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ORIGINAL PAPER

Evaluation of adsorption potential of bamboo biochar for metal-complex dye: equilibrium, kinetics and artificial neural network modeling

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Abstract Metal-complex dyes are widely used in textile industry, but harmful to the environment and human health due to aromatic structure and heavy metal ions. The objective of this work was to evaluate the adsorption potential of bamboo biochar for the removal of metal-complex dye acid black 172 from solutions. Freundlich model was more suitable for the adsorption process of bamboo biochar than Langmuir isotherm, indicating multilayer adsorption of acid black 172 on a heterogeneous bamboo biochar surface. Adsorption kinetics analysis of pseudo-second-order and Weber–Morris models revealed that intraparticle transport was not the only rate-limiting step. The bamboo biochar exhibited a good adsorption performance even at high ionic strength. Analysis based on the artificial neural network indicated that the temperature with a relative importance of 29 % appeared to be the most influential parameter in the adsorption process for dye removal, followed by time, ionic strength, pH and dye concentration.

Keywords Bamboo biochar · Metal-complex dye · Artificial neural network · Adsorption equilibrium · Ionic strength

Introduction

Large amounts of dye effluents are annually discharged by textile, cosmetics, paper, leather, pharmaceutical, food and other industries (Safarikova et al. 2005). The dye-containing wastewater can adversely affect the aquatic environment by impeding light penetration. Moreover, most of the dyes are toxic, carcinogenic and harmful to human health (Yang et al. 2012; Debrassi et al. 2012). As a result, many governments have established environmental restrictions with regard to the quality of colored effluents and have required dye industries to decolorize their effluents before discharging (Shen et al. 2009). Hence, various treatment processes such as physical separation, oxidation, biological degradation, coagulation and flocculation have been developed to remove dyes from wastewaters (Forgacs et al. 2004; Yang et al. 2012). Adsorption process is one of the most efficient and attractive methods for removing pollutants from wastewater, which has interesting characteristics, such as easy process control, low cost and energy requirements (Chen et al. 2010; Mittal et al. 2012). Biochar is produced by the combustion of biomass under limited oxygen conditions. Biochar is a fine-grained and porous substance and has been applied recently to remove dyes from aqueous solutions (Mahmoud et al. 2012; Qiu et al. 2009; Xu et al. 2011). The effects of manufacturing the parameters of biochar on dye removal were discussed in detail in the literatures, such as heating rate, carbonization temperature and carbonization time and so on (Demirbas 2009; Mui et al. 2010a, b; Wang and Yan 2011).

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Acid black 172 (molecular formula: $C_{40}H_{20}O_{14}N_6S_2Na_2Cr$; molecular weight: 970; molecular structure was shown in Fig. 1) containing Cr (VI) was selected as a model for the anionic metal-complex dyes, which could cause more serious problems as the dyes are discharged into the environment. In this study, the objective was to investigate the adsorption behavior of bamboo biochar for the removal of the anionic metal-complex dyes and evaluate the adsorption potential of bamboo biochar at high ionic strength. Langmuir and Freundlich models were used to analyze the adsorption isotherms. The experimental data were fitted using the pseudo-second-order kinetic and Weber–Morris models. The limiting factor of adsorption process was also analyzed. Furthermore, artificial neural network (ANN) was used to investigate the effects of the operational parameters on the adsorption capacity. The experiments were carried out in Wuhan Botanical Garden during May to July of 2012.

Materials and methods

Materials and reagents

The biochar derived from bamboo used in this study was supplied by Zhuzhikang products factory (Quzhou, China) where it was produced by pyrolysis at 1000 °C. The biochar was ground to powder in a disintegrator and sieved through a No. 100 standard sieve to obtain uniform size for adsorption studies. The characteristics of biochar were as follows: 517.28 m^2/g specific area; the point of zero charge

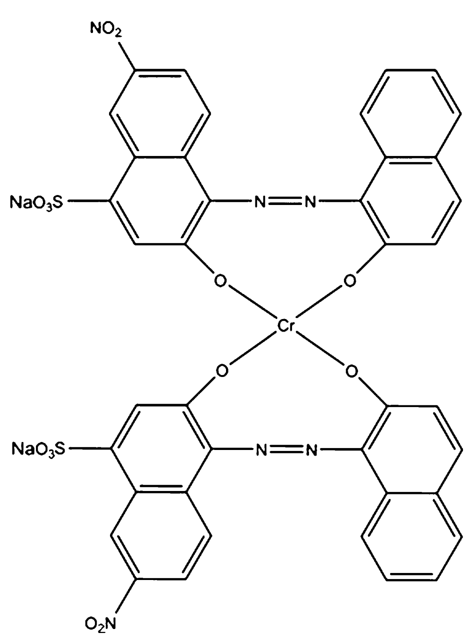

Fig. 1 The molecular structure of acid black 172 used in this study

(pHzpc), 8.45; and 0.407 cm^3/g pore volume. The dye acid black 172 was obtained from Shanghai Sangon Biological Engineering Technology & Services Co., Ltd, Shanghai, China.

Preparation of dye solution and determination of dye concentrations

Stock solutions (1,000 mg L^{-1}) of dye were prepared in deionized and double distilled water and diluted to get the desired concentration of dye. A calibration curve for acid black 172 was prepared by measuring the absorbance of different concentrations. The acid black 172 was measured at 597 nm to determine the concentration in the solution at pH 1.0–8.0.

Adsorption experiments

Adsorption experiments were carried out with 100 mL dye solution of desired concentration mixing 0.1 g bamboo biochar in 250-mL Erlenmeyer flasks. The mixture was agitated (200 rpm) at 30 °C for 8 h unless otherwise stated. The influence of hydrogen ion concentration on the adsorption process was studied over a pH range of 1.0–8.0, with adjustments being made using 0.1 mol L^{-1} HCl or NaOH. The effect of dye concentration was studied in the range from 50 to 500 mg L^{-1} at pH 1.0. The effect of temperature on the adsorption capacity was investigated in the temperature range from 20 to 40 °C at pH 1.0. Adsorption kinetics was investigated with an initial dye concentration of 100, 200 and 300 mg L^{-1} from 15 to 480 min at pH 1.0. The influence of ionic strength on adsorption process of bamboo bichar for acid black 172 was tested with $NaNO_3$ added to the dye solution.

The adsorption capacity, Q_e (mg g^{-1}), was calculated as follows:

$$Q_e = \frac{(C_O - C_e)V}{M} \tag{1}$$

where C_o and C_e are the initial and final concentrations (mg L^{-1}), respectively, M is the adsorbent dosage (g), and V is the volume of solution (L).

Mathematical modeling study

Adsorption isotherm models

The adsorption isotherm models described the relationship between the adsorbate loading on the adsorbent (Q_e) and liquid phase concentration of adsorbate (C_e) at equilibrium conditions (Kumar et al. 2011). In this research, Langmuir (Langmuir 1918) and Freundlich (Freundlich 1906) models were selected to fit the equilibrium data. The Langmuir model corresponds to the homogeneous monolayer adsorption, whereas the Freundlich model defines the adsorption onto the adsorbent with heterogeneous surface as follows:

$$Qe = \frac{Q_{\max} K_L C_e}{1 + K_L C_e}; \quad \text{Langmuir model} \tag{2}$$

$$Qe = K_F C_e^{1/n}; \quad \text{Freundlich model} \tag{3}$$

where $Q_{\max}$ is the maximum adsorption capacity calculated by Langmuir model, K_L is the Langmuir constant (L mg^{-1}), and K_F (L g^{-1}) and n (dimensionless) are Freundlich constants that indicate the extent of the adsorption, and the degree of nonlinearity between solution concentration and adsorption, respectively.

Adsorption kinetic models

Adsorption kinetic models give information regarding the mechanisms of adsorption that are important for the efficiency of the process. In general, the pseudo-second-order equation has been more successfully applied to the adsorption of metal ions, dyes, herbicides and other organic substances from aqueous solutions compared with the pseudo-first-order model (Park et al. 2010). In this study, the pseudo-second-order (Ho and McKay 1998) and Weber–Morris (Özer et al. 2006) models were applied to describe the adsorption kinetics as follows:

$$\frac{t}{Q_t} = \frac{1}{K_2 Q_{\max}^2} + \frac{1}{Q_{\max}} t \quad \text{Pseudo-second-order model} \tag{4}$$

$$Q_t = K_w t^{1/2} + I \quad \text{Weber-Morris model} \tag{5}$$

where Q_{max} and Q_t are the adsorption capacities (mg g^{-1}) at the equilibrium and at time t, respectively, K_2 and K_w are the constant of pseudo-second-order and Weber–Morris models, respectively, and I is the value of intercept, which gives an idea about the thickness of the boundary layer, that is, the larger is the intercept, the greater is the boundary layer effect (Özer et al. 2006). For Weber–Morris model, if the intraparticle diffusion is involved in the sorption process, then the plot will be linear, and if it is the rate controlling step, then the plot will pass through origin. If there are two stages for Weber–Morris model, the initial curve portion represents the boundary layer diffusion, while the later linear portion defines the intraparticle diffusion (Vijayaraghavan et al. 2007).

Artificial neural network model

Artificial neural network (ANN) has been widely used to model the effect of parameters influencing adsorption or degradation of dyes (Khataee et al. 2010; Yang et al. 2011; Zarei et al. 2010). A three-layer ANN was used in this study. The input layer had five neurons as pH, initial dye concentration, temperature, ionic strength and contact time. The output layer had one neuron as the amount of adsorbed acid black 172 on the adsorbent. The data sets were randomized and divided into training, validation and test subsets, which included 108, 36 and 36 samples, respectively. All samples were normalized in the range 0.1–0.9 using the following equation:

$$A_i = 0.8\left(\frac{X_i - \min(X_i)}{\max(X_i) - \min(X_i)}\right) + 0.1 \tag{6}$$

where $\min(X_i)$ and $\max(X_i)$ are the extreme values of variable X_i (Khataee et al. 2010). Mean squared errors (MSE) were used to determine the structure of three-layer feed-forward back propagation neural network, which was calculated by the following equation:

$$\text{MSE} = \frac{1}{N} \sum_{i=1}^{i=N} (y_{i,\text{pred}} - y_{i,\exp})^2 \tag{7}$$

where $y_{i,\text{pred}}$ and $y_{i,\exp}$ are the values predicated by the neural network and obtained by experiments, respectively, N is the number of data point, and i is an index of data. Levenberg–Marquardt (LM) algorithm, which was an algorithm for least-squares estimation of nonlinear parameters and had been proven to be the fastest and the most robust, was used as a training method in this study (Marquardt 1963). The sensitivity analysis was analyzed by Garson method (Garson 1991) to calculate the relative importance of different input variables on sorption capacity.

Results and discussion

Effect of pH on adsorption capacity

The pH has an important effect on dye adsorption since the pH of the aqueous solution will control the magnitude of the electrostatic charges that are imparted by ionized dye molecules and adsorbate (Mahmoud et al. 2012). Figure 2 shows the effect of pH on adsorption capacity of acid black 172 onto bamboo biochar. The maximum adsorption capacity (81.88 mg g^{-1}) for acid black 172 was obtained at pH 1.0 and then decreased as the pH increased from 1.0 to 9.0. Acidic conditions could be favorable for the adsorption between acid black 172 and biochar, because a significantly high electrostatic attraction could exist between the positively charged surface of the adsorbent under acidic conditions and the anionic dye (acid black is anionic dye in solution). At high pH, the biochar become less positively charged (pHzpc of biochar 8.45) and excess OH^- ions competed with the anionic groups of dye for adsorption

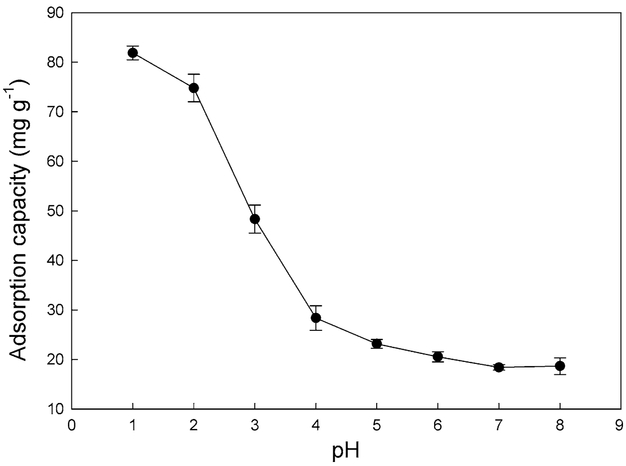

Fig. 2 Effects of pH on the adsorption capacity of bamboo biochar for acid black 172 (100 mg L^{-1} of dye concentration, 200 rpm, 0.1 g bamboo biochar, 30 °C)

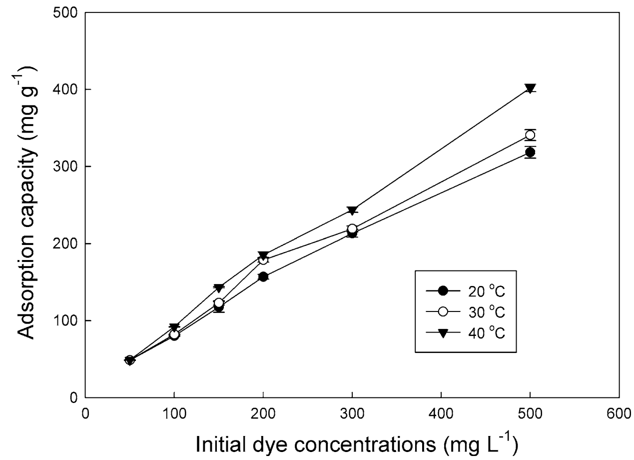

Fig. 3 Effects of dye concentration and temperature on the adsorption capacity of bamboo biochar for acid black 172 (pH 1.0, 200 rpm, 0.1 g bamboo biochar, 30 °C)

sites. Hence, the adsorption capacity of biochar at high pH was significantly lower than that at low pH. Similar results were reported in the literature for the adsorption of acid black 172 onto nonviable *Penicillium* YW 01 (Yang et al. 2011).

Effect of initial dye concentration and temperature on adsorption capacity

The initial dye concentration provided the driving force to overcome mass transfer resistances of the dyes between the aqueous and solid phases (Aksu and KarabayIr 2008). Figure 3 showed a linear increase in the adsorption capacity for acid black 172 was observed as the initial dye concentration increased up to 500 mg L^{-1}. The adsorption capacity increased with temperatures from 20 to 40 °C for the tested dye concentrations (Fig. 3). The effects of temperature could be attributed to the decrease in the viscosity of the solution with the temperature increasing. The maximum adsorption capacity (401.88 mg g^{-1}) was obtained at 40 °C with initial dye concentration of 500 mg L^{-1}. Most of the chars derived from many sources have been found to exhibit good performance for basic dyes (Table 1). However, the bamboo biochar in this study exhibited a high adsorption capacity for acid dye and was found to be comparable to those of many corresponding chars from rice straw, sewage sludge and crop residues (Hameed and El-Khaiary 2008; Jindarom et al. 2007; Xu et al. 2011).

Adsorption isotherms

Langmuir and Freundlich models have been most commonly used to describe the adsorption isotherm (Park et al. 2010), which were evaluated for their ability to fit the equilibrium data in this research. The parameters for bamboo biochar obtained for the different isotherm models are listed in Table 2. The R^2 values of Langmuir model were less than 0.90 under tested conditions, while the values for Freundlich model were more than 0.90. This showed that Freundlich model was more suitable for the adsorption process of bamboo biochar in this study, indicating multilayer adsorption of acid black 172 occurred on a heterogeneous bamboo biochar surface with a nonuniform distribution of heat of adsorption. The values of Freundlich constant n for bamboo biochar (Table 2) were all in range 2–10, indicating that good adsorption occurred (Subramanyam and Das 2009). The high values of K_F (Table 2) implied uptake of the dyes from solution with high adsorptive capacities of bamboo biochar.

Effect of contact time and adsorption kinetics

Figure 4a shows that the rates of adsorption of acid black 172 on bamboo biochar were initially fast and then gradually decreased until equilibrium reached. According to the determination coefficients (Table 3), the data best fit a pseudo-second-order model which gave $R^2 > 0.98$ for all the tested dye concentrations. The results indicated that the boundary layer resistance was not a rate-limiting step since dye adsorption followed pseudo-second-order kinetics (Xiong et al. 2010). Linear regression plots by fitting experimental data to Webber–Morris model showed that the intraparticle diffusion of acid black 172 within bamboo biochar under different dye concentrations occurred in two stages involving macropore diffusion followed by micropore diffusion (Fig. 4b). Since the $k_{w,2}$ values of biochar were smaller than the $k_{w,1}$ values (Table 3), the intraparticle diffusion was predicted to be the rate-limiting step for the adsorption of acid black 172 onto the biochar. The

Table 1 Adsorption capacities of dyes onto various chars reported

Chars	Dyes	pH	Adsorption capacity (mg g^{-1})	References
Acid-treated kenaf fiber char	Methylene blue	8.5	22.73	(Mahmoud et al. 2012)
Biochar from wheat straw	Methylene blue	9.0	16.92	(Liu et al. 2012)
Tyre char	Acid yellow 117	–	93.28	(Mui et al. 2010a)
Rice straw-derived char	Malachite green	5	148.74	(Hameed and El-Khaiary 2008)
Biochars from crop residues	Methyl violet	9.0	104.62	(Xu et al. 2011)
Char from sewage sludge	Acid yellow 49	–	116	(Jindarom et al. 2007)
	Basic blue 41	–	588	
Biochar from bamboo	Acid black 172	1.0	401.88	Present work

Table 2 Adsorption isotherm parameters for the adsorption of acid black 172 onto the biochar derived from bamboo at various temperatures

Temperature (°C)	Langmuir constants			Freundlich constants		
	Q_{max} (mg g^{-1})	K_l (L mg^{-1})	R^2	n	K_F (L g^{-1})	R^2
20	384.62	0.017	0.858	2.69	37.61	0.901
30	416.67	0.018	0.803	2.52	36.09	0.917
40	434.78	0.049	0.885	2.26	48.54	0.928

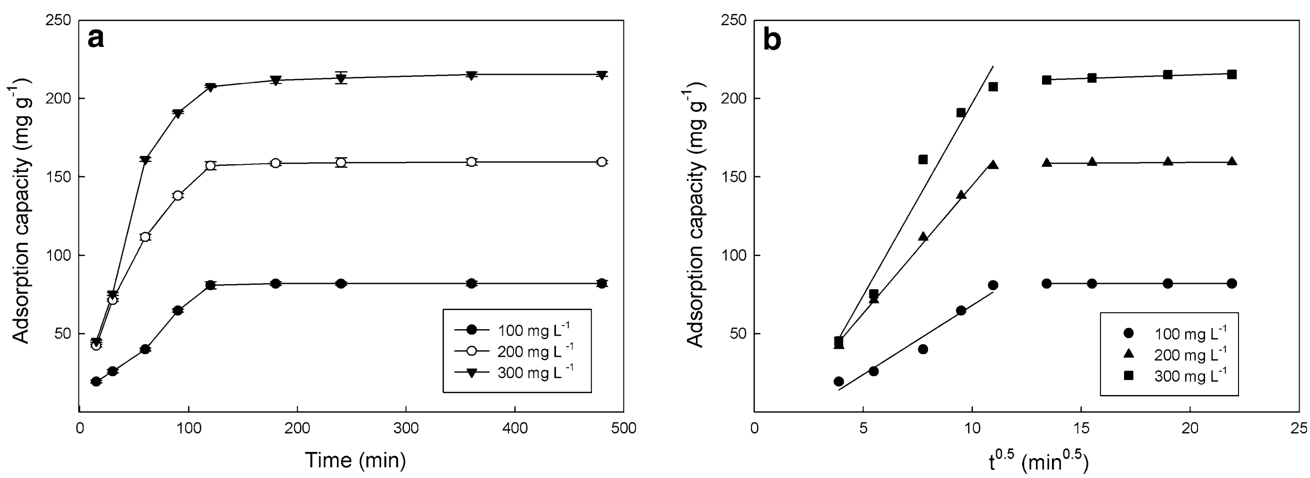

Fig. 4 Effects of contact time (**a**) and Weber–Morris plots (**b**) for the adsorption process (pH 1.0, 100 mg L^{-1} dye concentration, 30 °C, 200 rpm)

deviation of the regression lines from the origin (I_l values in Table 3) further indicated that intraparticle transport was not the only rate-limiting step. The transport of the dye through the particle-sample interphase onto the pores of the bamboo biochar and adsorption on the available surface of the biochar could also be responsible for the adsorption. This agrees with the findings of the herbicide adsorption onto the derived activated carbon (Itodo et al. 2010).

Effect of ionic strength on adsorption

The high ionic strength in wastewater results from metal ions (Na^+, K^+, Cu^{2+}, Ca^{2+} Cr^{3+}, etc.) and its counter ions (NO_3^-, PO_4^{3+} SO_4^{2-}, etc.), which may significantly affect the performance of the adsorption process (Maurya et al. 2006). Therefore, $NaNO_3$ was used to evaluate the adsorption potential of bamboo biochar for dyes from aqueous solution. It could be observed that the adsorption capacity increased 6.4 % at 0.1 mol L^{-1} compared with the control, and then the adsorption capacity decreased as ionic strength increased (Fig. 5). Most of the reported literatures about the effect of ionic strength were evaluated at the Na^+ concentration ranging from 0.1 to 1.0 mol L^{-1} (Chiou and Li 2003; Al-Degs et al. 2008; Maurya et al. 2006). But, the adsorption capacity of biochar for acid black 172 was found to have little decrease even at a very

Table 3 Parameters of pseudo-second-order and Weber–Morris model for the adsorption of acid black 172 onto the bamboo biochar

$Q_{eq,\exp}$ (mg g^{-1})	Pseudo-second-order kinetic model			Initial linear portion (Weber–Morris)			Second linear portion (Weber–Morris)		
	k_2x10^{-2}(g mg^{-1} min^{-1})	$Q_{e,cal}$ (mg g^{-1})	R^2	$K_{w,1}$	I_1	R^2	$K_{w,2}$	I_2	R^2
82.09	0.020	94.33	0.981	8.83	−19.981	0.955	0.017	81.738	0.859
159.45	0.019	172.41	0.994	16.33	−18.424	0.996	0.092	157.54	0.851
215.50	0.011	238.09	0.987	24.53	−48.037	0.966	1.325	210.66	0.917

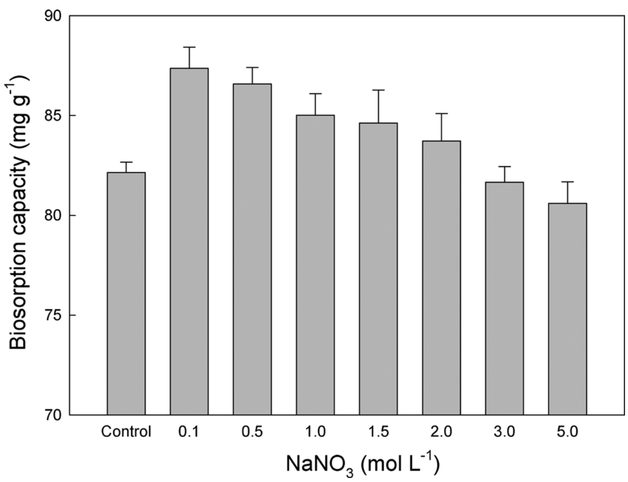

Fig. 5 Effects of ionic strength on adsorption capacity of bamboo biochar for acid black 172 (control: without $NaNO_3$ added, pH 1.0, 100 mg L^{-1} dye concentration, 30 °C, 200 rpm)

high $NaNO_3$ concentration (3.0 and 5.0 mol L^{-1} in Fig. 5). Theoretically, an increase in ionic strength will decrease the adsorption capacity in this system if the electrostatic forces between the adsorbent surface and adsorbate ions were attractive, and vice versa. The effect of ionic strength on dye adsorption was studied at pH 1.0, where dyes and activated carbon were oppositely charged. The experimental data from this study did not follow this convention, as the adsorption capacities of negatively charged dye molecules on positively charged bamboo biochar were more than the control at a concentration range of 0.1–2.0 mol L^{-1}. Similar phenomenon was also found in the adsorption behavior of reactive dyes on activated carbon (Al-Degs et al. 2008).

ANN modeling

A back propagation ANN was used for modeling the adsorption behavior of bamboo biochar for metal-complex dye acid black 172. Input variables of neural network were as follows: pH (1–8), initial dye concentration (50–500 mg L^{-1}), temperature (30–40 °C), ionic strength (0.1–5.0 mol L^{-1}) and contact time (15–480 min). The adsorption capacity (19.51–401.88 mg g^{-1}) of bamboo biochar was chosen as the output variable.

The ANN having 5 nodes on the hidden layer with MSE value 1.048×10^{-4} was selected for modeling the adsorption process of biochar for acid black 172. A regression analysis between experimental data and predicted values using ANN model had a high determination coefficient of 0.996 (Fig. 6a), indicating that ANN model reproduced the adsorption well in this system. Similar results also observed in the previous studies (Çelekli et al. 2012; Khataee et al. 2010; Yang et al. 2011). Therefore, the network weights could be used to calculate the relative importance of input variables as follows:

$$Ij = \frac{\sum_{m=1}^{m=Nh}\left(\left(\frac{\left|W_{jm}^{ih}\right|}{\sum_{k=1}^{Ni}\left|W_{km}^{ih}\right|}\right)\times\left|W_{mn}^{ho}\right|\right)}{\sum_{k=1}^{k=Ni}\left\{\sum_{m=1}^{m=Nh}\left(\frac{\left|W_{jm}^{ih}\right|}{\sum_{k=1}^{Ni}\left|W_{km}^{ih}\right|}\right)\times\left|W_{mn}^{ho}\right|\right\}} \quad (8)$$

where I_j is the relative importance of the jth input variable on the output variable; N_i and N_h are the numbers of input and hidden neurons, respectively; W is connection weight; the superscripts '*i*,' '*h*' and '*o*' refer to input, hidden and output layers, respectively; and subscripts '*k*,' '*m*' and '*n*' refer to input, hidden and output neuron numbers, respectively. The temperature with a relative importance of 28.67 % appeared to be the most influential parameter in the adsorption process of bamboo biochar for dye removal (Fig. 6b), followed by time (21.05 %), ionic strength (18.25 %), pH (17.77 %) and dye concentration (14.23 %). All the values of relative importance for parameters were above 10 %, indicating that all of the variables had strong effects on the adsorption capacity for metal-complex dye acid black 172. pH was found to be the most efficient parameter, followed by the initial dye concentration for the adsorption process of Lanaset Red G onto walnut husk (Çelekli et al. 2012). The initial dye concentration was found to be the most important factor, followed by pH for the acid black 172 onto the nonviable *Penicillium* YW 01 (Yang et al. 2011). It seems that the most influential variable depended upon the experimental ranges adopted in the fitting model.

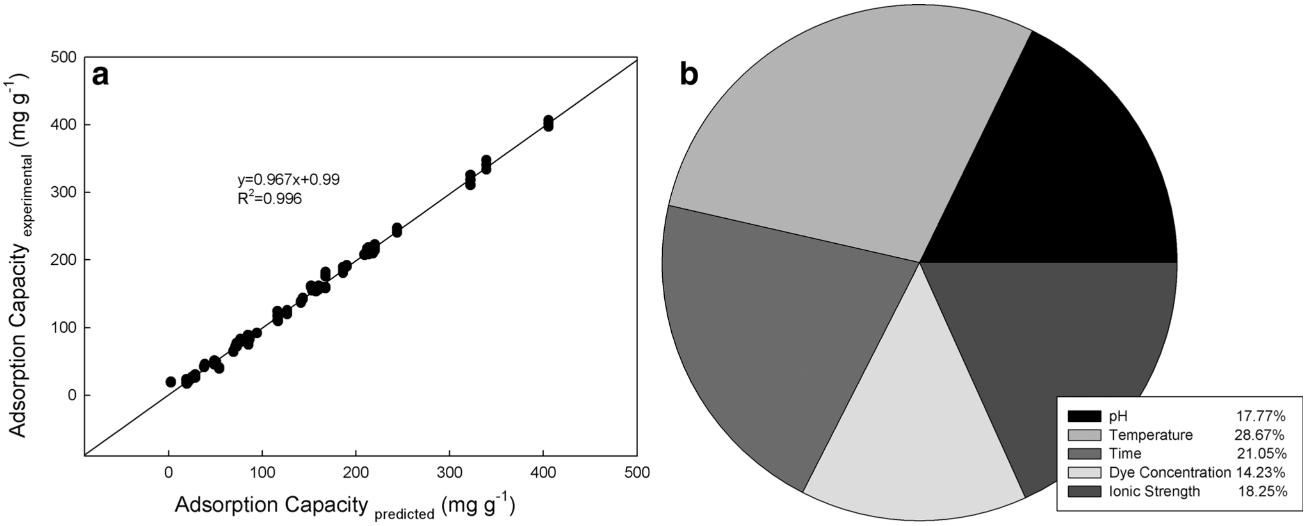

Fig. 6 Regression analysis of experimental results and predicted data (**a**) and sensitivity analysis (**b**) using artificial neural network

Conclusion

The bamboo biochar was found to be efficient as a low-cost adsorbent for the removal of metal-complex dye acid black 172 from aqueous solutions. The adsorption capacity of biochar for acid black 172 appeared to be less affected by ionic strength even at very high $NaNO_3$ concentration. Kinetic study revealed that intraparticle transport was not the only rate-limiting step. The temperature with a relative importance of 28.67 % appeared to be the most influential parameter in the adsorption process by artificial neural network. The results in this study provided the operational parameters for bamboo biochar used for removing acid dyes in effluents.

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