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Simultaneous removal of acid dye and cationic surfactant from water by bentonite in one-step process

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Abstract

Organobentonite is suggested as potential super-sorbents for the removal of organic pollutants such as dyes from wastewater. Limited by the high operation cost and the complicate process of its synthesis, however, organobentonite is not widely applied in wastewater treatment. Based on the truth that organobentonite was normally synthesized by cationic surfactants and bentonite with cationic exchanging, a one-step process was proposed here to remove an acid dye (Orange II) and a cationic surfactant (cetyltrimethylammonium bromide, CTMAB) simultaneously from wastewater using bentonite alone. The effects of contact time, pH and inorganic salt on the removal of the acid dye and surfactant from wastewater were also examined. High removal efficiencies (>99%) of Orange II and CTMAB by bentonite were observed. In the one-step process, the cationic surfactants in the wastewater were utilized to form organobentonite and then available for the removal of acid dye as sorbents. Furthermore, the separated synthesis process of organobentonite was consequently deleted. Therefore, this one-step process would be an efficient, simple and low cost for the treatment of organic wastewater especially for textile wastewater and makes it possible that bentonite could be applied widely in wastewater treatment as sorbent.

Keywords: Bentonite; Sorption; Cationic surfactant; Acid dye; Wastewater treatment; One-step process

1. Introduction

Bentonite is a very rich clay mineral, consists of layers of two tetrahedral silica sheets sandwiching one octahedral alumina sheet. Though bentonite did well in sorption of basic dyes [1–3], it is failed in sorption of acid dyes [4]. However, organobentonite, normally synthesized by cationic surfactants and bentonite with cationic exchanging, was observed to adsorb acid dyes well [5,6]. Organobentonite is also widely suggested as a potential super-sorbents for the removal of other organic pollutants from wastewater [7–9].

Organobentonite should be synthesized, before it was applied to wastewater treatment [9,10]. The mostly used synthesis process included: (i) pulverizing bentonite, (ii) mixing bentonite and cationic surfactants solution for several hours, and then (iii) the product was centrifuged, washed, dried and ground. However, this synthesis requires large quantity of surfactants, water and power, having a complex and time consuming process [8,11], which results in the high cost of organobentonite and limits its application in wastewater treatment.

In wastewaters from pigments, dyestuffs or textiles manufactures, cationic surfactants are always coexisted with dyes [12]. Cationic surfactant such as quaternary ammonium compounds could significantly reduce the activity of cells, which may inhibit the biodegradation of other pollutants [13]. Based on the truth that organobentonite was normally synthesized by cationic surfactants and bentonite with cationic exchanging [8,14], it gives a possibility that cationic surfactants in wastewaters could be utilized to synthesize the organobentonite directly in the wastewaters. And then, this organobentonite would be available for the removal of acid dye as sorbents. So the solely added bentonite may achieve the surfactant and dye removed from those wastewaters in a simple process.

Therefore, a one-step process was suggested in this study to remove the surfactant and dye simultaneously by bentonite from wastewater. Due to the deletion of the separated organobentonite synthesis process and the utilization of surfactants in wastewater, the operation cost of wastewater treatment could be reduced. It is also hoped that the biodegradability of the wastewater would be improved by the removal of the nonbiodegradable

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organic pollutants and antimicrobial cationic surfactants after the wastewater was treated by the one-step process. Furthermore, the sorbed organobentonite could be recycled [15]. The objectives of this study are (i) to determine the feasibility to both synthesis of organobentonite and removal of acid dye (Orange II) and cetyltrimethylammonium bromide (CTMAB) from simulated wastewater in one-step process; (ii) to investigate the main effects on the sorption, such as pH and the initial concentration of cationic surfactant and inorganic salt; and (iii) to reveal the removal mechanism of the sorption of Orange II on bentonite.

2. Materials and methods

2.1. Materials

The natural bentonite composed primarily of Ca^{2+}-montmorillonite, was obtained from Tianyu Co., Ltd. (Inner Mongolia, China). Its cation exchange capacity (CEC) is 1.084 mmol/g bentonite. The bentonite samples were gently ground to powder. CTMAB, Orange II (color index number: 15510) and Orange G (color index number: 16230) were provided from CR Co. Ltd., Shanghai (China) and they were of analytical grade. All the reagents were used as received.

2.2. Sorption experiments

For equilibrium measurements, a known amount of the bentonite was added into a set of 22 mL centrifuge tubes, containing a known amount of Orange II and CTMAB. The concentrations of Orange II ranged from 500 to 3000 mg/L in the presence of CTMAB with fixed concentration at 7.9 g/L. The tubes were capped and placed on an orbital shaker with 180 rpm for 5 h to ensure apparent equilibrium. Preliminary kinetic investigations revealed that sorption equilibrium was reached in less than 3 h. When the equilibrium was attained, the sorbent was separated by centrifugation at 3000 rpm for 15 min. After the supernatant was analyzed for residual concentrations, the sorbed amount was calculated. This procedure was used in all batch sorption experiments.

For kinetic measurements, 1.25 g bentonite and 0.492 g CTMAB with 0.5 g Orange II was added to 1000 mL distilled water and continuously agitated. In the predefined time intervals, small portions of the mixture were taken out (5 mL), and the concentrations of the dye were determined. All the sorption experiments were carried out at ambient laboratory temperature (25 ± 1 °C).

The influence of pH on the sorption was studied using the solution with different pH (from 3 to 12) at the initial concentrations of 500 mg/L and 740.8 mg/L, respectively, for Orange II and CTMAB. The pH was adjusted by adding 1 mol/L HCl or 1 mol/L NaOH. The effect of NaCl concentration was tested in the range from 0.5 to 100 mmol/L.

Results of control experiment indicate the negligible evaporation and sorption on glass surface. All samples were run in triplicates under identical conditions.

2.3. Analyses

Concentrations of the Orange II and Orange G in solution were determined spectrophotometrically using a UV/Vis spectrophotometer Shimadzu UV-2450 (Shimadzu, Japan). For every investigated system, the complete UV–vis spectra were measured in the presence of the respective additives, such as inorganic salts or surfactants, to examine an effect on the spectrum shapes and thus to avoid a misinterpretation of the spectrophotometric determinations [16]. The result of control experiment showed that CTMAB at low concentration (<0.01 mol/L) had negligible effect on the examination of dyes. Total organic-carbon (TOC) was determined by a total organic carbon analyzer (TOC-V CPH, Shimadzu). The amount of sorbed CTMAB was calculated by a simple mass balance. Bromide ion concentration was analyzed by an ion chromatograph (IC-1000, Techcomp Ltd.).

X-ray diffraction (XRD) patterns of the prepared samples were acquired with an X-ray diffractometer (Max-2550PC, Rigaku D) using Cu Kα radiation (40 kV, 300 mA). All XRD patterns were obtained from 0.5° to 30° with a scan speed of 4°/min. The sample of natural bentonite was dry. The sorbed samples were the centrifugal sediment without any drying.

3. Results and discussion

3.1. Kinetics of sorption

The results of the kinetics experiments of sorption of CTMAB alone and co-adsorption with Orange II at 25 °C are shown in Fig. 1. The sorption of both Orange II and CTMAB reached equilibrium within 2 h.

The rates of sorption were determined by testing pseudo-first order and pseudo-second order kinetic models. The correlation coefficients of CTMAB sorption obtained from pseudo-second order model (0.998–0.999) are higher than the pseudo-first order model.

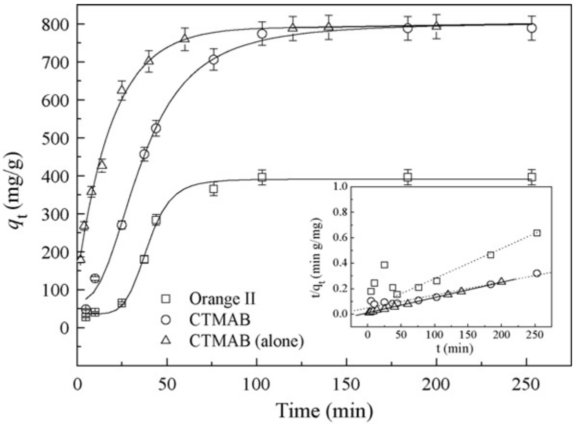

Fig. 1. Sorption of CTMAB and Orange II onto bentonite as a function of time (insert: plots of pseudo-second-order kinetic model for the adsorption). Bentonite dose: 1.25 g/L, CTMAB: 987.8 mg/L, Orange II concentration: 500 mg/L.

Table 1
Pseudo-second-order kinetic parameters for the sorption of Orange II and CTMAB

	k ($\times 10^{-4}$) (g/(mg min))	q_e (mg/g)	r^2
CTMAB	0.22	980.39	0.998
CTMAB (alone)	1.36	833.33	0.999
Orange II (45 min later)	1.64	423.7	0.998

The pseudo-second-order kinetic model is expressed as[17]:

$$\frac{dq_t}{dt} = k(q_e - q_t)^2 \quad (1)$$

where q_t is the sorbed amount at time t (mg/g), k is the equilibrium rate constant of the second-order sorption (g/mg min) and q_e is sorbed amount at equilibrium (mg/g). Integrating Eq. (1) for the boundary conditions $t = 0$ to $t = \mathrm{t}$ and $q_0 = 0$ to $q_t = q_t$, gives:

$$\frac{1}{(q_e - q_t)} = \frac{1}{q_e} + kt \quad (2)$$

Eq. (2) can be rearranged to obtain a linear form:

$$\frac{t}{q_t} = \frac{1}{(kq_e^2)} + \left(\frac{1}{q_e}\right)t \quad (3)$$

Rate parameters, k and q_e, can be directly obtained from the intercept and slope of the plot of (t/q_t) against t. The equilibrium concentration can be further calculated as the value of q_e has been obtained from the fitting of Eq. (3).

The summarized kinetic parameters acquired from fitting results are shown in Table 1. It should be noted that the sorption of Orange II was fit neither pseudo-second nor pseudo-first order model well. However, 45 min later the coadsorption, the sorption result fitted the pseudo-second order model well. That implied that the sorption of Orange II is correlated with the sorption of CTMAB.

3.2. Sorption isotherms

The coadsorption of Orange II and CTMAB isotherm data at various initial concentrations are presented in Fig. 2. Adsorption isotherms models such as Langmuir and Freundlich were tested to fit the experimental data and the model parameters are compared in Table 2. The sorption of Orange II, alone, on bentonite was also tested (the data were not shown). The result verified that bentonite had no affinity to Orange II for the repellence effect of the anionic chargers [4].

Table 2
Langmuir and Freundlich constants for the sorption of Orange II and CTMAB

		Q_{max} (mg/g)	b ($\times 10^{-3}$L/mg)	r_L^2
Langmuir	CTMAB (alone)	982.7	84.1	0.986
	CTMAB	980.8	83.8	0.984
	Orange II	868.1	5.43	0.920
		K_F (mg/g)	n	r_F^2
Freundlich	CTMAB (alone)	532.5	13.3	0.668
	CTMAB	532.3	13.1	0.651
	Orange II	302.5	8.2	0.864

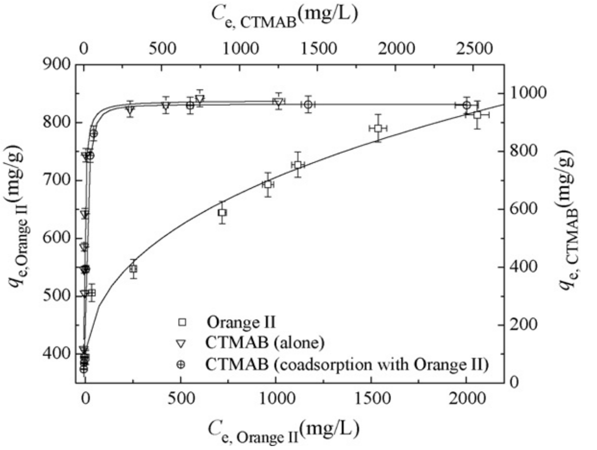

Fig. 2. Isotherms of Orange II sorption onto bentonite in presence of CTMAB at various temperatures and sorption of CTMAB in the presence/absence of Orange II.

The generalized Langmuir isotherm can be written in the form[18]:

$$\frac{Q}{Q_{max}} = \frac{bC_e}{(1 + bC_e)} \quad (4)$$

where b is the Langmuir constant; Q_{max} is the maximum adsorption amount; C_e is the equilibrium concentration. On the other hand, the Freundlich equation is [19]

$$Q = K_F C_e^{1/n} \quad (5)$$

where K_F (L/g) and n are the Freundlich constants and indicators of adsorption capacity and adsorption intensity, respectively.

For the examined Orange II and CTMAB, best fit was achieved using the Langmuir isotherm. The high adsorption capacity at about 787 mg/g was achieved (shown in Fig. 2). The maximum sorbed amount of CTMAB was about 265% CEC of the bentonite, which was similar to the sorption of CTMAB on bentonite in the absence of Orange II. The result showed that the presence of Orange II had a little effect on the sorption of CTMAB. High sensibility of sorbed amount towards concentration variations at low equilibrium concentration suggested high affinity between CTMAB and the adsorbent surface. The saturation level of the isotherm was reached at very low equilibrium concentration (approximately 3.84 mg/L) and the high adsorption capacity, indicating a high degree of irreversibility of the adsorption system [1].

3.3. Effect of surfactant concentration on sorption of Orange II

A detailed study was performed to elucidate the influence of initial CTMAB concentration on the sorption of Orange II and the results are given in Fig. 3. When the amount of bentonite was maintained at 25 mg, as the initial CTMAB concentration increased from 49.4 to 1481.7 mg/L, the removal efficiency of Orange II sharply increased from 7.1% to 99.8% then decreased

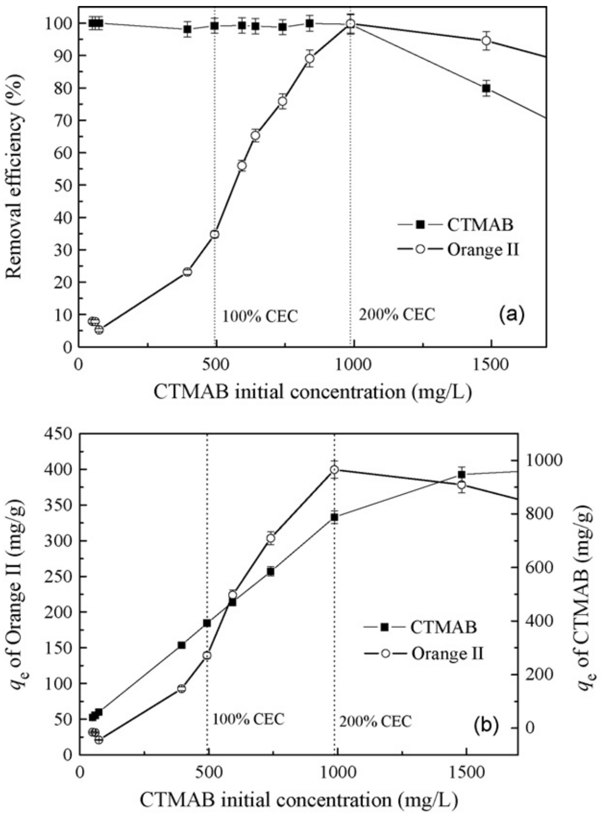

Fig. 3. Effect of initial CTMAB amount on the sorption. (a) Removal efficiency of CTMAB and Orange II and (b) sorption amount of CTMAB and Orange II. Bentonite dose: 1.25 g/L, Orange II concentration: 500 mg/L.

to 79.9%. The maximum removal efficiency of Orange II was achieved at CTMAB initial concentration of 987 mg/L (the amount of CTMAB is at about 200% CEC of added bentonite). The removal efficiency of TOC more than 99% was obtained under that condition.

As shown in Fig. 3a, CTMAB ($CTMA^+$) was almost completely removed when the initial concentration was no more than 987 mg/L. In this region, $CTMA^+$ is firstly anchored in the interlayer of bentonite by cationic exchange [20]. Then when the sorption amount exceeded 100% CEC, CTMAB molecule was sorbed. van der Waals hydrophobic interactions are suggested to operate in such cases and lead to a bilayer of interdigitated alkyl chains [21,22]. The sorbed amount of Orange II and CTMAB was calculated and is shown in Fig. 3b. It was found that the sorbed amount of Orange II increased nearly linear with the increase of initial concentration of CTMAB when the initial concentration of CTMAB was in the range of 310–987.8 mg/L, then reached a peak and decreased. Similar decrease was observed in sorption of basic dye on iron humate when the SDS concentration was more than CMC [23]. The steeply suppression of sorption is resulted from the micelle formation and dye solubilization. However, interestingly, the residue CTMAB concentration in this study was very low and far from CMC, when the sorbed amount began to decline. The decrease may be ascribed to the pore-blocking effect. When the concentration of CTMAB increased, the sorbed amount of CTMAB increased and reached to the maximum amount close to 260% CEC of bentonite. Previous configurations study on the sorption of cationic surfactants on bentonite found that the packing density abruptly increased when the sorbed amount was beyond 200% CEC [22,24], which may hinder the accessibility of Orange II to reach the affinity site and then led the sorption amount of Orange II to decrease.

3.4. Sorption mechanism

The highest sorbed amounts of Orange II and CTMAB were 399.6 mg/g (1.15 mmol/g) and 787.2 mg/g (2.16 mmol/g), respectively. It is surprisingly found that the maximum sorbed amount mole ratio of Orange II to CTMAB is close to 1:2. It could be supposed that Orange II may be sorbed by anionic exchange with bromide ion from the CTMAB molecule that was sorbed into the interlayer of bentonite by van der Waals hydrophobic interactions. The schematic diagram of the structure of bentonite unit sorbed CTMAB and Orange II is shown in Fig. 4. The residual concentration of bromide was determined to reveal its state in the system. It was found that almost all the bromide ions were remained in the solution when CTMAB initial concentration is below 987.8 mg/L. Normally, when the sorption of CTMAB was above 100% CEC, the counterion Br^- would be sorbed with $CTMA^+$ as ionpairs onto bentonite by tail–tail interactions [20,21]. However, almost all Br^- dissociated from sorbed $CTMA^+$ in that range. The mole of exchanged Br^- was calculated (shown in Fig. 5) and found it linearly increased with the increase of sorbed mole of Orange II. It is surprisingly found that almost two Br^- isolated from $CTMA^+$ led to one Orange II ion sorbed on bentonite in the range of 100–200% CEC.

XRD patterns were employed for the investigation of the structure of the sorbed bentonite layer. The result is shown in Fig. 4. The X-ray results showed the basal spacing of bentonite was swelled from 1.55 to 4.85 nm after sorbed 200% CEC $CTMA^+$ and Orange II. However, the interlayer spacing of bentonite only sorbed 200% CEC CTMAB is 4.12 nm, which is smaller than that with both CTMAB and Orange II sorbed. The results indicated that the Orange II was sorbed into the interlayer. Referred to our previous research [22], the increase of interlayer spacing suggests that the $CTMA^+$ radiated away from the silicate surface, forming an extended structure with a tilt angle. The CTMAB was sorbed among the $CTMA^+$ by van der Waals interaction. When Orange II was sorbed, the Br^- was exchanged by the Orange II anion and the interlayer spacing was increased to 4.85 nm.

The stoichiometric reaction of overall ionic exchange in the specific region of initial CTMAB amount between 100% and 200% CEC can be written as:

$$\text{bentonite–CTMA} + \text{CTMA}^+ + \text{R–SO}_3{}^- \rightleftarrows \text{bentonite–(CTMA)}_2\text{–SO}_3\text{–R} \qquad (6)$$

with a coefficient, K, defined as [25]

$$K = \frac{[\text{bentonite–CTMA}_2\text{–SO}_3\text{–R}]}{[\text{bentonite–CTMA}][\text{CTMA}^+][\text{R–SO}_3^-]} \qquad (7)$$

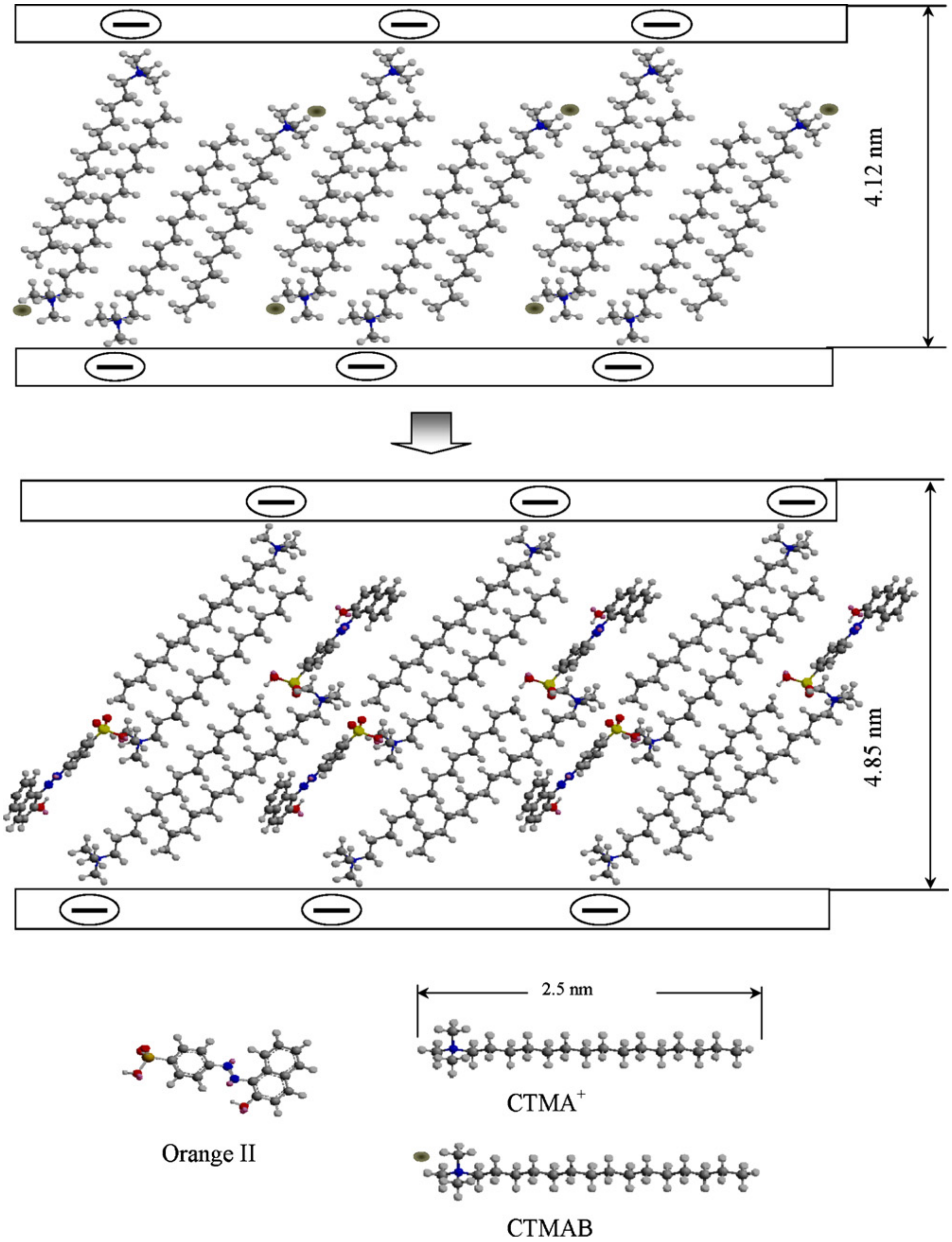

Fig. 4. Schematic diagram of the structure of bentonite unit sorbed $CTMA^+$ and Orange II.

where $R{-}SO_3^-$ stands for Orange II ion. The square brackets denote concentrations in mmol/L for the solution phase and mmol/g for the sorbed phase. *K* can be calculated using linear regression. The bentonite molecular weight was regarded as 1/1.084 g/mmol (The bentonite CEC is 1.084 mmol/g bentonite.). So the initial [bentonite] is 1.355 mmol/L. Equilibrium [bentonite–$CTMA_2{-}SO_3{-}R$] could be calculated from the difference between the initial and equilibrium solution [$R{-}SO_3^-$]. These results also followed a straight line (Fig. 6). A good correlation coefficient was obtained at $r^2 = 0.996$.

The sorption of another acid dye, Orange G, which is divalent anion in the solution, was studied at various initial concentration of CTMAB. The result is shown in Fig. 7. The similar trends of sorption amount and removal efficiency were obtained to the sorption of Orange II. However, the maximum sorption molar amount is almost the half of Orange II sorbed. The result implied that one Orange G molecule might be exchanged with two Br^-, which decreased the sorbed amount. Ozcan et al. [14] calculated the data of sorption of acid dye (Acid Blue 193) on organobentonite modified by 200% CEC surfactant. Their results showed that the maximum sorption amount was about half of the molar amount of cationic surfactant. These results further verified that acid dye was mainly sorbed by anion exchange on surfactant modified bentonite.

It was observed that there was a little acid dye sorbed at low loading amount of CTMAB. There were two reasons, (i) $CTMA^+$ firstly sorbed on the broken-bonds surface, neutralised the negative surface and led to the acid dye sorbed on the outside

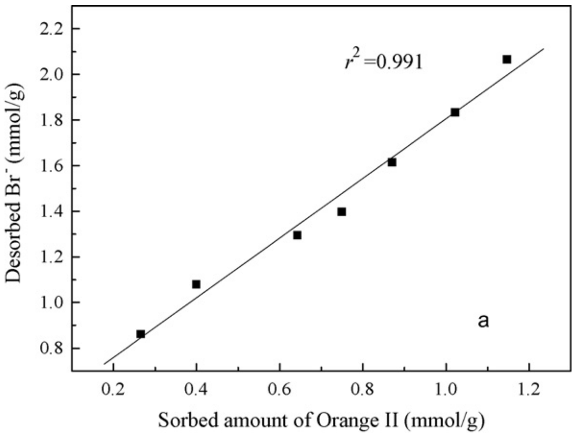

Fig. 5. Relationship between desorbed Br$^-$ and sorbed Orange II.

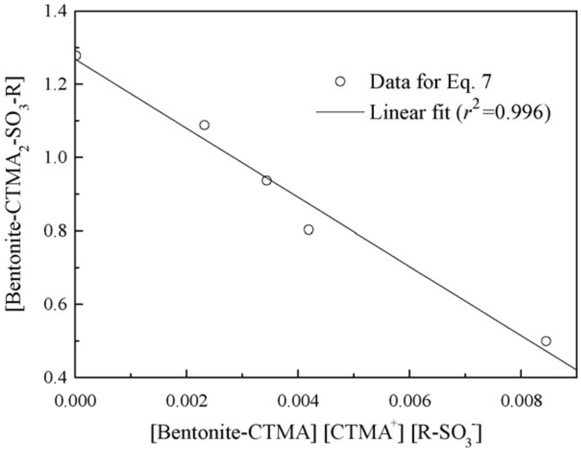

Fig. 6. Plot of the equilibrium of [bentonite—CTMA$_2$—SO$_3$—R] vs. [Bentonite—CTMA] [CTMA] [R—SO$_3$$^-$].

the interlayer space; (ii) the bilayer of interdigitated alkyl chains was formed even at low loading of CTMAB, the sorbed CTMAB will favor the sites just next to the previously adsorbed surfactants [21], to minimize the contact surface with water, that resulted in more active site were available at low loading amount.

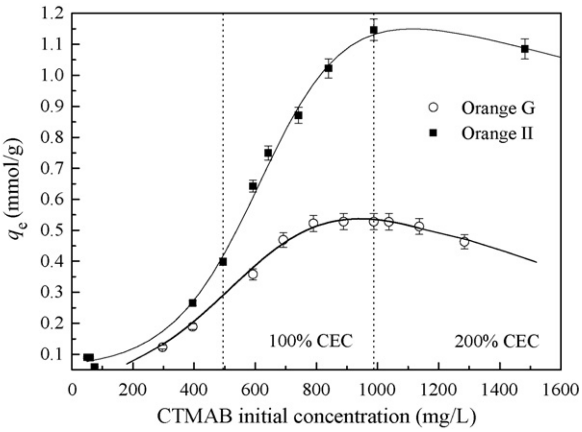

Fig. 7. Effect of initial CTMAB amount on the sorption of Orange G. Bentonite dose: 1.25 g/L, Orange G concentration: 300 mg/L.

3.5. *Effect of pH and inorganic salt on the removal*

The sorption of CTMA$^+$ on bentonite is mainly dominated by cation-exchange. So the sorption capacity may vary with pH and the concentration of inorganic salt, which may influence the removal of Orange II. The effect of pH and NaCl concentration on the sorption was tested. The result showed that the TOC removal efficiency was higher than 98.5% in all pH range. The sorption of Orange II in acidic region was slightly higher than that in basic region, which showed that pH had a minor influence on the dye sorption. At lower pH, more protons will be available, thereby increasing electrostatic attractions between negatively charged dye anions and positively charged sorption sites and causing an increase in dye sorption. When the pH increased, the sorbent surface appeared negatively charged, which resulted in ionic repulsion between the negatively charged surface and the anionic dye molecules [14].

Although at relatively high concentrations of NaCl (100 mmol/L), no influence on the dye sorption was observed. The TOC removal efficiency was higher than 99.1%. Evidently, the dyes exhibit greater selectivity toward the sorbent and are not “pushed out” by the metal cations at that concentration, which is important for applications to real wastewaters treatment. El-Nahhal [26,27] found that the adsorption of pesticide and phenanthrene on organobentonite increased in comparison to salt-free solutions. However, if the NaCl concentration was higher than 800 mmol/L, the sorption of organic cation would decrease [27].

4. Conclusion

Organobentonite as a sorbent could be synthesized and simultaneous removal of acid dye and cationic surfactant from wastewater by bentonite in one step process. The experimental isotherms data were fitted well to the Langmuir-type equilibrium equation and bentonite had very high sorption capacity for Orange II and CTMAB of 868.1 mg/g and 980.8 mg/g, respectively. Maximum removal efficiencies (>99.6%) of the Orange II and CTMAB were achieved in aqueous solutions. Orange II was sorbed mainly due to anionic exchange with Br$^-$ of the sorbed CTMAB in the interlayer of bentonite. The inorganic salt and pH had negligible effects on the sorption. The results indicate that one-step process is promising for bentonite application as sorbent in industrial scale for wastewater treatment especially for textile wastewater.

Acknowledgments

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