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Removal of basic yellow dye from aqueous solutions by sorption onto reed as an adsorbent

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ABSTRACT

This work investigates the possibility to use the reed as an adsorbent for removal of basic yellow 28 (BY28), an industrial textile dye from aqueous solution. Batch adsorption experiments at 25 °C and agitation speed of 400 rpm were carried out and the effects of operational parameters including contact time, initial dye concentration, adsorbent dose, as well as solution pH on sorbed amount were investigated and discussed. The maximum uptake of BY28 was about 140 mg/g of adsorbent and it was obtained at pH6, initial dye concentration of 100 mg/L, and adsorbent dose of 0.5 g/L. It was also established that almost 15 min are sufficient to attain equilibrium sorption. Results of batch experiments showed that this adsorbent exhibited high sorption capacities toward BY28. Experimental data were analyzed using pseudo-first-order and pseudo-second-order kinetics and it was found that kinetic followed a pseudo-second-order equation. The equilibrium results have been modeled and evaluated using Langmuir, Freundlich, Sips, and Generalized model isotherms. The Langmuir model provides the best fit of equilibrium data of BY28 and according to this model, the maximum sorption capacity was estimated to181 mg/g of reed. FTIR spectrum of reed confirms the existence of various functional groups such as carboxyl, phenolic, hydroxyl, and amine groups on their surface and these functional groups have always been considered to effectively form some physicochemical interactions, e.g. ion exchange or inner-sphere complex formation, with BY28. Results from this study suggest that reed is an effective adsorbent for the removal of BY28 from aqueous solutions.

Keywords: Reed; Sorption; Basic yellow 28; Batch experiments; Langmuir isotherm; Pseudo-second-order model

1. Introduction

Dyes are widely used in various industries such as textile, plastics, rubber, cosmetics, leather, food, and drug industries. These dyes can contaminate bodies of water, change biological cycles, and affect photosynthesis. Continual contact with dye products can lead to toxic reactions such as skin allergies and can affect respiratory tract function [1].

Furthermore, dyes may exert serious harm to aquatic life and cause severe damages, such as reproductive system, dysfunctions of the kidneys, liver, brain, and central nervous system [2–4]. It is extremely urgent and necessary for us to effectively detect and remove dyes from wastewater.

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The treatment of effluents containing dyes has been realized by several methods such as ultrafiltration through membranes, electrochemical, coagulation/flocculation, biological degradation, and photocatalytic degradation. Most of these methods require high capital costs which are not suitable for small-scale industries but adsorption process is found to be the most efficient, inexpensive, and economically viable [5].

Over the last decades, various materials have been developed as adsorbents for dyes removal from natural waters or industrial wastewaters, but high cost and complex preparation conditions usually limit their practical promotion and application. Recently, lowcost adsorbents developed from natural materials or certain waste materials (or byproducts) from agricultural or industrial activities such as eggshell waste [6], hen feather [7], de-oiled soya [8], apple pomace, wheat straw [9], orange peel [10], banana peel [11], maize cob [12], maize stalk, rice husk [13], peanut hull [14], wood chip [15], sawdust [16], bark [17], leaf [18], coir pith [19], bagasse pith [20], and aquatic plants [21] have gained greater attention due to many obvious advantages such as easy availability, comparable efficiency, resource generation, and pollution abatement.

Basic dyes are highly visible and have high brilliance and intensity of colors. Cationic dyes were intensely used as a model in dye adsorption studies such as crystal violet [22], methylene blue [22–24], basic blue 41 [25], and basic red 46 [26].

Reed is a broad-leafed grass, about 1.5–5 m tall, with feathery flower clusters and stiff, and smooth stems. Dried reed stems have been used for millennia as thatching and construction material, in basketry, for arrows and pens, and in musical instruments. They are also harvested for their cellulose content. This material which is produced in great quantity in our country (Algeria) can represent a true source for the preparation of the sorbent.

In the present work, the sorption of basic yellow dye 28 (BY28) from aqueous solution by using reed was investigated. The sorption capacity of adsorbent was investigated using batch experiments and the influence of contact time, initial dye concentration, adsorbent dose, and solution pH were investigated and the experimental data obtained were evaluated and fitted using adsorbent equilibrium isotherms, and kinetic models.

2. Experimental methods

2.1. Adsorbent and characterization

The reed used in the present study is collected from the Toudja region (Northern Algeria). The collected reed is washed with distilled water several times to remove dirt particles and water-soluble materials. The washing process is continued until the wash water contained no color. The washed materials are then completely dried in an oven at 110 $^{\circ}$ C. The dried reed is ground and sieved and then dried in an air circulating oven at 110 $^{\circ}$ C and stored in a desiccator until use.

A measurement of specific area has been made by nitrogen adsorption at -196 °C using a Brunauer– Emmett–Teller surface analyzer (Nova Station A). The scanning electron microscopy was carried out on a Phillips SEM 501 electron microscope. The nature of surface groups of reed was performed by IR analysis. Fourier transform infrared transmission spectra of biomaterial were obtained through potassium bromide technique on FTIR spectrometer (IR AFFINITY-1 SHI-MADZU) in the wave number range of 4,000–400 cm⁻¹. Reed was mixed with potassium bromide at a ratio of roughly 1/1,000. The amount of acidic and basic groups on the reed was obtained by Boehm titration method [27].

2.2. Adsorbate and analytical measurements

All reagents and materials used in this study are an analytical grade, and distilled water was used to prepare the synthetic dye solution. BY28, a monoazo cationic dye, was supplied by Textile Factory (Alfaditex Remila, Bejaia, Algeria). Its chemical structure is given in Fig. 1. HCl (37% purety) and NaOH (98% purety) were purchased from PROLABO (Paris, France). The concentration of BY28 in the aqueous solution was determined at maximum absorption wavelength (412 nm) using a UV–vis. (A SAFAS SP2000, Monaco— Principality of Monaco) spectrophotometer connected to a PC. The pH measurements were made using HANNA 211R pH meter (Mauritius-Republic of Mauritius).

2.3. Batch sorption procedure

Batch sorption experiments were carried out in a series of stoppered reagent bottles. A weighted amount (0.1 g) of adsorbent was introduced into reagent bottles



Fig. 1. Chemical structure of BY28.

(500 mL) containing various concentrations of BY28. The total volume of the aqueous solution is equal to 250 mL. The solution pH was adjusted to the desired value by adding HCl or NaOH. Then, the bottles were shaken at room temperature $(25 \pm 2 \,^{\circ}\text{C})$ using a mechanical shaker for a prescribed time to attain the equilibrium. Then the solutions were filtered and the final BY28 concentrations were determined by using a UV–vis Spectrophotometer (A SAFAS SP2000, Monaco —Principality of Monaco) connected to a PC at maximum absorption wavelength (412 nm). The effect of concentration (10–100 mg/L), contact time (5–60 min), solution pH (2–10), and adsorbent concentration (0.1–0.5 g) was studied.

Isotherm studies were recorded by varying the initial concentrations of BY28 solutions from 10 to 100 mg/L. A known amount of reed is added into solutions followed by agitating the mixture at 400 rpm till equilibrium.

2.4. Sorption isotherms

Langmuir, Freundlich, Generalized model, and Sips models were tested for equilibrium description. Langmuir equation, based on a theoretical model, assumes monolayer adsorption over an energetically homogeneous adsorbent surface. It does not take into consideration interactions between adsorbed ions. It can be represented by Eq. (1) [27]:

$$q_e = q_{\rm mon} \frac{K_L C_e}{1 + K_L C_e} \tag{1}$$

where C_e is the solute concentration (mg L⁻¹) at equilibrium, q_e is the amount of solute at equilibrium (mg g⁻¹), q_{mon} and K_L are Langmuir constants related to adsorption capacity (mg g⁻¹) and the energy of adsorption, respectively.

Freundlich's equation is an empirical model based on heterogeneous adsorption over independent sites and is given by Eq. (2) [28]:

$$q_e = K_F C_e^{1/n} \tag{2}$$

where K_F (mg^{1-1/n} L^{1/n} g⁻¹) and 1/*n* are the Freundlich constants related to adsorption capacity and adsorption intensity, respectively.

The Freundlich isotherm has been derived by assuming an exponentially decaying sorption site energy distribution.

The Sips model is a combination of the Langmuir and Freundlich isotherm type models. The form of the Sips model is given by Eq. (3) [29]:

$$q_e = q_{\rm mon} \frac{K_L C_e^{1/n}}{1 + K_L C_e^{1/n}} \tag{3}$$

At low concentrations, the Sips equation reduces to a Freundlich isotherm, while at high concentrations it predicts a monolayer adsorption capacity characteristic of the Langmuir isotherm.

The form of the Generalized empirical model is given by Eq. (4):

$$q_e = q_{\rm mon} \left(\frac{K_L C_e}{1 + K_L C_e}\right)^{1/n} \tag{4}$$

This equation reduces to a linear isotherm in the case of low surface coverage and to a Langmuir isotherm when 1/n = 1.

2.5. Sorption kinetics

The controlling mechanism of the metal adsorption process was investigated by fitting first- and secondorder kinetic models to the experimental data. The linearized first-order kinetic model is given as Eq. (5) [30]:

$$\log (q_e - q_t) = \log q_e - \frac{k_1}{2.303}t$$
(5)

where q_t is the amount of adsorbate adsorbed at time $t (\text{mg g}^{-1})$, q_e is the adsorption capacity at equilibrium (mg g^{-1}) , k_1 is the pseudo-first-order rate constant (min^{-1}) , and t is the contact time (min). The linearized form of the second-order kinetic model is given by Eq. (6) [31]:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t$$
(6)

where k_2 is the equilibrium rate constant of pseudo-second-order model (g mg⁻¹ min⁻¹).

3. Results and discussion

3.1. Sorbent characteristics

The structural characteristics of the reed with an average particle size of $356 \,\mu\text{m}$ and the results obtained from the Boehm's method are presented in Table 1. It is seen that the amount of acidic and basic groups are as follows: acidic > basic.

Fig. 2 shows the SEM micrograph of reed sample before dye adsorption. It is clear that reed has

Table 1 Physical and chemical characteristics of the reed

Parameter	Value
Surface area/m ² g ⁻¹	57
Porosity/%	0.236
Particle size/µm	356
Bulk density	0.701
Basic surface functional groups/mmol g ⁻¹	0.97
Acidic surface functional groups/mmol g^{-1}	1.36



Fig. 2. SEM micrograph of reed.

considerable numbers of heterogeneous layer of pores where there is a good possibility for dye to be adsorbed.

The FTIR spectrum of the reed (Fig. 3) displays a number of absorption peaks, indicating the complex nature of the reed. The principal chemical groups of the reed are:

- 3,409 cm⁻¹ corresponds to the vibration of hydroxyls fixed on the surface of reed and water chemisorbed on reed.
- 2,919 cm⁻¹ corresponds to bonds of CH₂- groups and of aldehyde groups.
- 1,732 cm⁻¹ corresponds to the vibration of C=O bond of lactones structures.



Fig. 3. FTIR spectra of the reed (a) and reed loaded with BY28 (b).

- 1,634 cm⁻¹ corresponds to C=O bond and C–N of protein bond.
- 1,606 cm⁻¹ corresponds to C=C bond of woody structures or C=O deformation in carboxylic acids.
- 1,515 cm⁻¹ corresponds to C–N bond or N–H bond of protein.
- 1,161 cm⁻¹ could correspond to C–O–C of polysaccharides.

The FTIR spectra of reed and reed loaded with basic yellow 28 (Fig. 3) display practically the same functional groups.

3.2. Effect of agitation time and initial concentration on BY28 sorption

Fig. 4 shows the effects of agitation time and BY28 concentration on sorption capacity. We note that the sorption of BY28 increases with an increase in agitation time and attains equilibrium at 10 min for different concentrations of BY28. This result is interesting because equilibrium time is one of the parameters for economical wastewater treatment plant applications. According to these results, the agitation time was fixed at 60 min for the rest of the batch experiments to make sure that equilibrium will be reached. It also shows that an increase in BY28 concentration increases the amount of dye uptake per unit weight of reed (mg/g). The increase in loading capacity of the reed related to dye concentration is due to a high driving force of mass transfer.

During our experiments, pH values of equilibrated solutions were often measured and we noted a decrease of this parameter at the end of experiments. This reduction in pH is due to the dissociation of the acid functional groups of the reed. A part of dye sorption is then attributed to the ionic exchange.



Fig. 4. Effect of agitation time and initial concentration of BY28 on the sorption of basic yellow dye by reed. Conditions: pH 6, agitation speed 400 rpm, and at 25 °C. ◆ 10 mg/L; ■ 30 mg/L; ▲ 50 mg/L; × 70 mg/L; ● 100 mg/L.

3.3. Effect of adsorbent dose

The dose of adsorbent is varied from 0.1 to 0.5 g/250 mL keeping all the other experimental variables, viz. pH 6, initial concentration (100 mg L⁻¹), and agitation speed (400 rpm) constant. It was found that when the adsorbent dose is increased, the sorbed amount of BY28 decreases and this is due to the split in the flux or the concentration gradient between dye concentrations in the solution and on the sorbent surface.

3.4. Effect of pH

The pH of the solution has a significant impact on the uptake of dyes, since it determines the surface charge of the adsorbent, the degree of ionization, and the speciation of the adsorbate. In order to establish the effect of pH on the sorption of BY28, batch equilibrium studies at different pH values were carried out in the range of 2–10 (Fig. 5). We note that the uptake is quite low at lower pH. However, with the increase in pH, a significant enhancement in sorption is recorded. The optimum pH for reed- BY28 system was found to be 6.0 with a sorbed amount of about 140 mg/g. At lower pH, sorbed amount of BY28 decreased because the surface area of the adsorbent



Fig. 5. Effect of initial pH on the sorption of basic yellow dye by reed. Conditions: agitation speed 400 rpm, initial concentration of BY28 100 mg/L, agitation time 60 min, and temperature 25 $^{\circ}$ C.

Table 2 Kinetic parameters for sorption of basic yellow dye onto reed

was more protonated and a competitive sorption occurred between H^+ protons and positively charged BY28 dye at the surface sites. Therefore, H^+ ions react with the anionic functional groups on the surface of reed and results on a reduction of the number of binding sites available for the sorption of BY28.

3.5. Sorption kinetics

In most cases, the first-order equation did not apply well throughout the whole range of contact times and is generally applicable over the initial 20–30 min of the sorption process. The plotting of log $(q_e - q_t)$ vs. time (curves not shown) deviated considerably from the theoretical one. The plots and intercepts of curves were used to determine the first-order constant k_1 and equilibrium capacity q_e . The calculated values of $q_{e(cal)}$ (Table 2) from the first-order kinetic model are lower than the experimental. So, the sorption system does not follow a first-order reaction.

Using the linearized form of the second-order kinetic model (Eq. (6)), t/q_t was plotted vs. t at different adsorbate concentrations. The second-order sorption rate constant (k_2) and q_e values were determined from the slopes and intercepts of the plots. In all the cases, the correlation coefficients for the linear plots were superior to 0.99 (Table 2). The theoretical $q_{e(cal)}$ values agree perfectly with the experimental $q_{e(exp)}$ values. This suggests that the sorption system follows a pseudo-second-order model (Eq. (6)). The latter is based on the assumption that the rate limiting step may be a chemical sorption involving valence forces through the share or exchange of electrons between adsorbent and adsorbate.

3.6. Sorption isotherm (non-linear method)

The analysis and design of sorption separation process require the relevant equilibrium sorption, which is the most important piece of information in understanding the sorption process. In the present study, the sorption capacity and equilibrium isotherm for BY28 onto

$C_0/({ m mgL}^{-1})$	Pseudo-first-order			Pseudo-second-order				
	$\overline{k_1}$	$q_{e(\text{cal})}/\text{mg}\text{g}^{-1}$	R^2	$q_{e(\exp)}/\mathrm{mgg}^{-1}$	<i>k</i> ₂	$q_{e(\text{cal})} \text{ mg g}^{-1}$	R^2	$q_{e(\exp)}/\mathrm{mgg}^{-1}$
10	0.073	6.37	0.772	22.2	0.034	22.57	0.999	22.2
30	0.039	17.29	0.589	58.8	0.008	58.48	0.996	58.8
50	0.058	36.17	0.833	93.49	0.002	84.74	0.996	93.49
70	0.084	49.02	0.914	114.64	0.004	117.64	0.999	114.64
100	0.036	41.85	0.573	138.92	0.003	138.88	0.995	138.92

reed are estimated using Freundlich, Langmuir, Sips, and Generalized model isotherms equations.

The non-linear method is a mathematically method that uses the original form of the equation. The constants of the Lamgmuir and Freundlich models were determined by minimizing the error function using the *solver* add-in with Microsoft's spreadsheet, Microsoft Excel. The error function employed was as follows:

$$\sum_{i=1}^{P} (q_e - q_{cal})_i^2$$
 (6)

where *P* is the number of experimental data.

The q_{mon} , K_L , n, K_F , and R^2 values calculated from isotherms are presented in Table 3. The determination of the correlation coefficients (R^2) suggests that the Langmuir isotherm provides a good fit to the isotherm data for the sorption of the used dye.

The equilibrium data were further analyzed using the Freundlich model. The calculated Freundlich isotherm constants and the corresponding coefficients of correlation are shown in Table 3.

The magnitude of the exponent n gives an indication on the favorability of sorption. It is generally stated that values of n in the range 2–10 represent good, 1–2 moderately difficult, and less than 1 poor sorption characteristics [32]. The results show that the values of n are superior to 2 indicating that the sorption is good.

The abilities of Sips and Generalized model isotherms to model the equilibrium data were also examined by using the non-linear method. The Sips and Generalized model isotherm constants as well as the determination coefficient R^2 for the BY28 sorption systems are summarized in Table 3. The Generalized

Table 3

Lamgmuir, Freundlich, Sips, and Generalized model constants for sorption of basic yellow dye by reed using the non-linear method

Langmuir	$q_{\rm mon}$	180.98
0	K_L	0.074
	R^2	0.948
Freundlich	K_F	25.39
	1/n	0.448
	R^2	0.894
Sips	$q_{\rm mon}$	180.98
	K_L	0.074
	1/n	1
	R^2	0.948
Generalized model	$q_{\rm mon}$	180.98
	K_L	0.074
	1/n	1
	R^2	0.948

model isotherm exhibited the highest determination coefficient. It can be seen that the value of 1/n is equal to unity indicating that the Generalized model, Sips, and Langmuir isotherms are identical. Consequently, these three models were found to be the most suitable for this sorption system.

In order to check the validity of these models, it is interesting to recalculate the adsorbed amount using the calculated constant parameters determined using the non-linear forms. The simulated curves at 25 °C determined using Freundlich, Langmuir, Sips, and Generalized model isotherms are given in Fig. 6. The obtained curves using Langmuir, Sips, and Generalized model isotherms are practically superimposed. Thus, Langmuir, Sips, and Generalized model isotherms generate a best fit of equilibrium data than Freundlich isotherm (Fig. 6).

The experimental data of the present investigation show that natural reed exhibit a high capacity for basic yellow dye sorption from aqueous solutions (181 mg/g). This result reveals that natural reed is an effective adsorbent for basic yellow dye from wastewater.

A comparison is made between the sorption capacity of reed for the removal of basic yellow dye and other adsorbents reported in the literature [33–35]. Julide et al. [33] studied the adsorption of BY28 from aqueous solutions with clinoptilolite and amberlite. They reported that the clinoptilolite have a higher adsorption capacity than amberlite. The adsorption capacities of the clinoptilolite are 59.6, 52.9, and 56.7 mg/g at 20, 30 and 40 °C, respectively. Cheknane et al. [34] studied the adsorption of BY28 from aqueous solutions by Granular inorgano-organo pillared clays. They found that the maximum adsorption capacity value is 514 mg/g.



Fig. 6. Sorption isotherms for basic yellow dye onto reed. Conditions: pH 6, agitation speed 400 rpm, and temperature 25 °C. ◆ Experiment; ——— Langmuir; ------Freundlich; ------ Sips; -——- Generalized model.

Vesna et al. [35] were also investigated the sorption potentials of Poly(methacrylic acid) basedhydrogels with neutralization degree of monomer of 0% (PMAA/0) and 80% (PMAA/80) toward cationic dye, BY28. Adsorption capacities reported are 102 mg g/g for PMAA/0 and 157 mg g/g for PMAA/80, respectively.

3.7. Sorption mechanisms

The sorption mechanism involves chemical bonding and ion exchange. The sorption mechanisms can be explained by the presence of several interactions, such as complexation, ion exchange due to a surface ionization, and hydrogen bonds. One problem with lignocellulosic materials is that the sorption results are strongly pH-dependent [30]. There is a neutral pH beyond which the sawdust will be either positively or negatively charged. Ho and McKay [36] showed that the sorption capacity of basic dye is much higher than acid dye because of the ionic charges on the dyes and the ionic character of sawdust.

Reactive dyes attach to their substrates by a chemical reaction that forms a covalent bond between the molecule of dye and the fiber of sawdust. Thus, the dye stuff becomes a part of the fiber and is much less likely to be removed by washing than are dyestuffs that adhere by adsorption. The most important characteristic of reactive dyes is the formation of covalent bonds with the substrate to be colored. Thus, the dye forms a chemical bond with cellulose which is the main component of cotton fibers.

4. Conclusion

The present investigation concerns the removal of basic yellow dye from aqueous solutions by sorption onto reed as an adsorbent. The adsorption process is pH dependent due to the ion-exchange mechanism of the C=O stretching and bonded-OH groups with toxic BY28 dye. The maximum uptake of BY28 at 25 °C was about 140 mg/g of adsorbent at pH 6, initial dye concentration of 100 mg/L, agitation speed of 400 rpm, and adsorbent dose of 0.5 g/L. It is also established that almost 10 min are sufficient to attain equilibrium in the present case and the system follows pseudo-second-order rate.

The Freundlich, Langmuir, Sips, and Generalized model isotherm models were used to represent the equilibrium data. Results obtained indicate that the sorption equilibrium data fitted well by the Langmuir isotherm model. Maximum sorption capacity calculated from the Langmuir isotherm is equal to 181 mg/g at an initial pH of 6.0. These results show that reed is an effective adsorbent for the removal and recovery of BY28 from wastewater.

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