Indian Journal of Chemical Technology
Vol. 28, July 2021, pp. 412-420

Kinetics and Isotherms modeling of methylene blue adsorption by Black Carbon using the shells of apricot kernels

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Received 22 February 2021; accepted 22 April 2021

Shells of apricot kernels have taken chosen to fabricate the activated black carbon to eliminate the basic dye (methylene blue) from aqueous solutions. The results show the effects of various working conditions counting initial dye concentration (0–25 mg/L), adsorbent dose (0.1–3 g/L), temperature (20–45°C) and *p*H (2–12) on the uptake of methylene blue were investigated in this experiment-lab study. The adsorption of cationic dye favoured at low temperatures near to the neutral and acids *p*H for the aqueous solution. Three models have been employed to describe the experimental data for the adsorption isotherm: Langmuir, Freundlich, and Temkin equations. Langmuir isotherm model shows the best results, the thermodynamic parameters such as ΔG, ΔH, and ΔS for the adsorption of methylene blue. The elimination kinetics of methylene blue on black carbon treated at 600°C have been examined by using the pseudo-first-order, pseudo-second-order model equations and intra-particle diffusion, and the pseudo-second-order model was found to define the adsorption process better than the pseudo-first-order equation.

Keywords: Adsorption mechanism, Black carbon, Isotherms models

Environment pollution is one of the major problems for the human, animals, and plants, this pollution coming from solids, liquids and gas effluents. From the dyeing and finishing process in the textile industry are known to contain colour[1]; the wastewater needs to be treated prior to discharge by effectively removing dye colour in order to protect the environment and as per the statutory guidelines[2].

The main treatment technologies to remove dyes from are: biological treatment, ion-exchange, coagulation/ flocculation, micro-electrolysis, Fenton process, advanced oxidation process, ozonization, adsorption, membrane filtration, photo catalysis[3-6]. Adsorption process is also one of the most effective and low-cost treatment process to remove dyes in wastewater[7], the major advantages of adsorption is to control water pollution and less invested in terms of initial development cost, simple design, easy operations free from generation of toxic substances[7-10].

Recently, activated carbons from agricultural waste are widely used as adsorbents in wastewater treatment, which proved by many races, removal of dyes by activated carbon is economically favourable and technically easier[11,12]. The aim of this work is to study the ability of the *black carbon* prepared from shell of apricot kernels to remove methylene blue (MB) dye from aqueous solution. Several parameters have effect on the adsorption performance for this reason this paper study the effect of different parameters such as initial dye concentration, adsorbent dose, *p*H, temperature and contact time , moreover kinetic and equilibrium modelling to fit experimental data and the adsorption thermodynamic.

Experimental Section

Materials and methods

Chemicals

All the chemicals used in this work are: Methylene blue (100 mg/l, *p*H=6. 8) of distilled water. The different *p*H of stock solutions was adjusted to target values by using 0.1 N HCl and 0.1 N NaOH.

Methylene blue was used as a surrogate indicator to simulate industrial wastewater in order to evaluate the adsorption capacity of this activated black carbon in the present study.

The dye of methylene blue with commercial characteristics without further purification, with and a molecular formula of $C_{16}H_{18}N_3SCl$ and molecular weight of 320 g/mol and the CI number of 52015 was chosen as the adsorbate.

Stock solution (100 mg/L) was prepared by dissolving MB in distilled water.

Working solutions were obtained by diluting the commercial solution with distilled water to achieve the desired concentration.

Carbonization of raw material

Shells of apricot kernels used as raw material; which collected from N' gaous town east Algeria, apricot kernels was frequently washed with distilled water to remove the dust, and other impurities. Then dried in the oven at 100°C for 24 h to remove the water, and carbonized for 3h at 600°C, finally the activated black carbon sieved to discrete particle sizes.

Characterization of the sample surface

The techniques used to characterize the activated black carbon surface are: Scanning electron microscopy, XRD patterns, Fourier transform infrared (FTIR) spectroscopy, and pH point of zero charges (pH PZC).

Scanning electron microscopy

The Scanning electron microscopy illustrates the surface physical morphology of activated black carbon.

X-ray diffraction (XRD)

X-ray diffraction (XRD) analysis of the activated black carbon sample was carried out on an equipped name in the 2 angle ranging from 5 to 80.

Fourier transforms infrared (FTIR) spectroscopy

Fourier transforms infrared (FTIR) spectroscopy measurements were conducted via potassium bromide tabulating technique using 6700 FTIR technique Spectrometer over the wave range from 4000 to 400 cm^{-1}.

p*H *point of zero charges (pH ZPC)

The experimental protocol for determining the *p*H point of zero charge is as follows:

50 mg of the adsorbent is added to 50 mL of water at different *p*H ranging from 2 to 12, the *p*H is adjusted by adding HCl or NaOH to 0.01 M. the adsorbent mass is agitated for 24 hours.

Adsorption experiments

Adsorption of methylene blue (MB) on activated black carbon was carried out using a batch experiments method, using a set of 600 mL Erlenmeyer flask containing 0. 25 g of adsorbent and 250 mL of dye (MB) solution in each flask at an fixed initial concentration (15 mg/L) with a constant solution pH, The flasks were then placed in a shaker at 200 rpm and the temperature of 20°C. The experience was examined at a fixed time (2h), and the solution was separated from the adsorbent by filtration then followed by centrifugation.

Dye concentrations in the supernatant solution were estimated by measuring absorbance at maximum wavelengths of MB dye with lambda 20 UV, visible spectrophotometer and computing from the calibration concentration curves. The concentration of dye methylene blue was measured with a UV spectrophotometer at 665 nm.

The amount of adsorbed dye q_t (mg/g) at a different time was calculated as follows:

$$q_e = \frac{(C_0 - C_e) \times V}{m} \quad \dots (1)$$

where C_0 and C_e (mg/L) are the initial and equilibrium concentrations of dye (MB) respectively, (L) is the initial solution volume and m is the mass of adsorbent used.

The percentage elimination of dye was calculated as follows:

$$E\% = \frac{(C_0 - C_e)}{C_0} \times 100 \quad \dots (2)$$

Modeling of the isotherms of adsorption

Model of Freundlich

According to Van Bemmelen (1888) and Freundlich 1909[13- 16], the first model is empirical and is based on the relationship between the quantity adsorbed q_e and the concentration in the aqueous solution of the solute C_e at equilibrium, according to the following relationship[17].

$$\frac{X}{m} = K_f C^{1/n} \quad \dots (3)$$

C_e: residual adsorbate concentration at equilibrium (mg/L)

K_f, n: Freundlich constants.

The linear form of this equation allows deducing the slope of the mean line, equal to 1/n, and the ordinate at the origin which is equal to Ln (K_f).

$$Ln\, q_e = Ln\, K_f + \left(\frac{1}{n}\right) Ln C_e \quad \dots (4)$$

Model of Langmuir

Established in 1918, this single-layer adsorption model is based on assumptions the following [18].

$$q_e = \frac{q_m K_L\, C_e}{1 + q_m K_L} \quad \dots (5)$$

The Langmuir isotherm model in the linear form can be presented as:

$$\frac{C_e}{q_e} = \frac{C_e}{q_{max}} + \frac{1}{bq_{max}} \quad \ldots (6)$$

where q_e is the equilibrium methylene blue concentration adsorbent (mg/g), C_e is the equilibrium methylene blue concentration in solution (mg/L), q_m is the monolayer capacity of the adsorbent (mg/g), K_L is the Langmuir adsorption constant (L/mg), $q_{\max}$ is the Langmuir constant related to the maximum monolayer adsorption capacity (mg g^{-1}), and *b* is the constant related the free energy or net enthalpy of adsorption (L mg^{-1}).

The Langmuir equation is applicable to homogeneous sorption, where the sorption of each sorbate molecule onto the surface has equal sorption activation energy.

The essential features of the Langmuir isotherm model can be expressed in terms of 'R_L' a dimensionless constant, separation factor or equilibrium parameter, which is defined by the following equation [19].

$$R_L = \frac{1}{1+bC_0} \quad \ldots (7)$$

where C_0 is the highest concentration (mg/L), K_L is the Langmuir constant (L/mg). The value of R_L indicates the type of isotherm to be irreversible (R_L = 0), favourable (0 < R_L< 1), linear (R_L =1) or unfavourable (R_L> 1).

Model of Temkin

The derivation of the Temkin isotherm assumes that the derivation of the Temkin isotherm assumes that the lowering of the adsorption heat is linear rather than logarithmic and the adsorption is characterized by uniform distribution of the energies of the bonds up to a certain maximum binding energy, the Temkin isotherm is represented by the following equation [20- 21].

$$q_e = \frac{RT}{b_T} Ln(K_T C_e) \quad \ldots (8)$$

qe: quantity of dyes adsorbed (mg/g),
C_e: solute concentration at equilibrium (mg/L),
R: universal constant of perfect gases (J. mol^{-1}. k^{-1}),
T: Absolute temperature (K),
B_T: variation of adsorption energy (J. mol^{-1}. g. mg^{-1}),
K_T: equilibrium constant (L. mg^{-1}).

Kinetic adsorption models

To determine adsorption rate constants, kinetic data are analyzed using two kinetic models namely the pseudo-first-order and pseudo-second-order model.

Model of Lagergren pseudo first order which is expressed as:

$$Log\frac{q_e - q_t}{q_e} = -\frac{K_1 t}{2.3} \quad \ldots (9)$$

And the pseudo-second order model which is expressed as:

$$\frac{t}{q_t} = \frac{1}{2K_2 q_e^2} + \frac{t}{q_e} \quad \ldots (10)$$

The most representative data model was selected based on the correlation coefficient R^2.

Intraparticle diffusion model

The intraparticle diffusion equation is expressed as Weber and Morris [21]:

$$q_t = K_p t^{0.5} + C \quad \ldots (11)$$

where k_p(mg g^{-1} $min^{-1/2}$) is the rate constant of intraparticle diffusion model. The values of k_p and *C* can be determined from the slope and intercept of the straight line of q_t versus $t^{1/2}$.

Results and Discussion

Activated black carbon characterization

Table 1 and Figures 1a, 1b, 1c, 1d and 1e bellows shows some characteristics of the black carbon prepared from the shells of apricot kernels (BCSAK):

Fourier transforms infrared (FTIR) spectroscopy

The infrared spectrum of the black carbon prepared from the shells of apricot kernels (BCSAK) presents a band located at 3417 cm^{-1} can be attributed to the group (O-H) of vibration of water molecules. The strong band of the grouping (C=0) located at 1740 cm^{-1}, the asymmetrical and symmetrical vibration bands of the groupings (C-H) observed in the range between 2930 to 2850 cm^{-1}, respectively (Fig.1a) .

Table 1 — Characterization of (BCSAK)

Characteristic	
Ash rate	5 .1
Humidity	0.1814
pH(PZC)	6.1
Oxygen (mass weight %)	33.54
Carbon (mass weight %)	57.09
Iodine number (mg/g)	2864.5

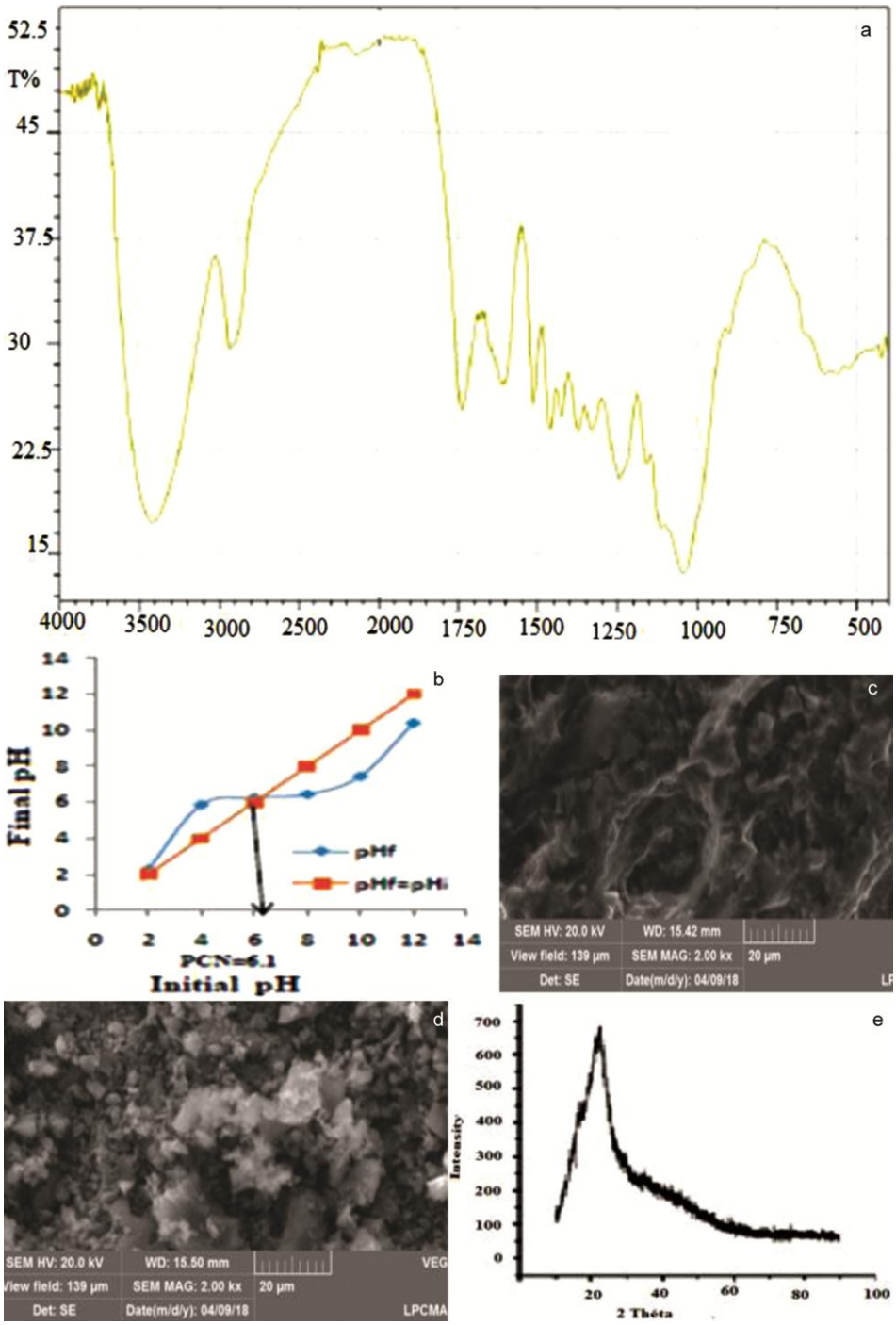

Fig. 1(a-e) — (a) FT-IR Spectrum of BCSAK; (b) *p*H Point zero charge (pH_{PZC}) of BCSAK; (c) Scanning electron microscope of BCSAK; (d) Scanning electron microscope of BCSAK after adsorption of MB and (e) x-ray diffraction profile of BCSAK

p*H *point of zero charges (pH ZPC)

Based on Fig. 1b, there is a quasi stability of the final *p*H in the initial *p*H range of 5 to 8, so the bearing formed by this constancy of the final *p*H can be attributed to the change of the surface charge of the adsorbent from positive to negative and vice versa, pH_{PZC} value found in this raw carbon equal 6.1.

The Fig. 1c presents the micrographs of the SEM of the raw carbon, homogeneity of the distribution of the grains is observed and uniform over the entire surface of this black carbon prepared from the shells of apricot kernels (BCSAK), the Fig.1d reflects the surface nature of the adsorbent after adsorption; the sample micrographs demonstrate that the pores as

well as the cavities are unavailable by the dye and that the surface is practically arbitrary.

X-ray diffraction (XRD)

X-ray diffraction patterns were obtained using a Philips PW 1730 diffract meter equipped with Cu-Ce radiation (40kV, 30 am).

The analysis of black carbon sample BCSAK was carried out on an equipped in the 2ϕ angle ranging from 5 to 80.

Absence of the characteristic peaks in the adsorbent reveals the amorphous state characterized by an irregular or random structure of the atoms in the sample.

The x-ray diffraction profile of BCSAK presented in the Fig. 1e shows that this black carbon BCSAK has an amorphous structure.

The effect of the physicochemical parameters

The study of the adsorption of MB dye on black carbon prepared from the shells of apricot kernels (BCSAK) requires knowledge of the parameters that influence this phenomenon, namely: Initial concentration, adsorbent dosage, *p*H, stirring time.

Effect of initial BM concentration

The initial dye concentration effect depends on the direct relation between the concentration of the dye and the existing sites on an adsorbent surface.

It is observed that the increase in the initial concentration leads to an increase in the quantity of adsorption of BM until the stabilization of the phenomenon (Fig. 2a). This is may be due to the high driving force for the mass transfer at a high initial dye concentration; which most researchers are in agreement for these resultants with the same conclusion[22- 24].

Effect of dosage of adsorbent

Figure 2b illustrates the increase in the adsorption efficiency of BM on the activated black carbon with the increasing in the mass of adsorbent, this can be explained by the increasing in the activated black carbon sites, subsequently it is observed that the stabilization of the quantity adsorbed for the raw carbon, due to the reversibility of the interaction between the adsorbent and the adsorbent, this stage expresses the state of equilibrium between carbon and organic pollutants (BM), we note that an optimal mass of 0.5 g of carbon is sufficient to reach equilibrium, similar results were reported by other studies [25].

Effect of pH and adsorption mechanism

The *p*H effect on the methylene blue adsorption by BCSAK can be explained on the basis of pH_{pzc}, for which the charge of the adsorbent surface is positive lower than pH_{pzc} of BCSAK (pH_{pzc} = 6.1). As the pH increase, the number of negatively charged sites augments and improves the methylene blue adsorption by electrostatic attractions.

The results in Fig. 2c show that the amount adsorbed is maximum at acidic pH (pH = 2.01) and believes for a neutral *p*H (*p*H=7). The surface of the carbon is positive and the molecules of the BM dye are cationic in solution are negatively charged with a removal at a rate equal to 100%, this may be explained that The adsorption mechanisms are mostly owing to the creation of hydrogen bonds, hydrophobic exchanges, and electrostatic forces of the pollutants with the raw carbon surface, this has furthermore established by the various studies carried out on the different types of inactivated and activated carbon[26-29].

Effect of time of contact

Figure 2d shows that, the maximum adsorption percentage is reached after a contact time of 10 min of agitation and corresponds to a rapid ion exchange, and the quantity of the BM fixed evolves towards a maximum value reaching 97% for black carbon.

An improvement is seen in the BM removal percentage in the presence of black carbon; this is due to the presence of the negatively charged active sites available on the surface of the adsorbent material.

Temperature is a significant controlling factor in the real application of adsorbent for the dye removal process[29]. The effect of temperature on methylene blue adsorption, were carried out at different temperatures 25, 30, 35 and 40°C. Fig. 2c presents the influence of temperature variation on methylene blue adsorption. The increase of the temperature in the range studied for the MB causes a small decrease in the adsorption capacity of the adsorbate on BCSAK at equilibrium. This small decrease means that the adsorption process of the adsorbate on CSAK is exothermic. It is therefore found that there is not a great difference between the maximum amounts adsorbed at different temperatures, this is shown essentially that the increase in temperature gently influences the adsorption process.

Isotherms and kinetic adsorption models

The isothermal adsorption data shown in Fig. 3 are fitted to obtain the Langmuir, Freundlich and Temkin isotherm model parameters. The parameters

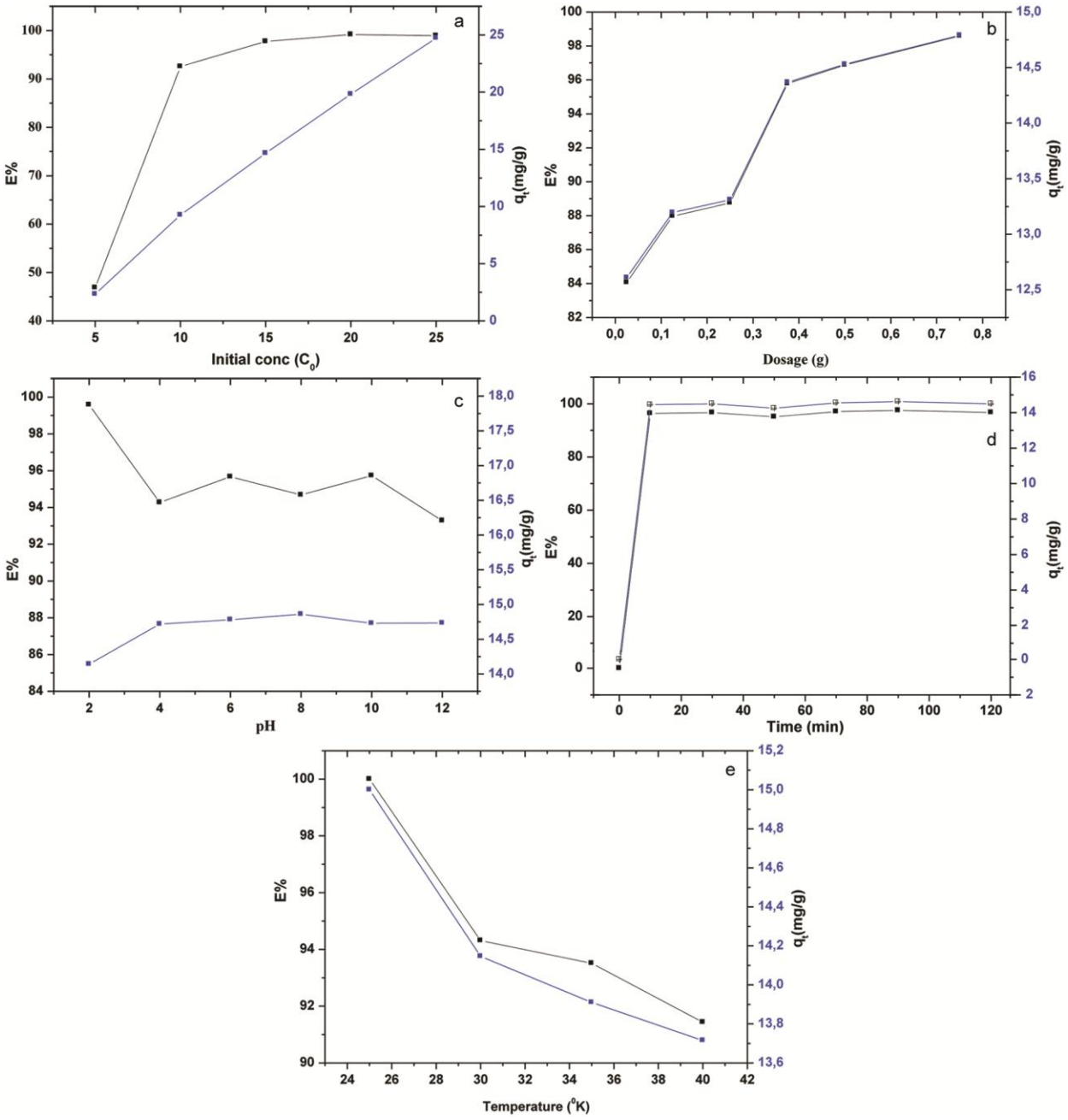

Fig. 2(a-e) — (a) Effect of initial concentration on removal of MB on BCSAK (*p*H=6, temperature 25°C, stirring rate 200 rpm and m=0.25g); (b) Effect of sorbent dosage on removal of MB on BCSAK (C_0= 15 mg/L, *p*H=6, temperature 25°C and stirring rate 200 rpm); (c) Effect of the solution *p*H on the adsorption of MB on BCSAK (C_0 =15 mg/L, temperature 25°C, stirring rate 200 rpm and m=0.25g); (d) Effect of the contact time on the adsorption of MB on BCSAK (C_0 =15 mg/L, *p*H=6, temperature 25°C, stirring rate 200 rpm and m=0.25 g) and (e) Effect of temperature on the adsorption of MB on BCSAK (C_0 =15 mg/L, *p*H=6, stirring rate 200 rpm and m=0.25g)

are presented in Table 2. higher values of R^2 for the three isotherms indicate that the adsorption of MB could be well described by the linear, Langmuir Freundlich and Temkin isotherms; this has also been found in the various studies carried out on the different types of coals [28-29].

Adsorption kinetics

From the results obtained indicated in the three Fig. 4a, 4b, 4c and Table 3, it is noted that the second-order pseudo model is the most reliable for determining the order of the kinetics of adsorption of methylene blue by black carbon have a correlation

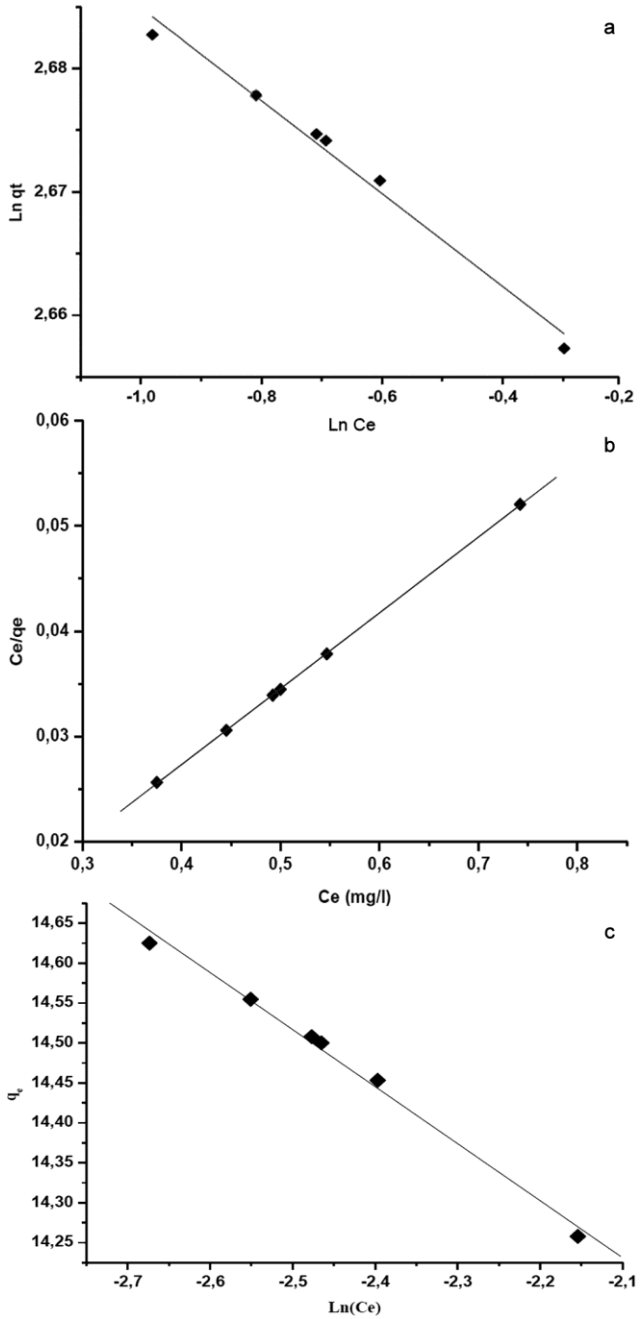

Fig. 3 — Linear transformation of the adsorption Isotherm using (a) the Freundlich model, (b) The Langmuir model and (c) Temkin model.

coefficient exceeding 0.99.So the kinetic model of the pseudo-second-order is very suitable for the adsorption of BM on the raw carbon this has also been found by the various studies carried out on the different types of coals [24-25].

Intraparticule diffusion model

The application of the internal diffusion model does not give a straight line, therefore this model is not limiting of the dye adsorption process (methylene blue) on the black carbon, and other mechanisms may be involved, for example: Fixing the dye by hydrogen bonds, electrostatic forces and ion exchange, similar results were reported by other studies [30]

Table 2 — Values of the coefficients of the three linearized Kinetic models with experimental results

Isotherm	Parameters
Freundlich	
n	-0.027
K	14
R^2	0.985
Langmuir	
q_m(mg/g)	13.88
b(l/mg)	48.35
R_L	0.0008
R^2	0.999
Temkin	
A	12.731
B	-0.71
R^2	0.992

Table 3 — Kinetics model parameters

Model	Parameter	
Pseudo first-order	q_e(mg/g)	-
	K_1	-
	R^2	0.10
Pseudo second order	q_e(mg/g)	14.85
	K_1 (g/min.g)	0.019
	h	4.18
	R^2	1
Intra particule diffusion	C(mg/g)	14.36
	K_p ($mg.g^{-1}min^{-0.5}$)	0.016
	R^2	0.013

Thermodynamic parameters and activation energy

The thermodynamic parameters including the standard enthalpy change (ΔH^0, Kj mol^{-1}), standard Gibbs free energy change (ΔG^0, Kj mol^{-1}) and standard entropy change (ΔS^0, Kj $mol^{-1}K^{-1}$) has an important role to examine the feasibility, spontaneity and nature of adsorption process.

Parameters can be calculated by:

$$\Delta G^0 = -RTLnK_c \quad \ldots (12)$$

$$K_c = \frac{q_e}{C_e} \quad \ldots (13)$$

$$LnK_c = \frac{\Delta S^0}{R} - \frac{\Delta H^0}{RT} \quad \ldots (14)$$

$$\Delta G = \Delta H - T\Delta S \quad \ldots (15)$$

For the black carbon (BCSAK) adsorbent, the negative values of ΔH and ΔS are -83.7 and -0.251 Kj mol^{-1}, respectively indicate the exothermic and decreased disorder at the solid-liquid interface

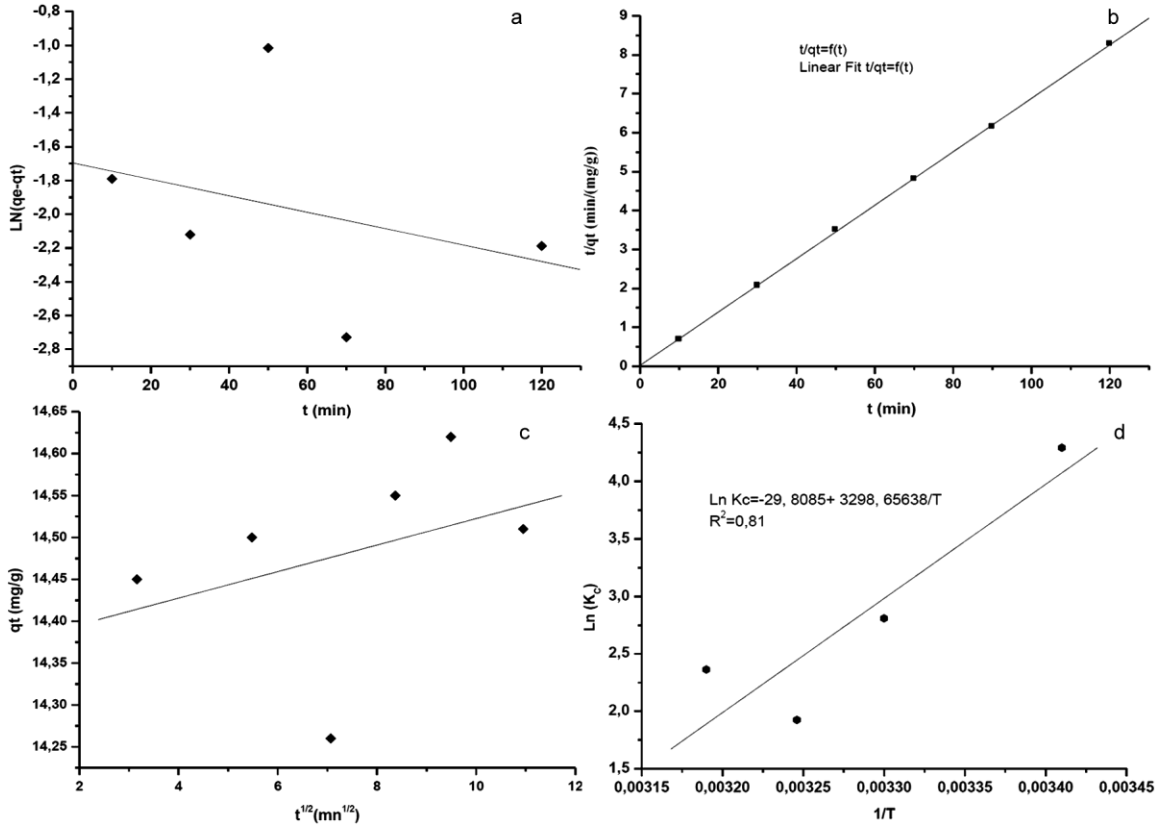

Fig. 4(a-d) — (a) Fit pseudo-first order of adsorption of MB on black carbon (BCSAK) at 25°C; (b) Fit pseudo-second order of adsorption of MB on black carbon (BCSAK) at 25°C; (c) Intra particule diffusion model plots and (d) Van't Hoff equation for adsorption of MB on black carbon (BCSAK) at different temperatures.

Table 4 — Thermodynamic parameters for the adsorption of MB on black carbon (BCSAK)

Adsorbent type	ΔH (Kj mol^{-1})	ΔS (JK^{-1}. mol^{-1})	ΔG(Kj mol^{-1})			
			298 K	303K	313K	313K
Black carbon (BCSAK)	-83 .7	-251.8	-8.7	-7.4	-6.2	-4.9

Fig. 4b and Table 4. In addition the adsorbent exhibited spontaneous nature of the adsorption process with negative values of Gibbs free energies during MB dye adsorption, similar results were reported by other studies[31]; on the other hand several researchers have found that the adsorption of methylene blue onto other adsorbents is endothermic[32-34].

Conclusion

In this work, the ability of black carbon prepared from the shells of apricot kernels (BCSAK) sorbent to remove MB from aqueous solution was investigated.

Experimental results show that a black carbon (BCSAK) was important for the elimination of MB from aqueous solution. Since BCSAK used in this work is free, abundantly and locally accessible, the resulting sorbent is expected to be economical, feasible for removal of basic dye from aqueous solution; results obtained were modeled using three isotherm models: Langmuir, Freundlich and Temkin.

Equilibrium isotherms were well described by the Langmuir equation, giving a maximum adsorption capacity of 13. 88 mg/g at 25°C. The adsorption kinetics can be well described by the pseudo-second - order model equation.

The negative free energy $\Delta G°$ and negative enthalpy $\Delta H°$ indicate that the adsorption of MB onto BCSAK is spontaneous and exothermic above the studied temperatures range.

The black carbon (BCSAK) appeared to be appropriate for the elimination of MB from aqueous solutions.

This study in batch mode leads to optimistic results, and we require appreciating the adsorption tests in column mode under the real conditions applicable to the treatment of industrial effluents.

Acknowledgements

The authors would like to acknowledge University of Mohammed Khieder Biskra, for financial and instrumental supports.

Nomenclature

b : Affinity parameter of Langmuir (L/mg).

BCSAK: Black carbon of shells of apricot kernels

$B_{T:}$ Variation of adsorption energy (J. mol^{-1}. g. mg^{-1}),

C : Intraparticle diffusion constant

C_o : Initial solute concentration (mg/L).

C_e: Concentration of solute at time t (mg/L).

E : Elimination rate (%).

h : Initial rate of adsorption(mg/g.min).

h : Initial rate of adsorption(mg/g.min).

k : Freundlich adsorption capacity (mg/g).

Kc : Distribution coefficient (KJ mol^{-1})

$K_{f\ :}$ Freundlich constants.

K_L : Langmuir adsorption constant (L/mg)

K_p *:* The rate constant of intraparticle diffusion model (mg g^{-1} $min^{-0.5}$)

$K_{T:}$ Equilibrium constant (L. mg^{-1}).

$K_{1:}$ Rate constant of the pseudo first order adsorption ($L{\cdot}min^{-1}$).

$K_{2:}$ Second order rate constant of the pseudo-order adsorption ($g{\cdot}mg^{-1}{\cdot}min^{-1}$).

m: Mass of the adsorbent (g).

MB : Methylene blue

n : Freundlich adsorption intensity.

qe : Amount of solute adsorbed per unit weight of adsorbent at equilibrium (mg / g)

q_m : Maximum capacity (mg / g).

q_t : Amount adsorbed at time t (mg/g)

R : Elimination rate (%).

R : universal constant of perfect gases (J. mol^{-1}. k^{-1}),

$R_{L:}$ Separation factor.

$R^{2:}$ Correlation coefficient.

t : Time (mn)

T : Temperature (^{0}C or ^{0}K)

V: Volume of the solution (L).

$\Delta G^{0:}$ Standard Gibbs free energy change

$\Delta H^{0:}$ Standard Gibbs free energy change

$\Delta S^{0:}$ Standard entropy change

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