

## Adsorption of dye from wastewater using chitosan–CTAB modified bentonites

Jianzhong Guo<sup>a,\*</sup>, Shunwei Chen<sup>b</sup>, Li Liu<sup>a</sup>, Bing Li<sup>a</sup>, Ping Yang<sup>a</sup>, Lijun Zhang<sup>a</sup>, Yanlong Feng<sup>a,\*</sup>

<sup>a</sup> Department of Chemistry, College of Science, Zhejiang A & F University, Lin'an, 311300 Zhejiang, China

<sup>b</sup> Zhejiang Provincial Key of Biological and Chemical Utilization of Forest Resources, Zhejiang Forestry Academy, Hangzhou, 310023 Zhejiang, China

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### ABSTRACT

Dyeing wastewater removal is important for the water treatment, and adsorption is an efficient treatment process. In this study, three modified bentonites, chitosan modified bentonite (CTS-Bent), hexadecyl trimethyl ammonium bromide (CTAB) modified bentonite (CTAB-Bent), and both chitosan and hexadecyl trimethyl ammonium bromide modified bentonite (CTS–CTAB-Bent) were prepared and characterized by FTIR and XRD analysis. Batch experiments were conducted to evaluate the adsorptive removal of weak acid scarlet from aqueous phase using modified bentonites under different conditions. The results show that the adsorption capacity of weak acid scarlet onto natural bentonite was low (4.9%), but higher for 1CTS-Bent and 1CTS–10CTAB-Bent. The optimal conditions for weak acid scarlet adsorption were 1% chitosan, 10% CTAB, at 80 °C and reaction time 2.5 h. The best removal efficiency was ~85%, and the adsorption capacity of weak acid scarlet was around 102.0 mg g<sup>-1</sup>, much higher than that of commercial activated carbon (27.2 mg g<sup>-1</sup>). These results suggest that 1CTS–10CTAB-Bent is an excellent adsorbent for effective weak acid scarlet removal from water. The adsorption isotherms of weak acid scarlet were investigated. It was found that Langmuir and Temkin models fitted the data very well ( $R^2 > 0.99$ ).

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### 1. Introduction

The dyeing industry is one of the biggest wastewater producing industries. The wastewater is characterized by high organic pollutant content, deep color, and significant impact on water quality. Color is recognized as the first contaminant in wastewater, because a very small amount of dyes in water is highly visible and undesirable. The discharge of dyeing wastewater to rivers and basins pose a potential risk to local resident's health and ecosystem. Therefore, the effective treatment of dyeing wastewater becomes a task of top priority to government. Currently, the dyeing wastewater treatment has been widely investigated, and several technologies have been developed including adsorption, oxidation, super filter film, and coagulation [1,2]. Adsorption using low-cost adsorbents is one of the most economically and viable method for dyeing wastewater decontamination. A large variety of conventional and non-conventional adsorbent materials have been proposed and studied for their ability to remove dyes. However, all of these adsorbents have various drawbacks. Activated carbons have the advantages of high adsorption capacity and selectivity for dyestuff removal but it is difficult to regenerate, which makes its application for dyeing wastewater restricted. It is now recognized that adsorption using low-cost adsorbents, naturally occurring adsorbents is an

effective and economic method for removing contaminants from wastewater. Recently, the use of natural mineral adsorbents for wastewater treatment increasingly attracts attention because of their abundance and low price [3,4]. Naturally, bentonite exhibits strong adsorption capability due to its large surface area and surface energy. However, the presence of surface negative charge and large amount of exchangeable positive ions result in the cover of a layer of water molecule on the mineral surface, which makes natural bentonite exhibit strong hydrophilicity, and therefore, it is not an effective adsorbent for organic pollutants. Chitosan is a type of amino homogeneous state polysaccharide and no risk to environment, which can be bio-synthesized or biodegraded. Because of containing large numbers of –OH and –NH<sub>2</sub> functional groups, it is widely applied in organic wastewater treatment [5–8]. Moreover, the occupation of exchange sites on the bentonite surface by organic cations (such as cationic surfactants, quaternary ammonium surfactants) will change the surface properties from hydrophilic to hydrophobic. Therefore, there has been much interest in the use of modified bentonites as adsorbents to prevent and remediate environmental organic contamination. Previous studies showed that chitosan and organic modified bentonites have been widely used to adsorb heavy metals [9–14], dyes [15–17], organic pollutants such as phenol [18,19], catechol [20], benzoic acids [21,22], and other environmental pollutants [23,24].

The main objective of this study is to evaluate the feasibility of using different modified bentonites to remove weak acid scarlet from dyeing wastewater. Three adsorbents, chitosan modified

\* Corresponding authors. Fax: +86 571 63732772.

E-mail addresses: gjianzhong123@163.com (J. Guo), fyanlong@123.com (Y. Feng).

bentonite (CTS-Bent), hexadecyl trimethyl ammonium bromide modified bentonite (CTAB-Bent), and both chitosan and hexadecyl trimethyl ammonium bromide modified bentonite (CTS-CTAB-Bent) were synthesized. The synthesized modified bentonites were characterized by Fourier Transform Infrared spectroscopy (FTIR) and X-ray diffraction (XRD). The adsorption performance of modified bentonites in weak acid scarlet wastewater was also investigated by batch experiments.

## 2. Experimental

### 2.1. Materials

Sodium bentonite used in this study was purchased from the Zhejiang Huate New material Co., Ltd. (Lin'an, Zhejiang Province, China). Chitosan (Degree of deacetylation >85%) was purchased from Jinan Haidebei Marine Bioengineering Co., Ltd. (Jinan, Shandong Province, China). Hexadecyl trimethyl ammonium bromide (CTAB), Hexadecylpyridinium chloride monohydrate (CPC), HCl, NaOH, Acetic acid were all of analytical grade and obtained from Sinopharm Chemical Reagent Beijing Co., Ltd., China.

### 2.2. Preparation of modified bentonites

The chitosan with a deacetylation degree of 85% was kept at 1.0% throughout the experiment. First, chitosan solution was prepared with 1.0% acetic acid in a three-neck flask with constant stirring and bubbling of nitrogen gas at room temperatures. A predetermined bentonite (5.0 g) was added into 200 mL of deionized water. A certain volume of quaternary ammonium salt solution ( $0.02 \text{ mol L}^{-1}$ ) was added in the above mixture with strongly stirring for 2 h. Then, 1.0% chitosan solution was added step by step under certain temperature. The mixture was stirred for 2 h and then cooled down to the room temperature. Then, the sample was filtered under reduced pressure and washed with deionized water until no bromide ion detected by silver nitrate solution. The filter cake is dried at  $80^\circ\text{C}$  overnight and ground. The modified bentonite was obtained and sieved to 150–200 mesh for use. The prepared modified bentonites were designated as xCTS-yCTAB-Bent, where *x* denotes the loading amount of CTS (wt% of the substrate) and *y* denotes the loading amount of CTAB (wt% of the substrate).

### 2.3. Adsorption experiments

Distilled water was used to prepare dye solutions with the desired concentration. The calibration curve of weak acid scarlet solution was obtained by measuring the absorbance of different predetermined concentrations of the samples at a wavelength of 507 nm using the 722 visible spectrophotometers.

A predetermined amount of dry modified bentonite (0.05 g) was placed in a 250 mL flask, followed by adding 40 mL certain concentration weak acid scarlet solution. The adsorption test was performed in a gas bath constant temperature oscillator under room temperature for certain time, then centrifugal separation at 3000 rpm for 10 min. The weak acid scarlet concentration of the filtrate solution was measured with the 722 visible spectrophotometers. Removal efficiency (%) and adsorption capacity *q* ( $\text{mg g}^{-1}$ ) were calculated according to the formula:

$$R = \frac{c_0 - c_t}{c_0} \times 100\%; \quad q = \frac{(c_0 - c_t) \times V}{m} (\text{mg g}^{-1})$$

where  $c_0$  ( $\text{mg L}^{-1}$ ) is the initial concentration of adsorbate;  $c_t$  ( $\text{mg L}^{-1}$ ) is the concentration of adsorbate by adsorbents; *V* (L) is the volume of adsorbate; *m* (g) is the mass of adsorbents;  $q_t$  ( $\text{mg g}^{-1}$ ) is the adsorbed amount at certain time;  $q_e$  ( $\text{mg g}^{-1}$ ) is the adsorbed amount at equilibrium.

### 2.4. Characterization

FTIR spectra were used to characterize the modified bentonites using a Nicolet 6700 instrument. Typically, 100 scans were collected at  $4 \text{ cm}^{-1}$  spectral resolution in the range of  $4000\text{--}400 \text{ cm}^{-1}$ . X-ray diffraction patterns (XRD) of the prepared samples were performed with X-ray diffractometer in XRD2600 (SHIMADZU, Japan) equipment using nickel-filtered  $\text{Cu K}\alpha$  radiation operated at 40 kV and 30 mA. All XRD patterns were obtained with a scan speed of  $2^\circ/\text{min}$ .

### 2.5. Regeneration studies

For re-use of modified bentonite, desorption and regeneration experiments were performed with NaOH aqueous solution. Desorption experiment was performed by placing used 1CTS-10CTAB-Bent (0.05 g) in 100 mL beaker with 30 mL  $0.25 \text{ mol L}^{-1}$  NaOH under shaking at room temperature on gas bath constant temperature oscillator for 5 h. Then, neutralized with  $0.2 \text{ mol L}^{-1}$  HCl. The regenerated chitosan-CTAB modified bentonite was tested for further adsorption of weak acid scarlet under same adsorption condition. Adsorption and desorption experiments were followed for three cycles.

## 3. Results and discussion

### 3.1. Characterization of bentonites

The FTIR spectra of natural Na-bentonite, 10CTAB-Bent, 1CTS-Bent, and 1CTS-10CTAB-Bent in the range of  $4000\text{--}400 \text{ cm}^{-1}$  were shown in Fig. 1.

The band at  $3650\text{--}3622 \text{ cm}^{-1}$  in the spectra showed H-O-H hydrogen bond of water molecule. The intensive band at around  $1030 \text{ cm}^{-1}$  was assigned to Si-O stretching vibrations. The Si-O-Al and Si-O-Si bending vibrations appeared at 525 and  $462 \text{ cm}^{-1}$ , respectively [21,25]. The small band at  $1634 \text{ cm}^{-1}$  was corresponded to the  $\delta_{\text{SiO-H}}$  deformation vibration. From Fig. 1b and d, it can be seen that the bands at  $2930$  and  $2850 \text{ cm}^{-1}$  were correspond to the  $\text{CH}_2$  asymmetric stretching mode ( $\nu_s(\text{CH}_2)$ ) and the symmetric stretching mode ( $\nu_s(\text{CH}_2)$ ), respectively. The results indicated that the CTAB molecule was impregnated into the inter-layer space of the bentonite. A new band at  $1564 \text{ cm}^{-1}$  was related to the  $\text{NH}_2$  vibration mode of the chitosan (Fig. 1c and d), which

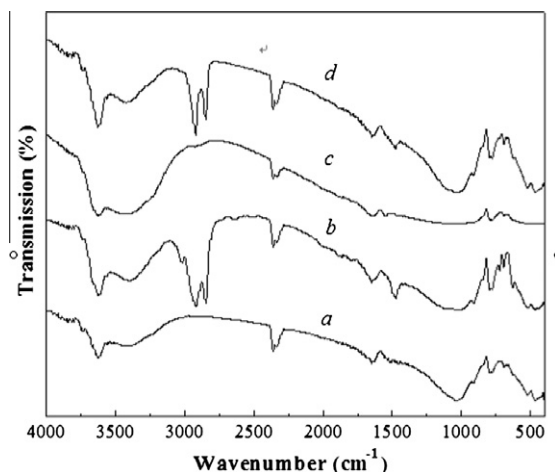


Fig. 1. FTIR spectra of natural bentonite (a), 10CTAB-Bent (b), 1CTS-Bent (c), and 1CTS-10CTAB-Bent (d).

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