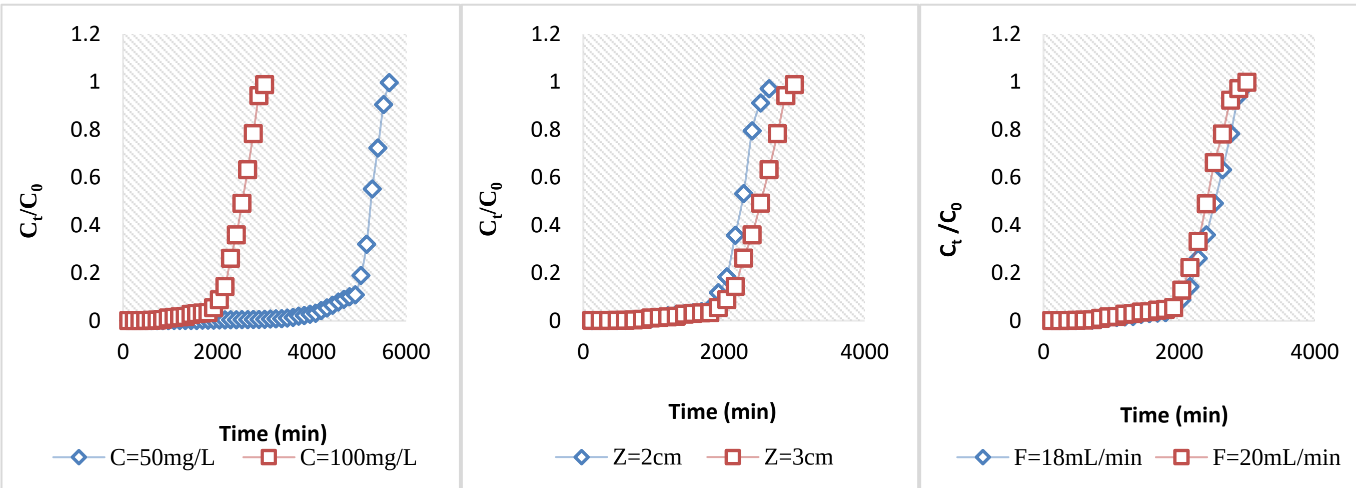
dye in the bed. Similar results were reported by Attia et al. (Attia et al., 2018) and (Fran Mansa et al., 2021).



(a) (b) (c)

Figure 4. Effects of adsorbate concentration (a), adsorbent bed height(b) and feed rate (c) on the breakthrough curve

Modelling of breakthrough curves

Thomas' model

The parameter values obtained after modelling using the Thomas model are collected in Table 3. The width of the adsorption front requires a long time to reach equilibrium in the packing layers. The values of the adsorbed amount under different conditions show that increasing the feed rate decreases the adsorption capacity of the BM and increases the Thomas adsorption constant (KTH). However, varying the concentration of BM from 50 to 100 mg. L-1 results in a decrease in the adsorption capacity from 334.7 to 291.36 mg.g-1 and a small increase in adsorption constant from 3.6 to 3.7 mL/min/mg (KTH). The values of the correlation coefficients R2 are higher than 0.90 except for the concentration of the dye equal to 50mg/L which has a determination coefficient R2 equal to 0.88 (figure 5a et 5b). Therefore, the Thomas model describes well all the BM breakthrough curves studied. These results show that the Thomas model is representative of the system studied.

Bohart and Adams model

The parameters obtained from the Bohart and Adams model for the dye studied on the composite material are shown in Table 4. From the results obtained it can be seen that as the bed height increases, the dynamic adsorption capacity for the BM on the adsorbent mixture decreases. However, increasing the concentration of BM from 50 to 100 mg. L-1 also leads to a decrease in the dynamic adsorption capacity from 236.75 to 206.10 mg.g-1 and a small increase in adsorption constant from 3.6 to 3.7 mL/min/mg (KAB).

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The best dynamic adsorption capacities are 279.71 and 236.75 mg. L-1. Thus, the adsorption capacity decreases and the adsorption rate constant increases with increasing dye feed rate. The values of the correlation coefficients R2 are higher than 0.90 except for the concentration of the dye equal to 50mg/L which has a coefficient of determination R2 equal to 0.88 (figure 5c et 5d). Then the Bohart and Adams model can be used to study the BM breakthrough curves.

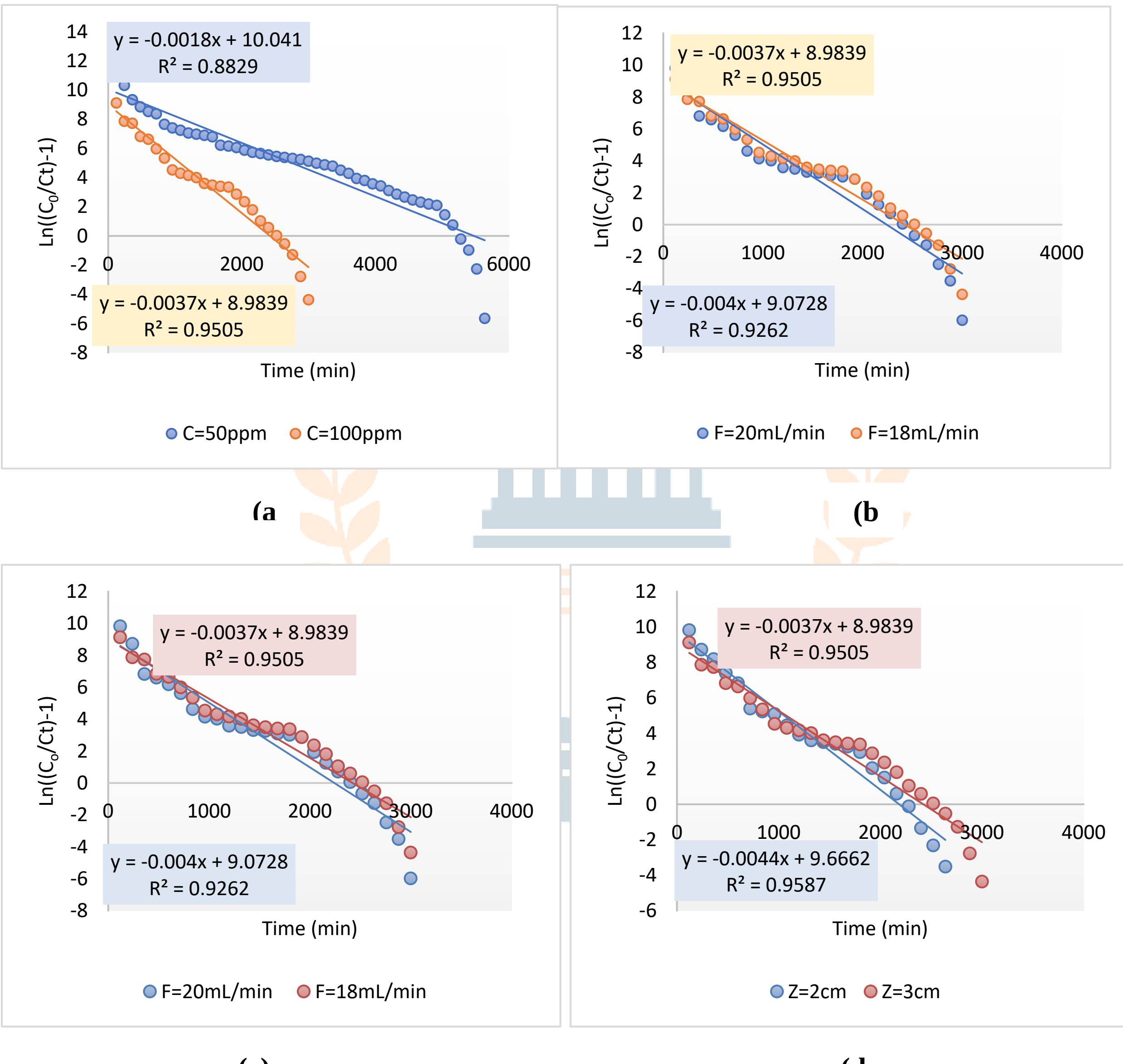

Figure 5. Modelling the variation of the initial concentration (a) and the feed rate (b) by Thomas' model, the variation of feed rate (c) and height bed (d) by Bohart and Adams model

Table 3 : Parameters of the Thomas model

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Z (cm)	C_0 (mg. L^{-1})	F ($L.min^{-1}$)	q_0 (mg.g^{-1})	K_{TH} (mL/min/mg) (10^{-5})
3	50	0.018	334.7	3.6
3	100	0.018	291.36	3.7
3	100	0.02	302.43	4

Table 4 : Parameters of the Bohart and Adams model

Z (cm)	C_0 (mg.L^{-1})	F ($L.min^{-1}$)	N_0 (mg.L^{-1})	K_{AB} (mL/min.mg) ($\times 10^{-5}$)
3	50	0.018	236.75	3.6
3	100	0.018	206.10	3.7
2	100	0.018	279.71	3.7
3	100	0.02	207.19	4.4

CONCLUSION

The objective of this study was to investigate the removal of methylene blue by an adsorption process on a fixed bed column of 3cm diameter and 1m height. The dynamic study carried out on the latter using activated titaniferous sand and attapulgite as adsorbents, allowed the determination of model parameters for a given bed height, initial concentration and flow rate. The results of the characterization showed that attapulgite has a much higher specific surface area than titaniferous sand with values of 70.68 m2. g-1 and 13.4 m2.g-1 respectively. Attapulgite is more porous than titaniferous sand with respective values of 72.5% and 38.33%. On the other hand, various mathematical models such as Bohart-Adams and Thomas were used to model the breakthrough curves. They showed that increasing the initial concentration from 50 to 100 mg. L-1 leads to a decrease in the dynamic adsorption capacity from 236.75 to 206.10 mg. g-1 with a virtually constant adsorption constant (KAB). The best dynamic adsorption capacities are 334.7 and 291.36 mg.g-1 with an almost constant adsorption constant (KTH). In the nutshell, fixed bed columns based on a mixture of titaniferous sand and attapulgite are feasible for the removal of dye molecules from industrial effluents. This mixture is reusable, biodegradable and inexpensive. This offsets the additional treatment cost of converting agricultural waste into adsorbents.

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