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Enhanced adsorption of Orange II on bagasse-derived biochar by direct addition of CTAB

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Abstract−Surface charge properties of an adsorbent always play an important role for the removal of contaminants from water. A cationic surfactant hexadecyl tri-methyl ammonium bromide (CTAB) was involved into adsorptive removal of Orange II (ORII) by bagasse biochars to realize an in-situ modification and an enhanced adsorption capability. Adsorption capacity of biochar (BC600) improved significantly from 1.66 mg/g in the absence of CTAB to 4.42 mg/g in the presence of 2.0 mg/L CTAB. A more hydrophobic surface of bagasse biochar was favorable for the dye uptake in the presence of CTAB. Linear pseudo-second-order kinetic model fitted the kinetics data better at three pH conditions than pseudo-first-order kinetic model, whether in the presence and absence of CTAB. Both nonlinear pseudo-first-order and pseudo-second-order kinetic models were suitable to describe the experimental data. The maximal adsorption capacity in the absence of CTAB was very limited (41.4 mg/g), while the adsorption isotherm curve in the presence of CTAB was almost linear, indicating a strong adsorption capability due to the introduction of CTAB. Direct addition of CTAB into wastewater is a potential technique for the enhanced removal of negatively-charged pollutants by bagasse biochar.

Keywords: CTAB, Orange II, Bagasse Biochar, Adsorption Kinetics, Adsorption Isotherm

INTRODUCTION

Considering water purification and wastewater treatment, various technologies have the capability for effective removal of most of the water-borne contaminants from water and wastewater. These technologies include bio-treatment technology [1], coagulation/flocculation [2], membrane [3], advanced oxidation technologies [4], and adsorption [5]. Although these technologies have their own superior advantages over other technologies for practical application, the adsorption process is regarded as one of the most powerful, efficient and cost-effective water treatment technologies due to ease of operation, universal nature and high efficiency [6,7]. It can simply transfer various pollutants from one phase to another effectively and efficiently. At the same time, a number of adsorbents can be selected for removal of different organic pollutants and heavy metals in view of specific water quality [6-9]. Therefore, adsorption is more adaptable for practical water treatment from this point of view.

Recent study has paid attention to low-cost adsorbents such as natural minerals, biomass and related composite adsorbents [7,10-12]. Among these low-cost adsorbents, biomass is usually accepted as one of the promising low-cost adsorbents for water purification because it has a huge annual generation. Nevertheless, one significant disadvantage of the raw biomass adsorbent is the dissolution of the raw biomass, which might lead to serious organic leaching and secondary pollution. Accordingly, biomass-based biochar has emerged as one of the most prominent adsorbent for water purification and wastewater treatment. Further, biochar has potential applications in environmental management such as soil improvement, waste management, climate change mitigation and energy production [11,13-15]. Stable pyrolytic biochar, a by-product generated during the pyrolysis of lignocellulose biomass to obtain biofuel, proves to be one of the best matrixes for adsorption of various pollutants. More and more studies have been focused on the adsorption capability of various biochars [16-21].

Even though, enhanced adsorption capability of an adsorbent is still highly anticipated in certain cases. Composite adsorbents, magnetic field and electric field were used to enhance the adsorption capability of related adsorbents [6,22,23]. Meanwhile, surfactants such as a cationic surfactant hexadecyl tri-methyl ammonium bromide (CTAB) were used to prepare surfactant-modified adsorbent, and the CTAB-modified adsorbents were used in the subsequent adsorption process. Researches have proven that the immobilization of CTAB onto an adsorbent substrate could significantly improve adsorption capability of the prepared adsorbent for the removal of negatively-charged pollutants such as dye congo red, weak acid scarlet [24-28].

As CTAB is normally applied in separation and adsorbent prepa-

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ration, release of CTAB unavoidably leads to some pollution. Yet, utilization of CTAB is still feasible considering its low concentration and subsequent purification process. However, so far, scant reports have been made on the direct introduction of surfactant such as CTAB into aqueous solution for the in-situ modification of adsorbent and enhanced adsorption capability. Meanwhile, various pollutants with different charge properties are present in the raw source of water and wastewater. Some surfactants even co-exist with these charged pollutants in some cases. The charge properties of adsorbents and pollutants always play an important role for the removal of various pollutants. In this research, a stable bagasse biochar was selected as an adsorbent and used for the adsorptive removal of an azo dye Orange II. Azo dyes are used for the production of textile, toys, pharmaceutical drug, foods and plastics. However, dyes in the wastewater are recalcitrant and tend to suppress photosynthetic activity in aquatic habitats by preventing the penetration of sunlight [29]. Quite interestingly, the surfactant CTAB was tentatively introduced into the solution directly in order to realize an in-situ modification of the biochar and enhanced adsorption capability. Adsorption kinetics, isotherm and mechanism were investigated and are discussed in this paper.

MATERIALS AND METHODS

1. Materials

Orange II (ORII) and cetyltrimethyl ammonium bromide (CTAB) were purchased from Beijing Chemical Reagents Company, and used without further purification. Other chemicals used were of analytical grade. Deionized (DI) water was used throughout the study.

2. Preparation of Sugarcane Bagasse Biochar

Sugarcane bagasse (bagasse) was collected from Guangxi province of China. It was washed, dried, crushed and sieved using a 100 mesh sieve. In a muffle furnace, the biochars from bagasse were prepared via pyrolyzing the bagasse biomass at different temperatures under oxygen-limited conditions. The furnace temperature was programmed to increase at a rate of 10 °C/min until it reached the desired temperature and kept for 2 h. Subsequently, the resultant bagasse biochars were put in a 4 mol/L HCl solution for 12 h and separated by filtration. Then the residues were rinsed with DI water until neutral solution pH was achieved and then dried in an oven at 80 °C overnight. The treated biochar was finally preserved in a desiccator before further use. For detailed procedures for the preparation of the bagasse biochars please refer to our previous study [30]. These bagasse biochars were designated as BC200, BC300, BC400, BC500 and BC600, respectively, wherein the suffix number represents the pyrolytic temperature.

3. Batch Adsorption of ORII in the Presence and Absence of CTAB

Batch adsorption of ORII onto the bagasse biochars in the presence and absence of CTAB was conducted in a series of 100-mL cylindrical flasks. The stock solutions of ORII (500 mg/L) were prepared in DI water. All working solutions/simulated wastewater were prepared by diluting the stock solution with DI water to the desired concentrations. Certain amount of CTAB was added into the prepared solution to achieve the required concentration. For the tests of adsorption isotherm and pH effect, 20 mg of bagasse biochar was added into 50 mL of ORII solution. These flasks were shaken on a horizontal shaker for 24 h at a speed of 140 rpm to achieve adsorption equilibrium based on the predetermined adsorption kinetics. For kinetic tests, a desired amount of bagasse biochar (200 mg) was added to a conical flask containing 500 mL of ORII solution with a concentration of 10 mg/L. Constant stirring was maintained by mechanical agitation for 24 h. Finally, samples were collected at desired time intervals. The reaction temperature was controlled at a constant of 298 K. All the solution pH was maintained at neutral pH except during the pH effect study. The solution pH adjustment was conducted by addition of diluted HCl or NaOH solution.

4. Analyses

Samples were collected and filtered through a 0.45 μm membrane before analyzing. The concentrations of ORII were determined by measuring the maximum absorbance at a fixed wavelength of 484 nm, using an UV8100B spectrophotometer (Labtech, China).

The removal efficiency of ORII was calculated as:

$$\text{Removal efficiency}=(1-C_t/C_0)\times 100\% \quad (1)$$

where C_t is the dye concentration at time t, and C_0 is the initial dye concentration.

The quantity of ORII adsorbed on the bagasse biochar was calculated by the following equation:

$$q_e=(C_0-C_e)\ V/W \quad (2)$$

$$q_t=(C_0-C_t)\ V/W \quad (3)$$

where q_e and q_t (mg/g) are the adsorption capacity at equilibrium and t min; C_0 is the initial concentration of ORII in solution, while C_e and C_t (mg/L) are the concentrations of ORII at equilibrium and t min, respectively; V (L) is the volume of solution, and W (g) is the mass of bagasse biochar used.

RESULTS AND DISCUSSION

1. Effect of Pyrolytic Temperature for Biochar Preparation on the Uptake of ORII

Not only can the increased pyrolytic temperature increase the biochar stability, but it also can regulate the surface characteristics of the biochars. As the surface characteristics of the biochars might influence their adsorption performance, the effect of the pyrolytic temperature for biochar preparation on the uptake of ORII was investigated in the presence and absence of CTAB, as presented in Fig. 1. In the absence of CTAB, an increase in the adsorption capacity was observed with increasing pyrolytic temperature, although the ORII uptake on BC200 and BC600 was only 0.63 and 1.66 mg/g, respectively. In contrast, once CTAB was introduced into the reaction mixture, the adsorption capacity on BC600 reached 4.42 mg/g and the ORII uptake was improved significantly. The adsorption capacity of BC200, BC300, BC400, BC500 and BC600 was enhanced by 109.5%, 66.4%, 129.7%, 132.0% and 166.3%, respectively. Our previous study demonstrated that the bagasse biochar surface became less hydrophilic, and a reduction of surface polar functional groups was observed with increasing pyrolytic temperature [30]. It can be deduced that, accompanied by the

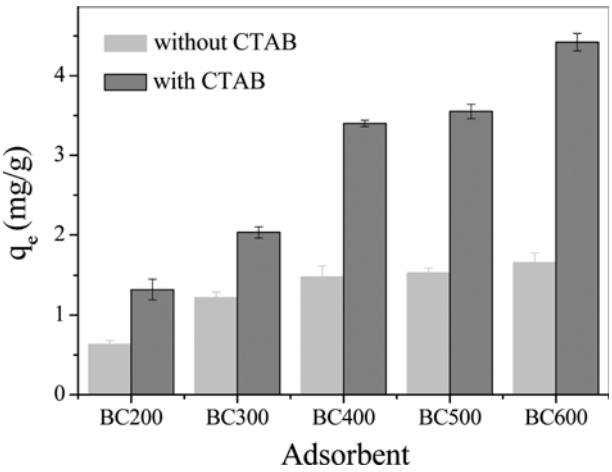

Fig. 1. Effect of CTAB concentration on the uptake of ORII. Biochar dose 20 mg, neutral solution pH.

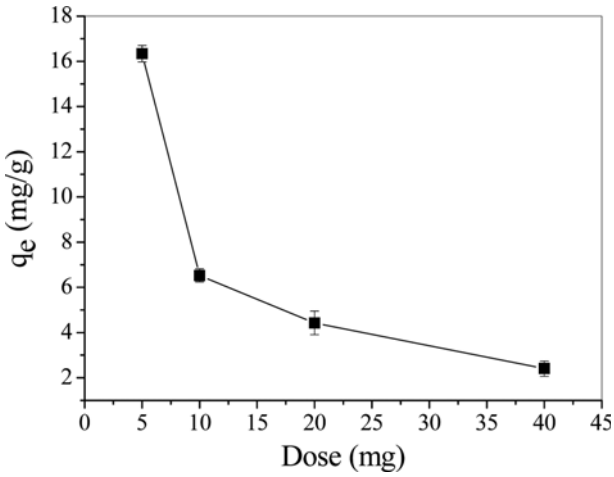

Fig. 3. Effect of biochar dose on the uptake of ORII. CTAB concentration 2.0 mg/L, neutral solution pH.

decrease of surface polar functional groups, a more hydrophobic biochar surface is favorable for CTAB to exert its influence on the water/biochar interface and subsequently enhance the ORII uptake. As such, considering the stability and adsorption performance, BC600 was employed for the following tests.

2. Effect of CTAB Concentration on the Uptake of ORII

The surfactant CTAB was innovatively added in the simulated wastewater instead of immobilization onto the adsorbent substrate. The effect of CTAB concentration on the uptake of ORII (5 mg/L) by BC600 was investigated, as illustrated in Fig. 2. The uptake of ORII at a CTAB concentration of 0, 1.0, 2.0, 5.0 and 10.0 mg/L were 1.66, 4.22, 4.42, 6.35 and 9.55 mg/g, respectively. An increase of CTAB concentration in the mixture increased the adsorptive removal of ORII obviously. As the raw biochar surfaces are normally negatively-charged [12], it is expected that the efficient uptake of negative-charged ORII molecules is quite difficult. As expected, the adsorption of ORII onto the bagasse biochar alone was quite low. Once CTAB was introduced into the mixture, the surface of bagasse biochar became more positively-charged, which facilitated the uptake of ORII dramatically. Considering the expenditure of application, the CTAB concentration of 2.0 mg/L was selected in the following study.

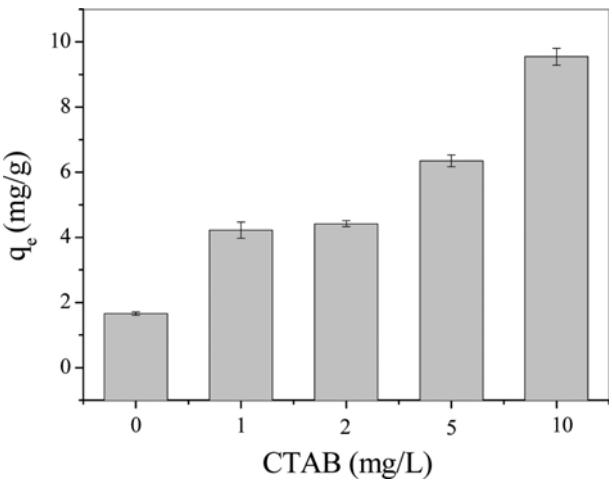

Fig. 2. Effect of CTAB concentration on the uptake of ORII. Biochar BC600 dose 20 mg, neutral solution pH.

3. Effect of Adsorbent Dose

At the CTAB concentration of 2.0 mg/L, the effect of biochar dose was examined concurrently. As presented in Fig. 3, the uptakes of ORII at the biochar BC600 dose of 5, 10, 20 and 40 mg were 16.34, 6.53, 4.42 and 2.41 mg/g, respectively. Although the ORII uptake decreased significantly, the removal percentage of the dye actually increased dramatically. Evidently, an increase in the bagasse biochar dose decreased the adsorption capacity of ORII onto the biochar as a consequence of the increased active sites for adsorption.

4. Effect of Initial Solution pH on Adsorption Kinetics

4-1. Linear Adsorption Kinetics

Adsorption kinetics for ORII onto the bagasse biochar BC600 in the presence and absence of CTAB was investigated and compared at pH 5.0, 7.0 and 9.0, respectively. Two typical kinetic models including pseudo-first-order and pseudo-second-order models were used to fit the experimental data. The mathematical representations of the linear and non-linear models of pseudo-first-order and pseudo-second-order kinetics are given in [31,32]:

$$q_t = q_e(1 - e^{-k_1 t}) \tag{4}$$

$$\ln(q_e - q_t) = \ln q_e - k_1 t \tag{5}$$

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

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \tag{7}$$

where q_e and q_t are the adsorption capacities (mg/g) at equilibrium and at time t (min), respectively; and k_1 (min^{-1}) and k_2 (g/mg·min) are the related adsorption rate constants for pseudo-first-order and pseudo-second-order model, respectively.

Apparently, judging from the experimental points and simulated curves in Fig. 4, a linear pseudo-second-order kinetic model

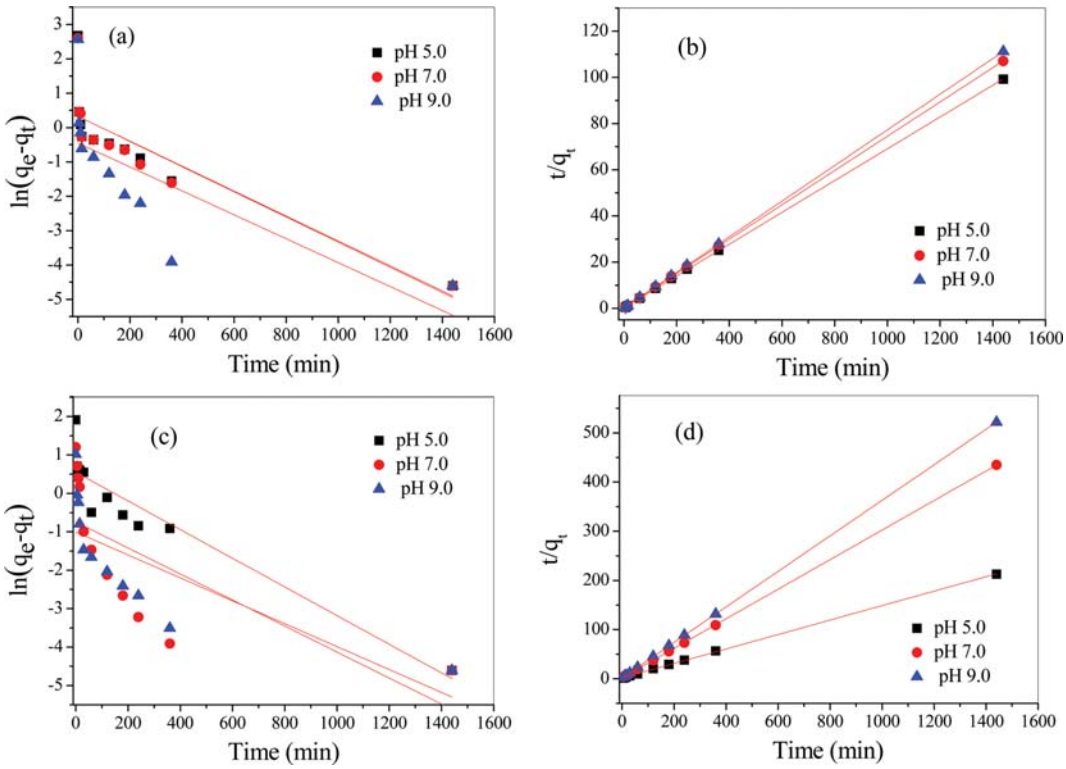

Fig. 4. Linear adsorption kinetic simulation for pseudo-first-order (a), (c) and pseudo-second-order (b), (d) models in the presence (a), (b) and absence (c), (d) of CTAB.

Table 1. The linear kinetic simulation parameters for pseudo-first-order and pseudo-second-order models

Model	Condition	Parameters	pH=5.0	pH=7.0	pH=9.0
Pseudo-first order	Without CTAB	R^2	0.873	0.464	0.548
		q_e (mg g^{-1})	1.73	0.48	0.37
		k_1 (min^{-1})	0.00373	0.00338	0.00298
	With CTAB	R^2	0.739	0.745	0.494
		q_e (mg g^{-1})	1.39	1.39	0.64
		k_1 (min^{-1})	0.00363	0.00363	0.00348
Pseudo-second order	Without CTAB	R^2	0.999	0.999	0.999
		q_e (mg g^{-1})	6.80	3.33	2.77
		k_2 (g mg^{-1} min^{-1})	0.0134	0.0553	0.0874
	With CTAB	R^2	0.999	0.999	0.999
		q_e (mg g^{-1})	14.53	13.47	12.96
		k_2 (g mg^{-1} min^{-1})	0.0190	0.0198	0.0568

fitted the kinetic data better at three pH conditions than pseudo-first-order kinetic model, whether in the presence and absence of CTAB. As presented in Table 1, the correlation coefficients (R^2) of the linear pseudo-first-order model were all less than 0.873, while those of the linear pseudo-second-order model were all 0.999. The calculated q_e values from linear pseudo-second-order kinetics were quite close to the experimental values. Apparently, the experimental data was well fitted only by linear pseudo-second-order kinetics model.

In the presence of CTAB, the calculated q_e values from linear pseudo-second-order kinetics were 14.53, 13.47 and 12.96 mg/g at pH 5.0, 7.0 and 9.0, respectively. However, in the absence of CTAB, the related q_e values from linear pseudo-second-order kinetics were only 6.80, 3.33 and 2.77 mg/g at pH 5.0, 7.0 and 9.0, respectively. At pH 7.0, the adsorption capacity for ORII on the biochar was increased by three times as a result of the introduction of CTAB. On the other hand, the uptake of ORII was observed to decrease with an increase in the solution pH, whether in the presence or absence of CTAB. ORII molecules are normally negatively-charged under all the pH conditions. Considering the presence of CTAB (2 mg/L), the biochar surface was deduced to be normally positively-charged, while it became more and more negatively-charged

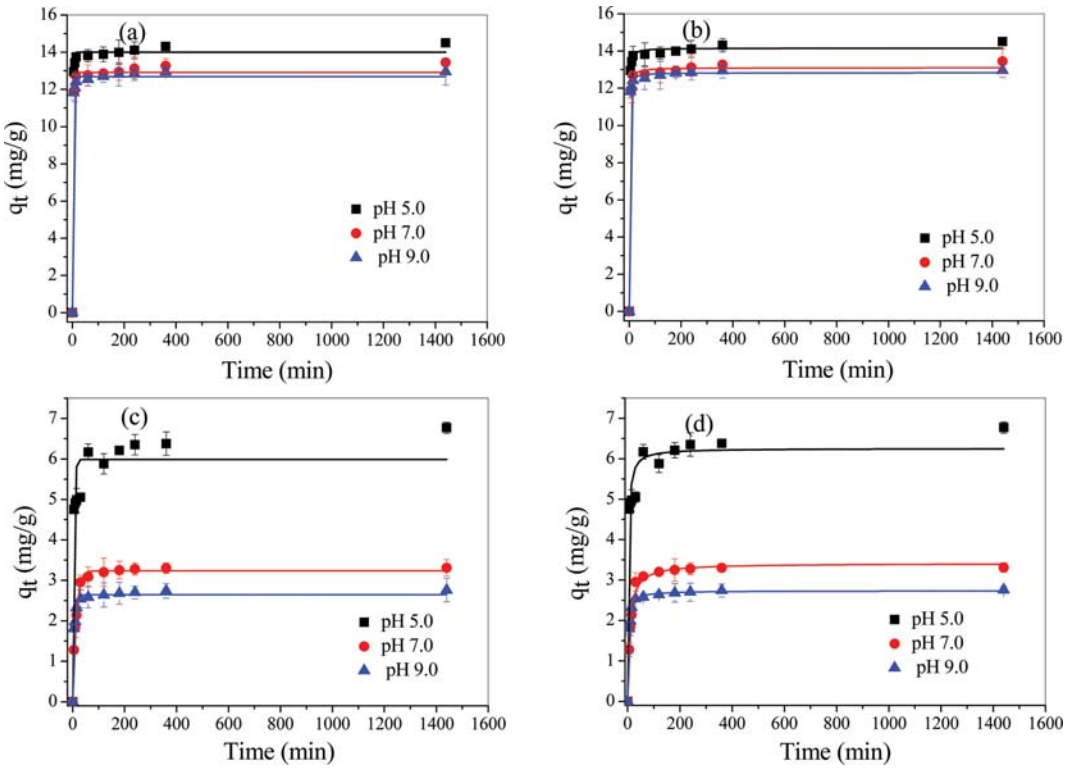

Fig. 5. Non-linear adsorption kinetic simulation for pseudo-first-order (a), (c) and pseudo-second-order (b), (d) models in the presence (a), (b) and absence (c), (d) of CTAB.

Table 2. The non-linear kinetic simulation parameters for pseudo-first-order and pseudo-second-order models

Model	Condition	Parameters	pH=5.0	pH=7.0	pH=9.0
Pseudo-first order	Without CTAB	R^2	0.898	0.992	0.971
		q_e (mg g^{-1})	5.98	3.24	2.64
		k_1 (min^{-1})	0.240	0.0822	0.178
	With CTAB	R^2	0.995	0.990	0.995
		q_e (mg g^{-1})	14.00	12.91	12.68
		k_1 (min^{-1})	0.501	0.477	0.519
Pseudo-second order	Without CTAB	R^2	0.954	0.991	0.994
		q_e (mg g^{-1})	6.25	3.41	2.73
		k_2 (g mg^{-1} min^{-1})	0.0664	0.0371	0.126
	With CTAB	R^2	0.998	0.996	0.999
		q_e (mg g^{-1})	14.15	13.10	12.83
		k_2 (g mg^{-1} min^{-1})	0.144	0.125	0.164

with an increase in the solution pH. The electrostatic repulsion force between BC600 and ORII molecules became stronger with increasing solution pH. As such, the increasing solution pH was not favorable for the adsorptive removal of negatively-charged pollutants.

4-2. Non-linear Adsorption Kinetics

Non-linear adsorption kinetics for ORII onto the bagasse biochar in the presence and absence of CTAB was also investigated and compared at pH 5.0, 7.0 and 9.0, respectively. As illustrated in Fig. 5, the adsorption processes typically consist of an especially rapid initial uptake and a subsequent smooth increase to equilibrium within 24 h. The removal efficiency at 5, 60 and 120 min reached 88.4%, 94.9% and 95.6%, respectively. Accordingly, almost all the ORII molecules were removed by the initial 60 min. By non-linear regressive method, the experimental kinetic data for ORII adsorption under the three pH conditions were simulated by pseudo-first-order and pseudo-second-order models, as illustrated in Fig. 5. Meanwhile, the parameters for the two models at pH 5.0, 7.0 and 9.0 are summarized in Table 2 for comparison. From Fig. 5, both non-linear pseudo-first-order and pseudo-second-order kinetic models were suitable to describe the experimental points as the experimental data were particularly close to the fitted curves. The R^2 values of pseudo-second-order model are all higher than 0.954, while

those of non-linear pseudo-first-order model were all higher than 0.898. This indicates that non-linear pseudo-second-order model was still slightly better than pseudo-first-order model. Additionally, in the presence of CTAB, the calculated q_e values from non-linear pseudo-second-order model at pH 5.0, 7.0 and 9.0 were 14.15, 13.10 and 12.83 mg/g, respectively. However, in the absence of CTAB, these values at pH 5.0, 7.0 and 9.0 were 6.25, 3.41 and 2.73 mg/g, respectively. The effect of CTAB was still pronounced by non-linear kinetic fitting. Additionally, the decreased q_e values with increasing solution pH, which corresponded to the increased k_2 values by increasing pH accordingly. As the biochar BC600 surface was expected to become slightly negatively charged with increasing solution pH, whether in the absence and presence of CTAB, it could be expected that the uptake of normally negatively-charged ORII molecules would be inhibited as solution pH increased [33,34].

As we know, the bagasse biochar BC600 possesses more graphitized surfaces with higher π-electron density. Aromatic ORII molecules are regarded as π-electron acceptors and biochar B600 as π-electron donors. A mechanism of π-π electron-donor-acceptor (EDA) interaction contributes to the uptake of ORII [35-37]. On the other hand, ORII molecules are normally negatively-charged, and electrostatic attraction might play an important role as well. Even in the absence of CTAB, a decrease in solution pH could improve the adsorption of ORII, which demonstrates an important role of electrostatic attraction. After the introduction of CTAB, the biochar surface becomes more positively-charged, which facilitates the uptake of ORII significantly. In contrast, in the presence of CTAB, the adsorption capacities of ORII under the three pH conditions are very close to each other, while those in the absence of CTAB are largely different. This indicates that the effect of CTAB performed excellently, whether under acidic and alkaline conditions, which facilitates the application of this technique. From this point of view, the electro-static force plays a dominant role for the uptake of ORII on the bagasse biochar.

5. Adsorption Isotherms

Both Langmuir and Freundlich isotherm models were employed to describe the adsorption isotherms [38,39]. The two equations can be expressed as follows:

Langmuir model: $$q_e = \frac{q_m k_L C_e}{1 + k_L C_e} \quad (8)$$

Freundlich model: $$q_e = k_F C_e^{1/n} \quad (9)$$

where q_e and q_m represent the amount of equilibrium adsorption capacity and the maximum adsorption capacity (mg/g), respectively; k_L (L/mg) is the Langmuir coefficient; C_e is the equilibrium concentration (mg/L); k_F is roughly an indicator of the adsorption capacity; n is the heterogeneity factor.

As illustrated in Fig. 6, in the absence of CTAB, both Langmuir and Freundlich models could well describe the isotherm data. From the fitted curves, it can be observed that the R^2 values of Langmuir and Freundlich model are 0.985 and 0.892, respectively. Langmuir model was more suitable to fit the isotherm data than Freundlich model. This indicates that the surface of the bagasse biochar was more homogeneous to some extent. By Langmuir model, the maximal adsorption capacity in the absence of CTAB achieved 41.4 mg/g. In contrast, as presented in Fig. 6, the simulated curve of the adsorption isotherm data in the presence of CTAB was almost linear (R^2=0.977). Both Langmuir and Freundlich models failed to fit the experimental data. Meanwhile, once CTAB was introduced into the reaction mixture, the adsorption of CTAB on the surface of bagasse biochar BC600 could be governed mainly by cationic exchange and hydrophobic interaction [40,41]. The uptake of CTAB overwhelmingly changed the surface charge properties of the BC600, and abundant positively-charged active sites appeared on the surface. Consequently, the fully positively-charged biochar surface has the capability to bond a number of purely negatively-charged ORII molecules. As such, the in-situ change of the charge properties of the biochar BC600 occurred as a result of CTAB introduction while an in-situ bonding underwent concurrently between the refreshed adsorbent surface and ORII molecules. The scheme for the uptake of ORII in the presence of CTAB is in Fig. 7. Considering the limited adsorption capability and regeneration cost of a CTAB-modi-

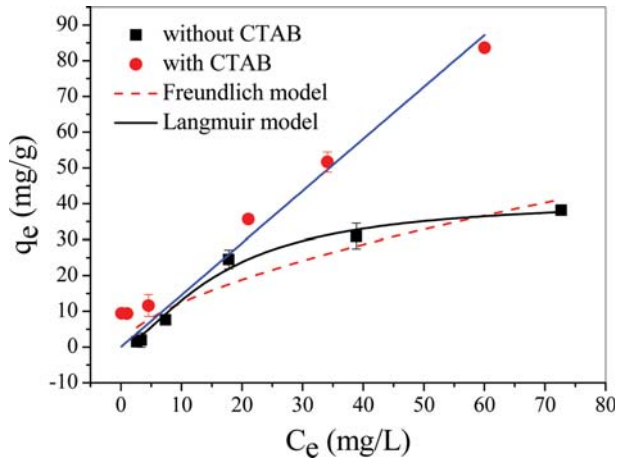

Fig. 6. Adsorption isotherms for the uptake of ORII in the presence and absence of CTAB.

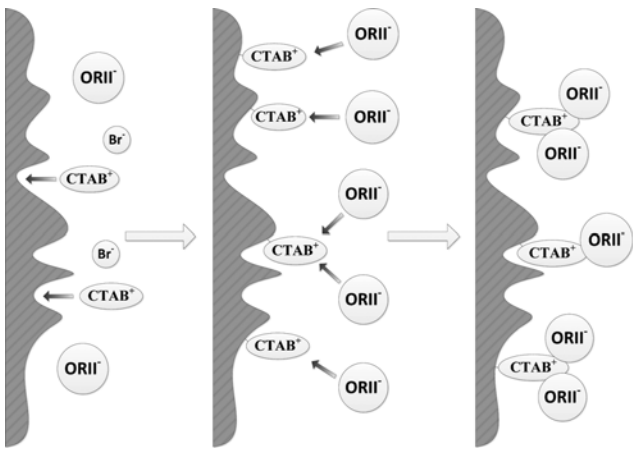

Fig. 7. Scheme for the uptake of ORII in the presence of CTAB. Step1: Adsorption of CTAB and positively-charged biochar interface forms, Step2: Diffusion of ORII molecules from bulk solution to biochar interface, and Step3: Adsorption of ORII molecules on the biochar.

fied adsorbent, it is deduced to be advisable to select the in-situ CTAB modification and adsorption. Hence, the direct addition of CTAB into the wastewater might be a promising and alternative technique for the adsorptive removal of negatively-charged pollutants.

CONCLUSION

The effect of CTAB on the adsorption of azo dye Orange II onto bagasse biochar was investigated systematically. An increase of CTAB concentration in the mixture increased the adsorptive removal of the dye obviously. An increase in the bagasse biochar dose decreased the uptake of ORII onto the biochar as a consequence of the increased active sites for adsorption. A more hydrophobic surface of bagasse biochar was favorable for the dye uptake in the presence of CTAB. Linear pseudo-second-order kinetic model fitted the kinetics data better at three pH conditions than pseudo-first-order kinetic model, whether in the presence and absence of CTAB. Both pseudo-first-order and pseudo-second-order kinetic models were suitable to describe the experimental points, as the experimental data were particularly close to the fitted curves. Adsorption isotherm curve in the presence of CTAB was almost linear, which was much different from that of the raw biochar adsorption.

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