



# Activating natural bentonite as a cost-effective adsorbent for removal of Congo-red in wastewater



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## ABSTRACT

The bentonite is a widely available and abundant natural mineral, and can be a low cost adsorbent for water and wastewater treatment. This study reported here was directed towards identifying a cost-effective activation protocol for enhancing the adsorption capacity of Australian bentonite for removal of toxic contaminants in wastewater. We investigated three protocols including thermal activation (TA), acid activation (AA), and combined acid and thermal activation (ATA). The results showed that these activation protocols under designed conditions can enhance the surface area and porosity of the raw bentonite. The best ATA protocol considered here brought a 70% increase in the surface area compared to 65% and 20% for the best AA and TA protocols, respectively. The optimal ATA protocol identified in the study leads to approximately 25% increase in the Congo-red adsorption capacity of the activated bentonite. This activation method could be a cost-effective approach to enhance the adsorption capacity, applicability and selectivity of natural clay materials, making them as promising and low cost adsorbents for wastewater treatment.

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## 1. Introduction

Clays are finding growing application in wastewater treatment as adsorbents due to their wide availability, low-cost, and good intrinsic adsorption characteristics. There are a number of natural clays that are used for the removal of chemical pollutants from wastewater, including kaolin, bentonite and zeolite [1–6]. Among these clays, bentonite is one of the most widely used adsorbents as it possesses a net negative surface charge, making it effective for removal of cationic compounds [6]. However, the adsorption capacity and selectivity of the natural bentonite towards anionic compounds appear to be less promising. Thus, a systematic surface modification of the bentonite is, however, necessary to enhance its adsorption capacity and possibly to improve its adsorption selectivity for the removal of anionic compounds from wastewater [7].

The chemical compositions which determine properties of the clays i.e. layer charge, cation exchange capacity, adsorption capacity and morphology, can be varied depending upon their

origins. These factors play a significant role in the modification of the natural clays. A number of methods for modifying clay minerals have been studied with a view to enhancing their adsorption capacities [8,9], including acid activation [10], treatment with cationic surfactants [11], clay-rubber composite, thermal activation [12], polymer addition by interparticle polymerization [13], binding of inorganic and organic anions, and grafting of organic compounds [14]. Among these, acid activation is one of the most commonly used modification techniques because it is a simple and low-cost process [12,15].

Dyes are used extensively in industry, including in the manufacture of like textile, cosmetics, pulp and paper, paints, pharmaceuticals, and carpet, as well as in printing [3]. Many dyes are toxic and biologically non-degradable due to complex chemical structure and the presence of aromatic ring in their structure [16]. Azo dyes, for example, contain one or more azo groups with aromatic ring and sulfonate group, and can be transformed into more hazardous substances under anaerobic conditions [17]. They can cause allergy, dermatitis, skin irritation and mutation in human bodies [5]. These dyes at even 0.005 ppm in water are highly visible and can reduce light energy transfer efficiency in a photosynthesis system [18]. The removal of the dyes from wastewater before discharging it into the mainstream is, therefore, essential.

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The adsorption of Congo red (CR) using three Australian clay minerals namely; sodium bentonite, kaolin and zeolite has been studied in our group [2,6,12]. We found that sodium bentonite was the most effective adsorption material for CR removal. The study reported here was aimed at identifying a technically and economically feasible protocol for the activation of raw Australian bentonite that would lead to a promising enhancement in its adsorption capacity to remove CR from industrial wastewater. The study focused on three commonly used methods: thermal activation (TA), acid activation (AA) and a combination of acid and thermal activation (ATA). Physical and chemical characteristics of the activated bentonites were evaluated by analyzing the surface area and pore size, and were characterized by scanning electron microscopy (SEM), Fourier transform infrared spectroscopy (FTIR), and X-ray diffraction (XRD).

## 2. Methods and materials

### 2.1. Materials

Australian sodium bentonite (Active Gel 150) obtained from Unimin Australia Limited contains high montmorillonite and low grit [2,12]. Congo red (obtained from Labchem Ajax Finechem Australia), an anionic dye, is widely used as a surrogate indicator to simulate industrial wastewater when testing for adsorption capacity of porous materials in wastewater. Chemical composition and characteristics of the bentonite and CR were described in detail previously [6,12].

### 2.2. Activation of raw bentonite

#### 2.2.1. Thermal activation

The thermal activation was performed by heating 10 g of the bentonite to the desired temperature in a range of 50–500 °C in a muffle furnace. The temperature was allowed to rise steadily to the desired temperature in 5 min. The samples were maintained at the desired temperature and were heated for predetermined time varying from 10 to 120 min. The heated samples were then cooled and stored in a desiccator.

#### 2.2.2. Acid activation

The acid activation was carried out in a 250 mL breaker with 100 mL working volume. The samples were mixed properly in a rotary shaker (Ratek OM 15 orbital) at 100 rpm for 10 min. The raw bentonite was treated with HCl at a determined concentration of 0.05–0.5 M at 60–100 °C. The acid to clay ratio was 1:10 (mL/g). The acid activation was terminated with addition of large amount of double-ionized water. The acidified bentonite was then washed several times until Cl<sup>-</sup> ion was undetectable in the supernatant using 0.1 M silver acetate solution. The final sample was centrifuged at 3000 rpm and was dried at 55 °C for 12 h and then stored in a desiccator.

#### 2.2.3. Combined acid and thermal activation

The combined acid and thermal activation of the bentonite was conducted by a two-step procedure. The bentonite was first activated by HCl over a concentration range from 0.05 to 0.5 M, as described above. The acidified bentonite was then subjected to thermal activation at a low temperature range of 50–150 °C for 20 min in a muffle furnace. The samples were cooled in a desiccator.

### 2.3. Characterization of bentonite

#### 2.3.1. Surface area and pore size

The specific surface area and average pore size of the bentonite samples were analyzed using, respectively,

Brunauer–Emmett–Teller (BET) Surface Area Analysis and Barrett–Joyner–Halenda (BJH) Pore Size Analyser. BET specific surface area and pore size measurements were performed using a Micromeritics gas adsorption analyser (Gemini Type 2375) at 77 ± 0.5 K in liquid nitrogen. Prior to the surface analysis, the sample vessels loaded with ca. 0.5–1.0 g were vacuum treated 12 h at 105 °C and evacuation pressure of 50 mTorr. Nitrogen adsorption isotherms of the samples were then analysed for the specific surface area using the BET equation.

#### 2.3.2. Fourier transform infra red spectra

FTIR spectroscopy was used to understand the effect of the acidification on the surface chemistry of the modified bentonite. The FTIR spectra were recorded in 4000–400 cm<sup>-1</sup> using Nicolet 6700 FT-IR with Smart orbit Attenuated Total Reflectance (ATR) accessory. The ATR was fitted with Diamond crystal on powders that can be analyzed without the formation of a pellet with potassium bromide required with other standard FT-IR approaches. The infra-red spectra of the raw and modified bentonites were obtained in the powder form by placing the samples on the Diamond crystal to obtain FTIR spectra.

#### 2.3.3. Scanning electron microscopy

Morphological characteristics of the bentonite were examined using a Philips XL30 SEM at accelerating voltage of 15 kV, beam size 3.0, working distance 10 and magnification of 8000. The samples were coated with carbon under vacuum before analysis to prevent the accumulation of static electric charge on the surface of the bentonite particles. Several microscope images were taken to compare surface properties of the raw and modified bentonite samples.

### 2.4. Experimental design and procedure

#### 2.4.1. Adsorption performance

Adsorption of CR on bentonites was carried out in a batch system. Initial CR concentration in the experiments was set at 100 mg L<sup>-1</sup>, otherwise as stated in the text. The bentonite–CR suspensions in the flasks were agitated in a rotary shaker (Ratek OM 15 orbital) at 150 rpm and 30 °C for 24 h to ensure that equilibrium was reached. The samples were then centrifuged in Eppendorf Centrifuge 5415R (Germany) at 3200 rpm for 20 min to separate the CR solution from the adsorbent. All sampling and tests were conducted in triplicates. Data were calculated from the average of the triplicates.

#### 2.4.2. Analysis and calculation of adsorption capacity

The CR concentration was determined by UV–visible spectrophotometry (model  $\gamma$ , Helios, UK) at 496.5 nm [6]. The absorbance concentration profile was obtained by plotting the calibration curve between the absorbance and CR concentration. The absorbance for each sample was converted using the calibration factor obtained from the calibration curve to calculate the final dye concentration. The amount of dye adsorbed on the surface of adsorbent at time  $t$  can be calculated from the mass balance equation:

$$q_e = (C_i - C_e) \frac{V}{m}$$

where  $q_e$  is the amount of dye adsorbed per unit mass of adsorbent (mg g<sup>-1</sup>),  $C_i$  is the initial dye concentration (mg L<sup>-1</sup>),  $C_e$  is the equilibrium dye concentration (mg L<sup>-1</sup>),  $V$  is the volume of dye solution (mL) and  $m$  is the mass of bentonite (g).

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