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Adsorption of a textile dye Ostazin Black NH from aqueous solution onto chitosan-coated perlite beads

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ABSTRACT

Chitosan-coated perlite beads were prepared by dropwise addition of gel containing chitosan and perlite into a precipitation bath. The structure of the beads was characterized using Fourier transform infrared spectroscopy. The surface area and microstructure of the beads were measured by Brunauer–Emmett–Teller (BET) instrument. The beads that contained chitosan enhanced the accessibility of OH and amine groups for adsorption process. Adsorption of Ostazin Black NH (OB) dye from aqueous solution on chitosan-coated perlite beads was studied using batch adsorption technique, and all parameters influencing the removal efficiency such as amount of adsorbent, pH, temperature and initial dye concentration were investigated. Adsorption of OB ions reached equilibrium concentration in 48 h. An increase in the initial dye concentration and a decrease in the temperature led to an increase in the adsorption amounts of chitosan-coated perlite beads. A decrease in the adsorption amount of OB dye was observed at high pH values. The optimum pH recorded for OB adsorption was 6–7. Experimental data were also evaluated in terms of kinetic characteristics of adsorption, and it was found that adsorption process followed well pseudo-second-order kinetics. Langmuir and Freundlich isotherms were studied for the adsorption data over a concentration range of 20–80 mg/L. Adsorption isotherms were well fitted with the Freundlich model, and the maximum adsorption capacity was 21.11 mg/g. Thermodynamic study was also performed to determine the feasibility of adsorption process. The results of thermodynamic parameters ($\Delta G°$, $\Delta H°$, and $\Delta S°$) showed that the adsorption of OB onto the chitosan-coated perlite beads was exothermic and spontaneous in nature. The chitosan-coated perlite beads are very promising for the removal of dye from wastewater due to their low cost of production and high removal efficiencies.

Keywords: Adsorption; Chitosan; Ostazin Black NH; Perlite

1. Introduction

Textile dyes used in textile dyeing processes are stable and have complex aromatic structure. Only 85% of the dyes get fixed to cloths, while the remaining 15% of dyes are discarded from dye baths as effluent [1]. Discharge of the colored effluent into streams and rivers results in a major threat to the aquatic environment as well as human health. Ostazin Black HN (OB), a reactive azo dye, is used for cotton, viscose, linen, silk, polyamide fiber and wool dyeing. The azo dyes poses very serious health risks to humans if they get into certain water supplies. Many azo dyes are toxic, mutagenic and/or carcinogenic to life [2–4]. A number of conventional methods such as biological [5], physical [6–8] and chemical treatments [9] are present for wastewater treatment. All of these methods have different color-removal abilities. The physicochemical and biological treatment methods are inefficient for the complete treatment of the organic compounds due to their resistance to photodegradation, oxidizing agents and

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biodegradation [10]. In the physical treatment of wastewater, adsorption is the most effective process for dye removal [6]. The adsorption process is also preferred over the other processes due to its low cost, easy operation, flexibility and simplicity of design [11,12]. In adsorption process, activated carbon has been proved as an efficient adsorbent for the dye removal from wastewater [13]. However, activated carbon has high cost and difficulty in regeneration [14]. Therefore, in recent years researchers have been studied on low-cost adsorbents such as modified sugarcane bagasse [15], *Hibiscus cannabinus* fiber [16], peanut husk [14,17], hen feather [18], bottom ash and de-oiled soya [19] for adsorption processes. While a number of investigations have been reported on low-cost adsorbents, more information is still needed to improve the new adsorbents for dye adsorption.

Perlite is an amorphous volcanic glass and can be expanded up to 20 times its original volume when heated rapidly at 800°C–1,200°C [20]. Perlite is very cheap and easily available in many countries. This fact can make it a viable candidate as an economical adsorbent for wastewater treatment. In the related literature, perlite has been used in the adsorption of dyes such as Methyl Violet [21], Malachite Green [22], Congo Red [23], C.I. Basic Blue 41 [24] and Maxilon Blue 5G [25].

Chitosan is a polycationic polymer derived from chitin and is readily available from seafood processing wastes [26]. Cross-linked chitosan particles could also be used as adsorbents due to the presence of amino and hydroxyl groups, which can serve as the active sites [27]. A composite material, prepared by cross-linking chitosan onto perlite surface, appeared to have much improved adsorptive features. The ability of chitosan-coated perlite beads to uptake metal ions such as copper(II) [28,29], nickel(II) [29], cobalt(II) [30] and chromium(VI) [31] has been studied in some works. However, there were no studies on the adsorption of dyes by chitosan-coated perlite beads.

In this work, we presented an economical and environmental adsorbent to uptake dye molecules. The chitosan-coated perlite beads were prepared as adsorbent and characterized by Brunauer–Emmett–Teller (BET) and Fourier transform infrared (FTIR) analyses. Moreover, dye adsorption performances were investigated by batch experiments, including the influence of contact time, initial pH, adsorbent dosage, temperature and initial dye concentration. More importantly, adsorption isotherms, kinetics and thermodynamic were also discussed to analyze the interaction between dye molecules and adsorbent.

2. Materials and methods

2.1. Materials

Expanded pelite used as a substrate for the preparation of beads was obtained from İzmir, Turkey. The expanded perlite particles were first washed with water to remove fine grains. The particles were then dried for 24 h at 110°C. Following this, the dried expanded perlite samples were mechanically sieved at 2–1.18 mesh. Chitosan (medium molecular weight) was procured from Aldrich Chemical Corporation, Germany. Oxalic acid dihydrate ($C_2H_2O_4.2H_2O$) was purchased from HiMedia, India. OB was obtained from a textile dyeing plant in Turkey, as a commercially available textile dye with a color index as Reactive Black 8, of 85% purity. Fig. 1 illustrates the molecular structure of the dye. A stock solution containing 1,000 ppm of OB was prepared using double distilled water. All the working solutions were obtained by diluting the stock solution with double distilled water. The pH of the experimental solutions was adjusted by adding 0.1 N NaOH (Merck, Germany) and 0.1 N HCI (Carlo Ebra, France) solutions.

2.2. Synthesis of chitosan-coated perlite beads

The chitosan-coated perlite beads were prepared by dropwise addition of the beads into an NaOH precipitation bath [28,32]. Expanded perlite was mixed with 0.2 M oxalic acid at room temperature overnight. Acid treated perlite was washed with deionized water and dried overnight at 70°C. 7.5 g of chitosan was dissolved in 250 mL of 0.2 M oxalic acid solution under continuous stirring at 40°C–50°C to prepare a viscous gel. 15 g of acid treated perlite added to the gel and stirred for 4 h at 40°C–50°C. The chitosan-coated perlite beads were prepared by dropwise addition of the mixture into a 0.7 M NaOH precipitation bath. The beads were seperated from NaOH bath and washed several times with deionized water to a neutral pH. The beads were dried in an oven.

2.3. Characterization of chitosan-coated perlite beads

Surface area of the chitosan-coated perlite beads was measured with a Micromeritics (ASAP 2020) BET instrument using nitrogen intrusion technique. The microstructure of the sorbent was characterized using physical adsorption/desorption of nitrogen at 77 K. FTIR spectra of pure chitosan and chitosan-coated perlite beads were obtained with an FTIR spectrometer (PerkinElmer Spectrum 100) in the range of 4,000–400 cm^{-1}.

2.4. Batch adsorption studies

All batch adsorption experiments were performed using a thermostat shaker (Termal H11960) with a shaking speed of 150 rpm. The concentration of OB in samples was determined using UV–visible spectrophotometer (Agilent Cary 60 UV–Vis) at a maximum absorption wavelength of 620 nm. All experiments were carried out in duplicate. The adsorption capacity q_e of dye was calculated using the following equation [33]:

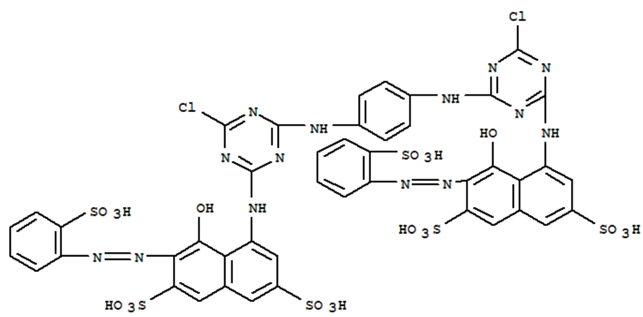

Fig. 1. Molecular structure of Ostazin Black HN.

$$q_e = \frac{V\left(C_0 - C_e\right)}{W} \quad (1)$$

where V is the volume of dye solution (L); C_0 stands for the initial concentration of the dye solution (mg/L); C_e is the equilibrium concentration of the dye solution and W represents the mass of adsorbent (g).

Removal percentage, R (%), was calculated using the following equation [33]:

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

3. Results and discussion

3.1. Characterization of chitosan-coated perlite beads

The FTIR spectrum of chitosan-coated perlite beads indicates the presence of predominant peaks at 3,550 cm^{-1} (–OH and –NH stretching) and 1,591.9 cm^{-1} (–NH bending in $-NH_2$) [26,30]. This reveals that all functional groups such as $-NH_2$ and –OH, originally present in pure chitosan, are unaffected even after coating on perlite and are available for interaction with the dye molecules [34]. In perlite spectrum, vaibration bands for Si–O–Si asymmetric stretching at 1,000–1,100 cm^{-1} and Si–O–Si symmetric at about 800 cm^{-1} are clearly observed due to silica content [35]. The peaks were observed at 1,019.4 cm^{-1} and 782.4 cm^{-1} in chitosan-coated perlite beads.

Surface area and pore volume of the composite adsorbent were determined with BET instrument. The chitosan-coated perlite beads show an average surface area of 1.9923 m^2/g and pore volume of 0.014 cm^3/g.

3.2. Contact time and adsorption kinetics

The initial OB concentrations of 20, 40, 60 and 80 mg/L were used to determine the equilibrium time for the adsorption of OB onto chitosan-coated perlite beads. Fig. 2 illustrates the effects of contact time on the adsorptive removal of OB dye from aqueous solution by chitosan-coated perlite beads. For each adsorbent–adsorbate system, the adsorption process is rapid in the first 24 h. Subsequently, the adsorption process is slow due to decrease in diffusion rate and concentration gradient [36]. After the contact time of 48 h, there is no change in the amount of adsorbed dye by chitosan-coated perlite beads. This means that the contact time needed to reach equilibrium is 48 h for the adsorption of OB (as seen in Fig. 2).

In order to understand the OB adsorption process on chitosan-coated perlite beads, the kinetic data were analyzed using the pseudo-first-order and pseudo-second-order kinetic models (Fig. 3). The pseudo-first-order and pseudo-second-order kinetic model can be expressed according to Eqs. (3) and (4), respectively [37]:

$$\log(q_e - q_t) = \log q_e - \frac{k_1}{2.303} t \quad (3)$$

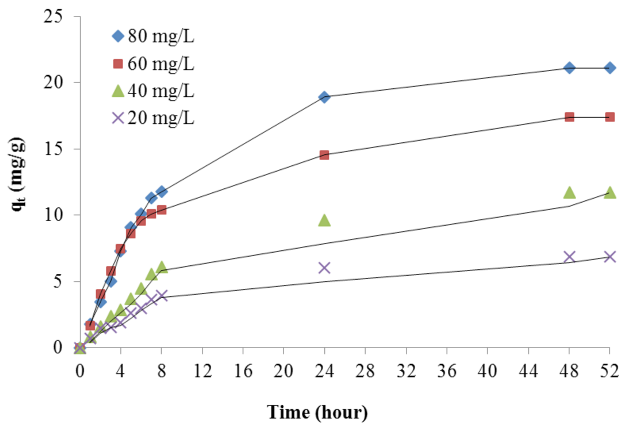

Fig. 2. The effect of contact time on the adsorption of OB onto chitosan-coated perlite beads at different initial concentrations (adsorption conditions: adsorbent dosage 2 g/L; 25°C; 150 rpm; pH 7).

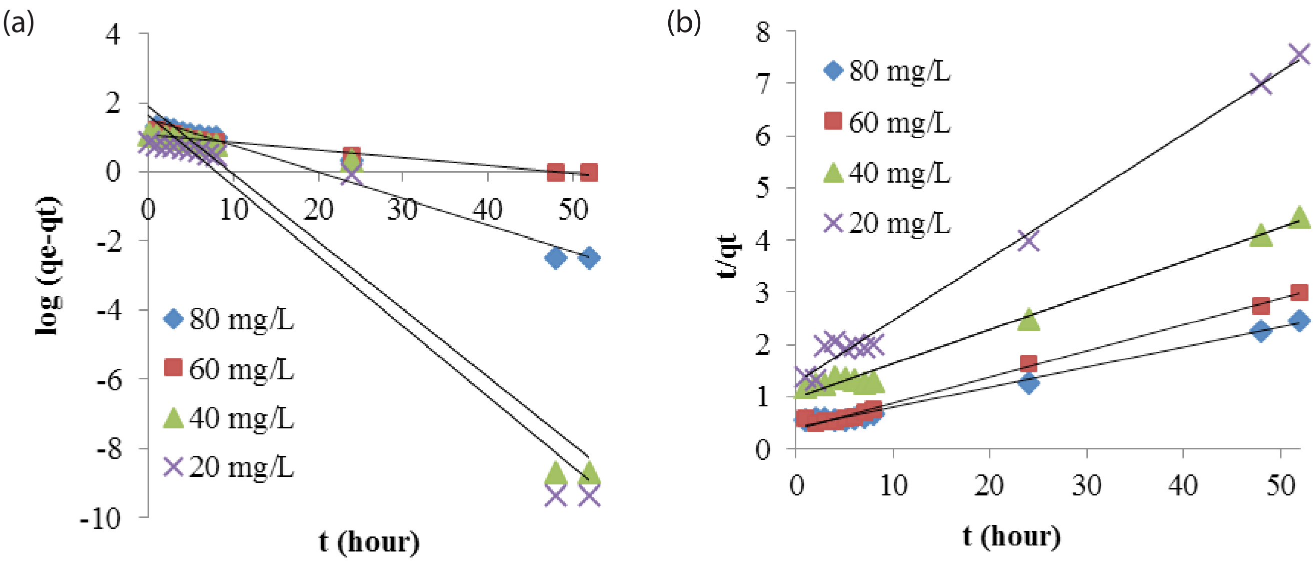

Fig. 3. Plot of the pseudo-first-order (a) and pseudo-second-order (b) equation at different initial concentrations (adsorption conditions: adsorbent dosage 2 g/L; 25°C; 150 rpm; pH 7).

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \qquad (4)$$

where q_e (mg/g) and q_t (mg/g) are the amounts of OB adsorbed at equilibrium and at different time intervals, respectively. k_1 (1/min) and k_2 (g/mg min) are the rate constants of pseudo-first-order and pseudo-second-order models, respectively.

The characteristics parameters of pseudo-first-order and pseudo-second-order models are given in Table 1. The calculated value of q_m from the pseudo-first-order kinetics model is dramatically higher than the experimental value. However, the pseudo-second-order kinetics model provided a near match between the theoretical and experimental q_m values. As a result, the adsorption process appeared to follow pseudo-second-order reaction kinetics. These results showed that the adsorption of OB onto the chitosan-coated perlite was best described by pseudo-second-order kinetic model with a high correlation coefficient.

3.3. Effect of pH on OB adsorption

The pH value of dye solution is one of the most important parameters affecting the adsorption capacity of adsorbent. The effect of pH on adsorption capacity of OB dye onto chitosan-coated perlite beads for 48 h is shown in Fig. 4. It indicates that the OB adsorption is low in the high basic medium. As the pH increases from 6 to 11, the adsorption capacity of the chitosan-coated perlite beads decreases, and maximum dye adsorption capacity is achieved in the pH range of 6–7. At a pH value less than 5, the structure of chitosan-coated perlite beads was distrupted due to the dissolution of chitosan [30].

3.4. Effect of adsorbent dosage

The effect of adsorbent dosage on removal of OB was conducted in batch experiments by adding various amount of adsorbent in the range of 0.1–0.5 g into the flask containing 50 mL of OB solution. For all experiments, the initial dye concentration and temperature were fixed at 80 mg/L and 25°C, respectively. The results are illustrated in Fig. 5. The amount of OB adsorbed onto the chitosan-coated perlite beads was found to decrease from 4.67 to 3.45 mg/g with an increase in adsorbent dose due to the concentration gradient between adsorbent and adsorptive [38]. The percentage removal of OB increased from 11.7% to 43.17% as the adsorbent dosage was increased from 2 to 10 g/L. It can be explained that a greater number of adsorption sites and a higher surface area for dye molecules were provided at 10 g/L chitosan-coated perlite dosages [39].

3.5. Effect of temperature and initial OB dye concentration

The adsorption capacity of OB dye on the chitosan-coated perlite beads was performed in 20–80 mg/L concentration range at temperatures 25°C, 35°C, 45°C and 55°C. As shown in Fig. 6, the adsorption capacity increases with an increase in the initial concentration of OB dye from 20 to 80 mg/L due to the driving force of increasing concentration gradient by causing an increase in mass transfer of OB molecules onto the surface of chitosan-coated perlite beads [40]. The obtained data showed that the adsorption capacity increased with a decrease in the temperature from 55°C to 25°C indicating the exothermic nature of dye absorption.

3.6. Adsorption isotherms

Adsorption isotherms can provide useful information on the adsorption mechanism. In this study, Langmuir and the Freundlich models were utilized to explain the experimental data.

The linear forms of the Langmuir and Freundlich isotherms are given by Eqs. (5) and (6), respectively [41]:

$$C_e/q_e = C_e/q_m + 1/(q_m.K_L) \qquad (5)$$

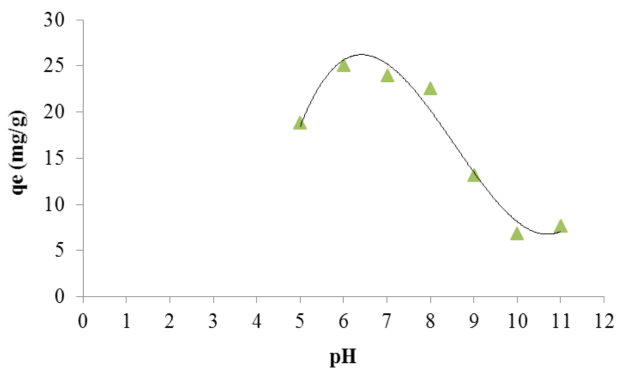

Fig. 4. The effect of pH on the adsorption of OB onto chitosan-coated perlite beads (adsorption conditions: initial concentration 100 mg/L; adsorbent dosage 2 g/L; 25°C; 150 rpm).

Table 1
Parameters of the pseudo-first-order and pseudo-second-order models for adsorption of OB onto the chitosan-coated perlite beads at different initial concentrations

C_0 (mg/L)	$q_{e,\exp}$ (mg/g)	Pseudo-first-order kinetic model			Pseudo-second-order kinetic model		
		$q_{e,\text{cal}}$ (mg/g)	k_1 (h^{-1})	R^2	$q_{e,\text{cal}}$ (mg/g)	k_2 (x10^3) (g mg^{-1} h^{-1})	R^2
20	6.86	44.49	0.4689	0.9188	8.40	11.05	0.9926
40	11.71	75.02	0.4491	0.9141	15.48	4.17	0.9903
60	17.41	12.27	0.0516	0.9699	20.04	6.48	0.9950
80	21.11	32.18	0.1762	0.9713	25.97	3.57	0.9916

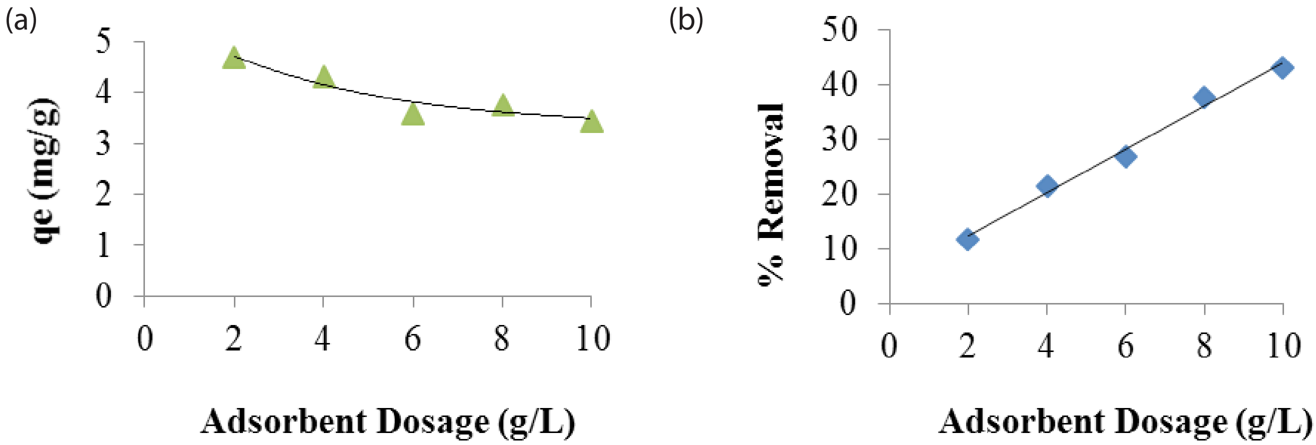

Fig. 5. The effect of adsorbent dosage on the adsorption capacity (a) and dye removal efficiency (b) of chitosan-coated perlite beads.

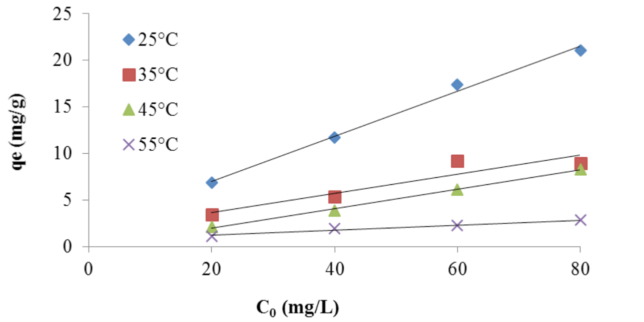

Fig. 6. The effect of initial OB concentration on the adsorption by chitosan-coated perlite beads at different temperatures (adsorption conditions: adsorbent dosage 2 g/L; 150 rpm; pH 7; time 48 h).

$$\ln q_e = \ln K_F + \left(\frac{1}{n}\right).\left(\ln C_e\right) \quad (6)$$

where C_e is the equilibrium dye concentration in the solution (mg/L); q_e is the equilibrium dye uptake on the adsorbent (mg/g); q_m is the maximum adsorption capacity (mg/g) and K_L is the Langmuir constant (L/mg) that is related to the affinity of binding sites and related to the energy of sorption. K_F [mg/g (mL/g)$^{1/n}$] and n are the Freundlich constants of the system related to adsorption capacity and intensity, respectively.

The linear plots for the both isotherm models are given in Fig. 7. It can be observed from the plots and correlation coefficients (R^2) that the experiment data are in excellent agreement with Freundlich model. This implied that these adsorption processes took place on heterogeneous surfaces of the chitosan-coated perlite beads with interaction between adsorbed OB molecules [37]. The Freundlich constants, K_F and n, were calculated as 2.4483 mg/g (L/g)$^{1/n}$ and 1.6431, respectively. The value of n obtained by Freundlich model gave a value greater than unity indicating the adsorption to be favorable process [42].

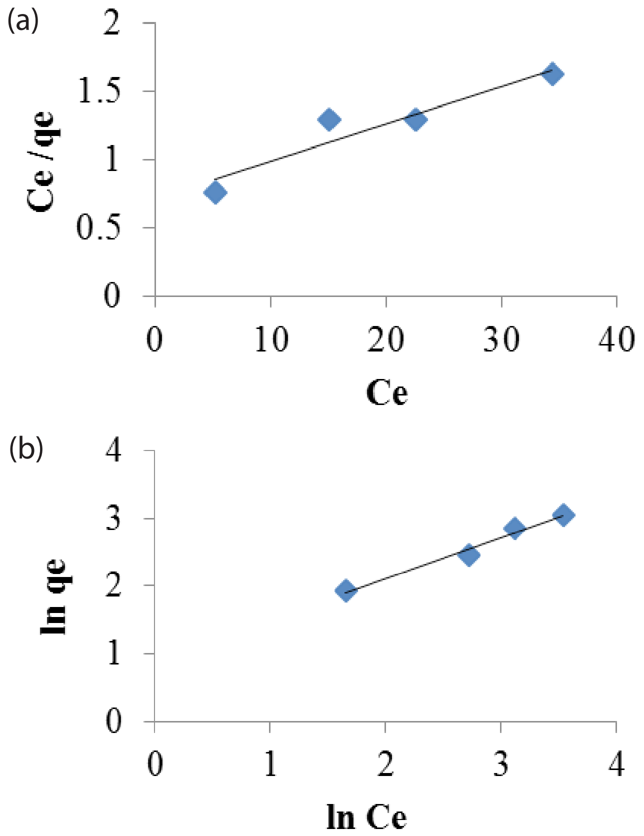

Fig. 7. Adsorption isotherms of Langmuir (a) and Freundlich (b) for OB adsorbed over chitosan-coated perlite beads (adsorption conditions: initial OB concentration 20–80 mg/L; adsorbent dosage 2 g/L; 150 rpm; pH 7; time 48 h; 25°C).

3.7. Adsorption thermodynamics

Thermodynamic study for the adsorption of OB onto chitosan-coated perlite beads was conducted in the temperature range of 25°C–55°C keeping OB concentration of 80 mg/L, adsorbent dose of 2 g/L and contact time of 48 h.

To evaluate the influence of temperature on adsorption process of OB onto chitosan-coated perlite beads, the thermodynamic parameters such as Gibbs free energy ($\Delta G°$),

Table 2
Thermodynamic parameters of OB adsorption by chitosan-coated perlite beads

T (K)	$\Delta G°$ (kJ mol^{-1})	$\Delta H°$ (kJ mol^{-1})	$\Delta S°$ (J mol^{-1} K^{-1})
298	–1.243	–14.330	–43.914
308	–0.804		
318	–0.365		
328	–0.073		

enthalpy ($\Delta H°$), and entropy ($\Delta S°$) can be calculated using the following equations [15]:

$$\Delta G° = -RT \ln K_C \quad (7)$$

$$\Delta G° = \Delta H° - T\Delta S° \quad (8)$$

$$\ln K_C = \left(\Delta S° / R\right) - \left(\Delta H° / RT\right) \quad (9)$$

$$K_C = q_e / C_e \quad (10)$$

where R is the universal gas constant (8.314 J/mol K); T is the absolute temperature (K) and K_C is the equilibrium constant. Plotting $\ln(q_e/C_e)$ against $1/T$ gives a straight line. $\Delta S°$ and $\Delta H°$ can be calculated from the intercept and the slope, respectively.

The calculated thermodynamic parameters are given in Table 2. The negative $\Delta G°$ values indicate that the adsorption processes are spontaneous thermodynamically. Generally, the values of $\Delta G°$ in between 0 and –20 kJ/mol indicate the adsorption process is physisorption, whereas the values in between –80 and –400 kJ/mol correspond to chemisorption [43]. As shown in Table 2, the values of $\Delta G°$ suggest that the OB adsorption onto chitosan-coated perlite beads is a physisorption. The negative value of $\Delta H°$ indicates the reaction to be exothermic in nature. In addition, the negative $\Delta S°$ value illustrates the decrease in randomness at the solid/liquid interface during the adsorption processes [15].

4. Conclusion

A type of biopolymer, chitosan is a good adsorbent to remove various kinds of anionic and cationic dyes. In this study, chitosan-coated perlite beads were developed and used for the removal of OB dye from aqueous solution. A significant effect of pH was observed. The maximum removal of OB by chitosan-coated perlite was observed at pH 6–7. Adsorption of OB onto chitosan-coated perlite beads was spontaneous, exothermic and accompanied with a decrease in entropy. Adsorption kinetic was modeled successfully by the pseudo-second-order rate equation. The equilibrium adsorption data were fitted to Freundlich adsorption isotherm models. This study convinced that the chitosan-coated perlite beads proved to be an alternative, economic and environmentally friendly adsorbent for dye removal from aqueous solution.

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