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Removal of methylene blue from aqueous solution by loquats nuclei

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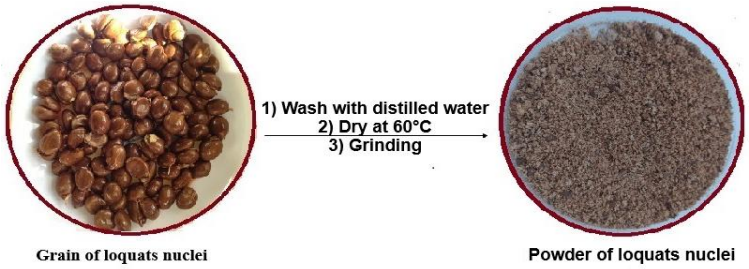
The aim of this paper is to study the performance of loquats nuclei in the removal of methylene blue (MB) from aqueous solutions. To achieve this, we conducted studies on adsorption kinetics, thermodynamics and isotherms. The adsorbent was characterized by X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR) and surface charge measurements. Adsorption tests were carried out by varying different parameters such as MB concentration, adsorbent mass, particle size, contact time and solution pH. The experimental results showed that the adsorption capacity of loquats nuclei for MB can reach up to 90% (q_m=8.728 mg/g). Based on the correlation coefficients R^2, the equilibrium data fitted well with pseudo-second order model (R^2= 0.999) and Freundlich isotherm (R^2=0.993). The adsorption equilibrium study showed that MB is adsorbed in multilayers on heterogeneous surfaces and the adsorption process is controlled by chemisorption. The thermodynamic study indicates that the adsorption of MB was spontaneous, exothermic and leads to an organized surface.

Keywords: Loquats nuclei, methylene blue, adsorption, pseudo-second-order, Freundlich, thermodynamic study.

Graphical abstract



INTRODUCTION

The release of dyes into the environment raises concerns for both toxicological and aesthetic reasons [1]. Industries such as textiles, leather, paper, plastics, etc., are some of the sources of dye effluents [2].

During the past three decades, several physical, chemical and biological bleaching methods have been reported. Adsorption is an economically feasible process for the removal and/or discoloration of dyes from textile effluents.

Researchers have demonstrated that a wide variety of natural materials have the ability to remove large amounts of organic pollutants and heavy metals present in water [3-14]. Currently, activated carbon is considered one of the most versatile adsorbents, and many studies showed its effectiveness; however, its use remains limited due to difficulties of its regeneration and its high cost. An alternative solution is to utilize other efficient and more economical adsorbent materials.

This work falls within this framework. More specifically, we aim to contribute to the recovery of biomass. We have chosen to study the loquats nuclei (agro-food waste and lignocellulosic material). These nuclei can be used for the manufacture of activated carbon [15], bio-adsorbents for dyes [16, 17], substrates of bacterial fermentation [18, 19], for their anti-cancer properties due to their richness in amygdalin [20], and as pesticides [21].

Our focus is on investigating their potential for use in their natural state in the adsorption of methylene blue in aqueous solution. This work is relevant and innovative because it falls within the framework of the valorization of low-cost biomass that could be integrated into the water decontamination sector. In addition, the adsorption process used here is environmentally friendly and

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does not require much energy compared to other processes (coagulation, oxidation, precipitation, filtration, electrochemical processes, etc.)

EXPERIMENTAL

Adsorbent: Loquats nuclei

Loquats nuclei are lignocellulosic materials with fibrous structures, mainly composed of organic and mineral substances (ash) [22]. They are usually accompanied by a certain amount of water [23, 24]. The organic substances consist primarily of four types of polymers, namely cellulose, hemicelluloses, lignin and pectin. The first two polymers are also referred to as carbohydrates or holocelluloses.

The loquats nuclei used in this work are sourced from Berkane region along the Zeggel Valley in Morocco. They were washed several times with distilled water until the smell disappeared and the rinse water was clear, and then dried in the oven at 60°C for 24 h. After grinding, they were sieved to retain only the fraction between 0.250 and 0.315 mm.

Adsorbate: Methylene blue

The adsorbate that we want to remove is methylene blue (MB), an organic compound with the chemical formula $C_{16}H_{18}N_3SCl$ (Figure 1). Its molar mass is 319.85g/mol. Methylene blue belongs to the xanthenes family and is classified as a cationic dye ($C_{16}H_{18}N_3S^+$), which is preferentially adsorbed by the acid sites of the adsorbents. This dye was chosen as a representative model for medium-sized organic pollutants. Methylene blue was used without any purification, and solutions were prepared by dissolving the amounts of the dye in distilled water.

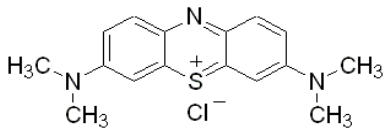


Fig. 1. Chemical structure of MB

X-ray diffraction. The powder diagram of loquats nuclei was recorded using a Siemens D5000 diffractometer with Cu $K\alpha_1$ radiation (λ = 1,5406 Å) operating at 45 kV and 40 mA within the 2θ range of 5-60°. Data were collected at a rate of 0.067°/min at 298K. The XRD patterns were identified by comparison with JCPDS standards.

Infrared spectroscopy. Infrared spectrum of loquats nuclei was collected on a Bruker-Tensor 27 spectrophotometer equipped with an ATR module fitted with a diamond crystal as the attenuated total reflection element. The spectrophotometer scanned the spectral range 4000 - 500 cm^{-1} with a resolution of up to 4 cm^{-1}.

Point of zero charge $pH_{pzc.}$ In order to determine the point of zero charge (pH_{PZC}), we used the same protocol described in the literature [25]. It consisted of placing 50 ml of NaCl (0.01 M) in a closed flask and adjusting the pH to values between 2 and 12 by adding NaOH or HCl solutions (0.1 M). We then added to each vial 0.15 g of adsorbent at room temperature for 48 h, and then the final pH was determined. On a graph of pH_f = f (pH_i), the intersection of the curve with the first bisector gives the isoelectric point.

Adsorption experiments

To follow the kinetics and adsorption isotherms of MB on loquats nuclei, the adsorbent was placed in 50 mL of a solution of methylene blue at different concentrations. The mixture, at a well-determined pH, was stirred at room temperature. The solid was separated by centrifugation and the filtrate was analyzed by measuring the absorbance using a Mapada 1600 UV-visible spectrophoto-meter at the wavelength corresponding to the maximum absorption (664 nm for MB).

The sorption yield was evaluated by determining the adsorption capacity of the adsorbent noted q_a or the retention rate of the adsorbate according to the following equations:

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

$$R\% = \frac{(C_0 - C_e)}{C0} 100 \quad (2)$$

where C_0 and C_e ($mg.L^{-1}$) are the initial and equilibrium concentration of MB solution respectively, V refers to the volume of the solution (L) and m is the adsorbent mass (g).

Kinetic modeling

In order to analyze the adsorption kinetics of MB onto loquats nuclei, different kinetic models have been tested: pseudo-first-order, pseudo-second-order, intra-particle diffusion and Elovich model. These models are represented by equations 3, 4, 5 and 6, respectively [26]:

$$\log(q_e - q_t) = \log(q_e) - \left(\frac{K_1 t}{2{,}303}\right) \quad (3)$$

$$\frac{t}{q_t} = \frac{1}{K_2 q_e^2} + \frac{1}{q_e} t \quad (4)$$

$$q_t = K_i t^{1/2} + C \quad (5)$$

$$q_t = \frac{1}{\beta} \mathrm{Ln}(\alpha\beta) + \frac{1}{\beta} \mathrm{Lnt} \quad (6)$$

where q_e and q_t ($mg.g^{-1}$) are the amounts adsorbed at equilibrium and at time t, respectively. K_1 (min^{-1}),

K_2 ($g.mg^{-1}.min^{-1}$) and K_i ($mg/g\ min^{1/2}$) are the rate constants of the pseudo-first order, the pseudo-second order and the intraparticle diffusion, respectively. C is the intercept. α is the initial adsorption rate constant ($mg.g^{-1}.min^{-1}$) and β is Elovich's parameter ($g.mg^{-1}$) associated with the degree of coverage of adsorbent surface and energy of chemisorption.

Isotherm modeling

For the adsorption equilibrium study, the following models were chosen: Langmuir, Freundlich, Temkin, and Dubinin-Radushkevich. The linear form of the Langmuir isotherm model is depicted by [9]:

$$\frac{1}{Q_e} = \frac{1}{bQ_m}\frac{1}{C_e} + \frac{1}{Q_m} \quad (7)$$

where Q_e is the quantity of MB adsorbed per unit mass of adsorbent at equilibrium ($mg.g^{-1}$), Q_m is the maximum adsorbed quantity ($mg.g^{-1}$), C_e is the MB concentration at equilibrium (mg/L), b is the thermodynamic constant of the characteristic adsorption equilibrium of the adsorbent, depending on the temperature and the experimental conditions ($L.mg^{-1}$).

In the Langmuir model, to determine whether the adsorption is favorable or not, it is imperative to estimate a dimensionless constant called the separation factor or equilibrium parameter, denoted R_L. The equation of R_L is:

$$R_L = \frac{1}{1 + b \times C_0} \quad (8)$$

where C_0 represents the MB initial concentration (mg/L).

If R_L>1, adsorption is unfavorable; if R_L=1, it is linear adsorption; if 0< R_L<1, adsorption is favorable and if R_L=0, adsorption is irreversible.

The Freundlich model is based on an empirical equation that describes the variation of energies with the adsorbed quantity. This distribution of interaction energies is explained by heterogeneity of adsorption sites. This model admits the existence of interactions between the adsorbed molecules. According to the linear form, the Freundlich model can be expressed as [9]:

$$\mathrm{Ln}(Q_e) = \frac{1}{n}\mathrm{Ln}(C_e) + \mathrm{Ln}(K) \quad (9)$$

where K is related to the adsorption capacity ($mg.g^{-1}$) and 1/n represents the empirical parameter of the adsorption intensity.

Temkin equation is based on the effect of some direct adsorbent-adsorbate interactive relation on sorption isotherm. Due to these interactions, the heat of adsorption decreases linearly with the increase in the rate of coverage of the adsorbent's surface.

The linear Temkin equation is [9]:

$$Q_e = \frac{RT}{b_T}\mathrm{Ln}K_T + \frac{RT}{b_T}\mathrm{Ln}C_e \quad (10)$$

where b_T is Temkin isotherm constant (J/mol) and R is the ideal gas constant (R=8,314J/mol.K)

The Dubinin-Radushkevich isotherm, unlike the Langmuir isotherm, does not assume a homogeneous surface or constant adsorption potential. This isotherm model is represented by the following equation [27]:

$$\mathrm{Ln}q_e = \mathrm{Ln}q_m - \beta\varepsilon^2 \quad (11)$$

where Ɛ is the Polanyi potential: Ɛ = RTLn (1 + $1/Ce$). β gives the mean free energy E (Kj/mol) of sorption per molecule of the sorbate when it is transferred to the surface of the solid from infinity in the solution and can be computed using the relationship [28]:

$$\mathrm{E} = \frac{1}{(2\beta)^{0,5}} \quad (12)$$

This parameter gives information about chemical or physical adsorption.

Thermodynamic study

The thermodynamic parameters of the adsorption of MB on loquats nuclei are determined from the study of the adsorption reaction at different temperatures (293, 308, 318, 328 and 333K) using the following equation:

$$\Delta G^0 = \Delta H^0 - \mathrm{T}\Delta S^0 \quad (13)$$

ΔG° is the standard Gibbs free energy (KJ/mol) and T is the temperature (K).

The standard enthalpy ΔH° (KJ/mol) and entropy changes ΔS° (KJ/K.mol) of adsorption can be determined from the following Van′t Hoff equation:

$$\mathrm{Ln}(K_d) = \frac{\Delta S^0}{R} - \frac{\Delta H^0}{RT} \quad (14)$$

with $K_d = q_e/C_e$.

K_d is the distribution coefficient, q_e is the quantity adsorbed at equilibrium (mg/g), C_e is the concentration of the adsorbate in the solution at equilibrium (mg/L) and R is the ideal gas constant [29].

RESULTS AND DISCUSSION

The surface morphology of loquats nuclei was also examined by El Marouani *et al.* [30]. The authors concluded that the material is organized in a few agglomerates of blisters of various sizes and geometries on the surface.

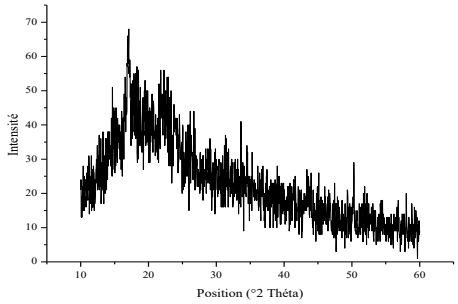


Fig. 2a. X-ray diffraction patterns of loquats nuclei

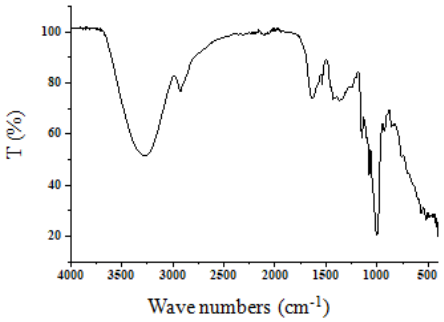


Fig. 2b. Infrared spectrum of loquats nuclei

Point of zero charge (pHPZC). The point of zero charge (pHPZC) is one of the parameters that characterize the adsorbents. It is the pH value at which the surface charge of the adsorbent is equal to zero. If the pH of the medium is lower than pHPZC, the surface of the adsorbent is positively charged. If the pH is higher than pHPZC, the surface is negatively charged. The pHPZC value is obtained from the intersection of the first bisector with the curve giving the final pH as a function of the initial pH. As it can be seen in Figure 3, the pHPZC of loquats nuclei is equal to 6.83.

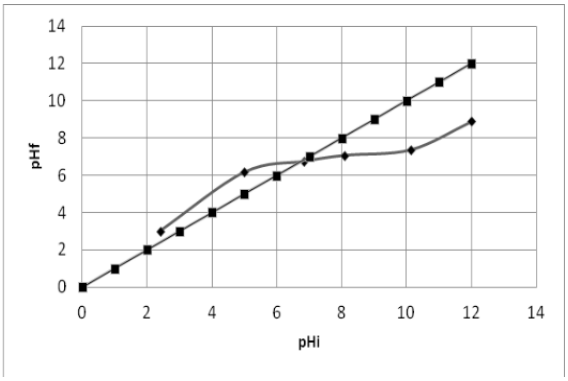


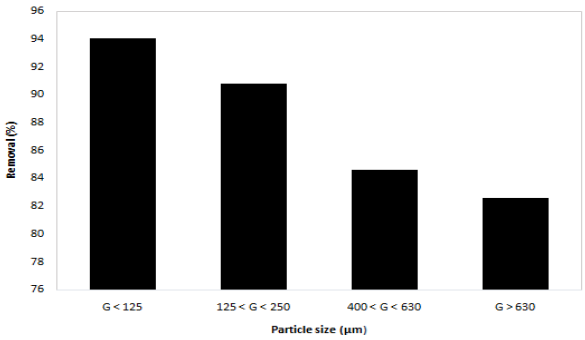
Fig. 3. Point of zero charge (pHPZC) of loquats nuclei

Adsorption of methylene blue by loquats nuclei

Effect of parameters. The adsorption process is controlled by several factors. In this study, the particle size, adsorbent dose, contact time, pH, and initial concentration of methylene blue were taken into account.

a) *Particle size effect.* Particle size is an important factor that affects the adsorption capacity. The retention rate was evaluated for various particle sizes of loquats nuclei (Figure 4). As shown in the figure, the percentage of MB retention decreases as particle size increases. This result can be explained by the fact that smaller particles provide a greater surface area for methylene blue adsorption. Similar results have been reported by other authors [29, 31]. The grain size selected for the remainder of our study was less than 125 µm.

Fig. 4. Particle size effect of loquats nuclei



b) *Adsorbent dose*. The study of the effect of adsorbent mass was carried out by changing the initial mass of the adsorbent from 20 to 120 mg. The volume of the solution was 50 mL (Figure 5).

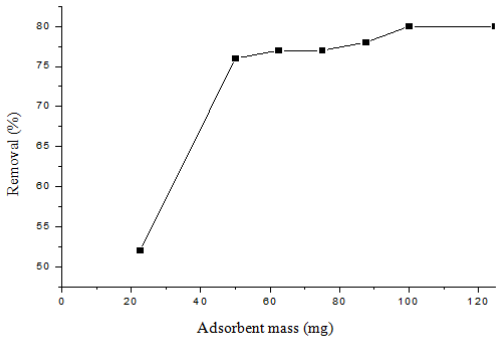


Fig. 5. Effect of the adsorbent mass

From this figure it can be observed that the percentage of MB adsorption increased from 52 to 86% with an increase of the adsorbent dose from 22.5 to 100 mg. This can be explained by the availability of surface sites with the increase of the adsorbent dose [10, 11]. Beyond this value, the retention capacity did not exhibit any variation. Therefore, 100 mg of the adsorbent was selected as the optimal dose.

c) *Effect of contact time*. The effect of contact time on the percentage removal of MB by loquats nuclei was investigated at an initial dye concentration of 5 mg/L and adsorbent dose of 100 mg. The results are presented in Figure 6.

This figure shows that the percentage removal of MB increased from 0% to 87% at 40 min. After this

time, the percentage removal doesn't exhibit significant changes. The adsorption rate is fast at the beginning of the process and becomes increasingly slow until the stirring time equilibrium is reached. The rapid adsorption kinetics during the first minutes of reaction can be attributed to the fact that, at the start of adsorption, the number of active sites available at the surface of the adsorbent material is much greater than that of the sites remaining after 20 min. According to the figure, 40 min is sufficient to achieve high MB removal.

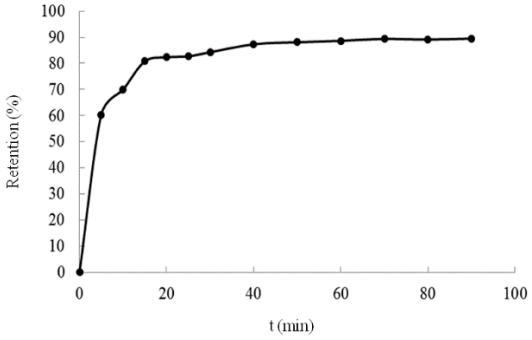


Fig. 6. Effect of contact time

d) Effect of solution pH on dye adsorption. The effect of pH on the MB adsorption onto loquats nuclei was studied by changing pH values from 3 to 12. These values were adjusted by addition of HCl or NaOH. The results are plotted in Figure 7.

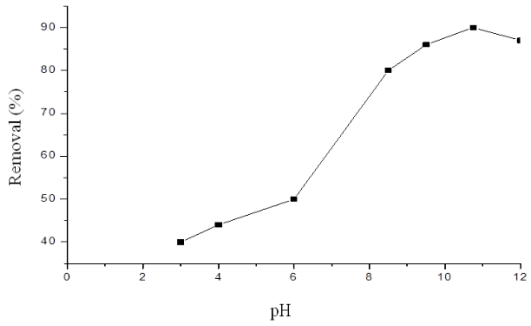


Fig. 7. Effect of pH on MB adsorption by loquats nuclei

From this figure, it can be seen that the adsorption capacity of MB increases when the pH increases, reaching high values in basic medium. This could be explained by the fact that at pH < pH_{PZC}, the surface of the adsorbent is positively charged, which weakens the interaction between methylene blue ions (cationic pollutant) and the adsorbent sites. When pH > pH_{PZC}, the surface of the adsorbent is negatively charged, generating a strong interaction between the dye ions and the surface sites. Similar results were obtained in previous works [11, 32]. Subsequently, the experiments were carried out at the initial pH of 10.75.

e) Effect of methylene blue concentration. The effect of the initial adsorbate concentration on the MB adsorption rate was studied by varying the adsorbate concentration from 5 to 20 mg/L. The pH was maintained at 10.75. The results obtained are shown in Figure 8.

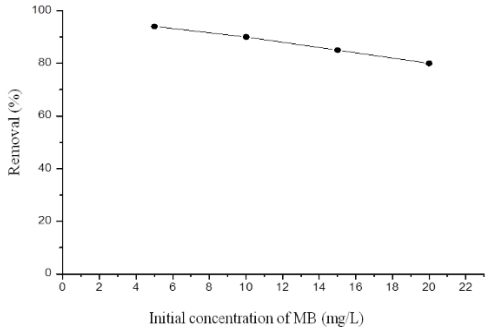


Fig. 8. Effect of the initial concentration of MB

As can be seen, a slight decrease in the retention rate is observed with increasing concentration. This can be explained by the fact that at lower concentrations, all the MB present in the adsorption medium can interact with the bonding sites on the adsorbent surface. The decrease in the retention rate with the increase in concentration is due to the initial saturation of the adsorption sites. The same results were reported previously [10, 11, 33].

Kinetic study. Figures 9-12 show the results obtained for the different kinetic models tested in this study. The values of the correlation coefficients, as well as the corresponding kinetic parameters deduced from the slope and the y-intercept of the various curves, are given in Table 1.

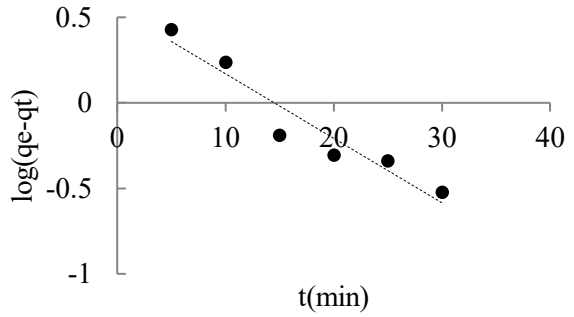


Fig. 9. Pseudo-first order kinetic plots for the adsorption of MB by loquats nuclei

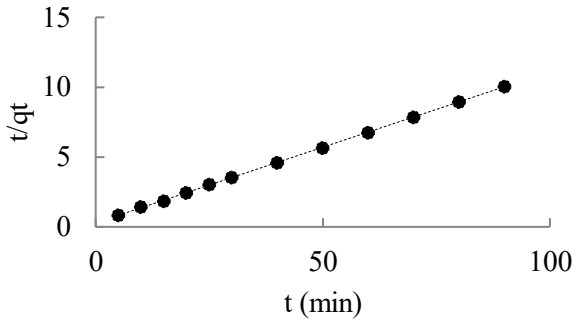


Fig. 10. Pseudo-second order kinetic plots for the adsorption of MB by loquats nuclei

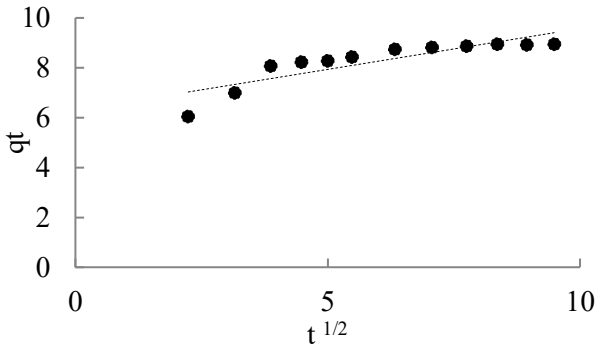


Fig. 11. Intra-particle diffusion model plots for the adsorption of MB by loquats nuclei

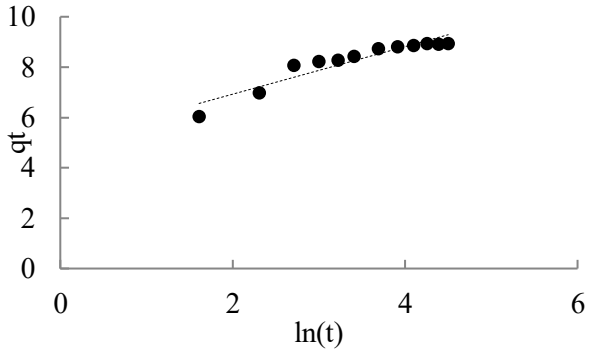


Fig. 12. Elovich equation model plots for the adsorption of MB by loquats nuclei

Table 1. Kinetic parameters for MB adsorption by loquats nuclei

Kinetic models	Parameters	
Pseudo-first-order model	R^2	*0.919*
	k_1 (min^{-1})	0.085
	$q_{e\ calc}$ (mg/g)	3.524
Pseudo-second order model	R^2	*0.999*
	k_2 ($g.mg^{-1}.min^{-1}$)	0.041
	$q_{e\ calc}$ (mg/g)	9.259
	$q_{e\ exp}$ (mg/g)	8.728
	$\Delta q=q_{e\ calc} - q_{e\ exp}$ (mg/g)	0.531
Intra-particle diffusion model	R^2	*0.740*
	K_i ($mg.g^{-1}min^{-0,5}$)	0.327
	C	6.297
Elovich equation	α (mg/g min)	195.547
	β (g/mg)	1.059
	R^2	*0.886*

According to Table 1 and Figures 9-12, we concluded that the mechanism of the MB adsorption onto loquats nuclei can be described by the pseudo-second-order model. Indeed, we noted that the R^2 correlation coefficient is higher than those of the other models and is very close to 1. Moreover, the quantities adsorbed at equilibrium, calculated from this model, agree very well with those obtained experimentally.

Adsorption equilibrium study. The linear representations of the experimental values of MB adsorption on loquats nuclei are shown in Figures 13-16 and the corresponding parameters are given in Table 2.

As shown in Table 2, the Freundlich equation represents the adsorption process very well, the value of the correlation coefficient is close to 1 (R^2=0.993), indicating a very good mathematical fit. The Freundlich model is the most adequate to describe the adsorption of MB on loquats nuclei, suggesting that the adsorption of MB occurs in multilayers on heterogeneous surfaces. In addition, the values of K and n indicate good adsorption capacity.

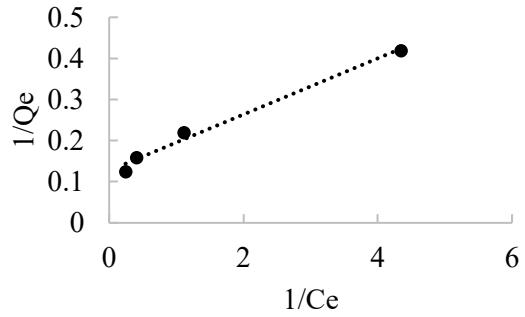


Fig. 13. Linear representation of Langmuir isotherm.

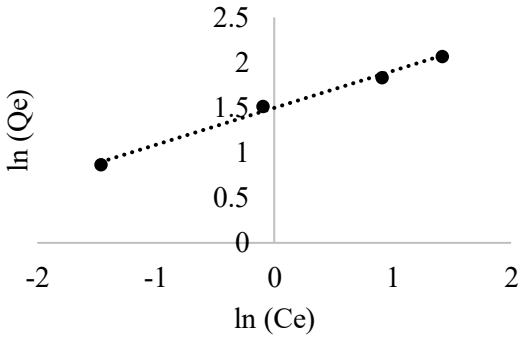


Fig. 14. Linear representation of Freundlich isotherm.

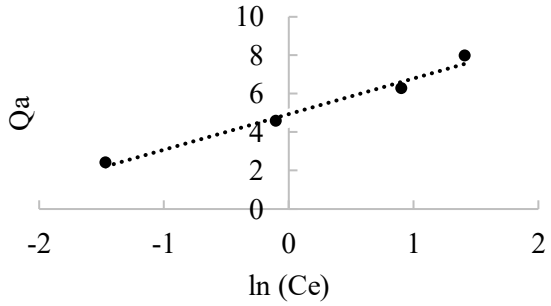


Fig. 15. Linear representation of Temkin isotherm.

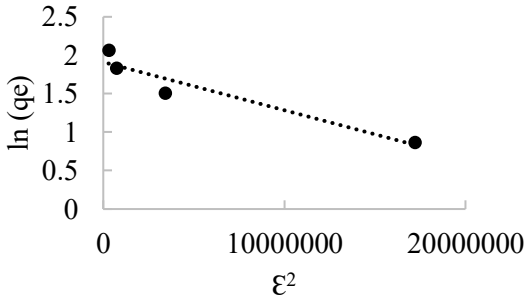


Fig. 16. Linear representation of Dubinin-Radushkevich isotherm.

Table 2. Linear isotherms parameters

Isotherm	Parameters	
Langmuir	$q_{m,cal}$ (mg.g^{-1})	7.874
	B	1.868
	R_L	0.026
	R^2	*0.987*
Freundlich	n	0.409
	K	4.464
	R^2	*0.993*
Temkin	b_T (J/mol)	1.854
	K_T (L/g)	8.360
	R^2	*0.980*
Dubinin-Radushkevich	E (K_J/mol)	11.785.10^6
	q_s(mg/g)	6.753
	β (mol^2/K_J^2)	6.10^{-8}
	R^2	*0.917*

On the other hand, the b_T value estimated by the Temkin model (Table 2) is very low, indicating weak interactions between the adsorbent and the adsorbate. Finally, the high mean free energy value calculated from the Dubinin-Radushkevich model (Table 2) indicates that the mechanism is controlled by chemisorption [5].

Thermodynamic study. The values of ΔH° and ΔS° were obtained, respectively, from the slope and intercept of the curve of Ln(K_d) as a function of 1/T (Figure 17).

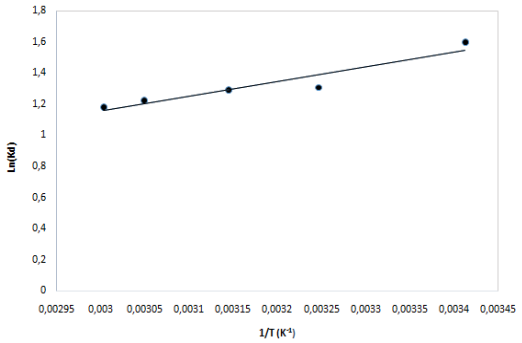


Fig. 17. Representation of Ln(K_d) as a function of 1/T

Table 3. Thermodynamic parameters of the adsorption of MB on loquats nuclei

T(K)	q_e(mg/g)	ΔG° (KJ/mol)	ΔH° (KJ/mol)	ΔS° (J/K.mol)
293	9.0804	-3.889		
308	8.8082	-3.347		
318	8.7902	-3.411	-7.831	-13.857
328	8.7171	-3.335		
333	8.6704	-3.272		

Table 3 gives the thermodynamic parameters of the adsorption of MB on loquats nuclei. The negative values of ΔG° indicate that the adsorption of MB by

the loquats nuclei is spontaneous. Moreover, the negative value of ΔH° confirms that the adsorption process is exothermic. The negative value of ΔS° indicates that the order of distribution of the dye molecules on the adsorbent is important compared to that in the solution. This also suggests a probability of thermodynamically favorable adsorption [34].

Comparison with other adsorbents. The adsorption quantity (qe) of MB by loquats nuclei has been compared with other adsorbents reported in the literature, which are listed in Table 4. From this Table, it can be considered that the loquats nuclei tested here is effective in the removal of MB from aqueous solutions and, moreover, it may be used as an alternative to expensive commercial adsorbents.

Table 4. Comparison of adsorbed quantity (Qe) of MB onto loquats nuclei with those for other reported adsorbents

Adsorbent	Adsorbed quantity qe exp. (mg/g)	Ref.
Pure cellulose	0.11	35
Modified cellulose	0.35	35
Thuya lignin	3.55	8
Algerian data stones	4.9	36
Loquats nuclei	8.728	This study

Adsorption mechanisms. The adsorption mechanism mainly depends on the surface of the loquats nuclei and the structure of methylene blue. Two mechanisms can be proposed in this work: Firstly, the percentage elimination of MB increases considerably in basic medium. In this environment, the adsorbent surface is negatively charged ($pH>pH_{PZC}$), and methylene blue is a cationic dye, suggesting an electrostatic interaction mechanism between the adsorbent and the adsorbate. Secondly, the surface of the loquats nuclei contains alcoholic OH functional groups [30]. Hydrogen bonds can form between the nitrogen of the MB and the hydrogen of the OH groups.

CONCLUSIONS

The present study investigated the efficiency of loquats nuclei in the removal of MB from aqueous solutions. Infrared and XRD analysis were performed to characterize the adsorbent. The point of zero charge (PZC) was also determined.

The adsorption tests were carried out by varying the adsorbent mass, particle size, initial concentration of the adsorbate, contact time and pH of the adsorbate solution. The optimal conditions are: m=100 mg of loquats nuclei, particle size less than 125 µm, 5 mg / L of dye, 40 min of contact time, and pH of dye solution=10.75.

The kinetic study of MB adsorption on this adsorbent is relatively fast, with an equilibrium time of 40 min. The retention of methylene blue is described by pseudo-second order kinetics. Freundlich model best represents the experimental values, indicating that MB is adsorbed in multilayers on heterogeneous surfaces. The thermodynamic study indicates that the adsorption process is favorable, exothermic and spontaneous.

Two mechanisms of MB adsorption by the loquats nuclei were proposed: (i) electrostatic interaction mechanism and (ii) hydrogen bond formation mechanism between the nitrogen atoms of the dye and the OH groups of the adsorbent.

From this study, we can conclude that loquats nuclei are effective biosorbents for the removal of MB from aqueous solution.

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