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# Activated carbon coated palygorskite as adsorbent by activation and its adsorption for methylene blue

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## ABSTRACT

An activation process for developing the surface and porous structure of palygorskite/carbon (PG/C) nanocomposite using  $ZnCl_2$  as activating agent was investigated. The obtained activated PG/C was characterized by X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), field-emission scanning electron microscopy (SEM), and Brunauer–Emmett–Teller analysis (BET) techniques. The effects of activation conditions were examined, including activation temperature and impregnation ratio. With increased temperature and impregnation ratio, the collapse of the palygorskite crystal structure was found to accelerate and the carbon coated on the surface underwent further carbonization. XRD and SEM data confirmed that the palygorskite structure was destroyed and the carbon structure was developed during activation. The presence of the characteristic absorption peaks of C=C and C–H vibrations in the FTIR spectra suggested the occurrence of aromatization. The BET surface area improved by more than 11-fold ( $1201 \text{ m}^2/\text{g}$  for activated PG/C vs.  $106 \text{ m}^2/\text{g}$  for PG/C) after activation, and the material appeared to be mainly microporous. The maximum adsorption capacity of methylene blue onto the activated PG/C reached  $351 \text{ mg/g}$ . The activated PG/C demonstrated better compressive strength than activated carbon without palygorskite clay.

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## Introduction

Dyes are widely used in the chemical industry, and ever-increasing industrial applications of organic dye compounds have resulted in significant pollution of effluent waters and other environmental hazards (Benadjemia et al., 2011). These dyes are also harmful to humans with prolonged contact. Methylene blue (MB), a common pollutant in dye effluents, is difficult to remove completely because of its stable aromatic structure consisting of a chromophore and polar groups (Liu et al., 2012). Various techniques have been developed to improve the efficiency of MB removal, including adsorption, chemical oxidation, and electro-coagulation. Among these, adsorption

has proven particularly effective, mainly because of its simplicity and efficiency.

Activated carbon works as an excellent adsorbent for the removal of most organic contaminants because of its high surface area and wide porosity. Activated carbon can be produced by physical or chemical activation using various carbonaceous materials (Mohanty et al., 2005; Juang et al., 2000; Theydan and Ahmed, 2012; Zhu et al., 2007; Khezami et al., 2005; Yue et al., 2013). Compared to physical activation, chemical activation can confer particular surface and pore characteristics to activated carbon through the use of various biomass materials as precursors, including lignin, peat, and leaves (Hayashi et al., 2000; Chiu and Ng, 2012; Donald et al.,

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2011; Saka, 2012; Altener et al., 2009). Furthermore, development of the pore structures can be easily adjusted by changing activation conditions such as temperature, impregnation ratio, and activating agents (Williams and Reed, 2006; Uçar et al., 2009; Nakagawa et al., 2007; Yavuz et al., 2010). Reffas et al. (2010) prepared activated carbon with a total surface area of 925 m<sup>2</sup>/g from coffee grounds using H<sub>3</sub>PO<sub>4</sub> as the activating agent. Chiu and Ng (2012) synthesized activated carbon fiber with Brunauer–Emmett–Teller (BET) surface area of 2060 m<sup>2</sup>/g from cotton using ZnCl<sub>2</sub>. The demand for activated carbon is growing rapidly because of increasing industrial pollution; however, high price has limited its application.

Palygorskite (PG) is a type of magnesium aluminum silicate mineral that exists widely in nature. In our previous study, a novel palygorskite/carbon (PG/C) sorbent was prepared by hydrothermal carbonization of glucose or cellulose using palygorskite as template (Wu et al., 2011, 2014a,b). Given the combination of the crystal structure of PG and the organophilic nature of carbon, PG/C has high adsorption capacity for some typical organic pollutants, such as MB and phenol. PG/C offers advantages such as its inexpensive raw materials, low cost, and green production. However, its specific surface area is rather low compared to that of activated carbon.

This study focused on the activation process of PG/C nanocomposite and aimed to produce activated samples with high surface area and porosity. Previous studies have showed that porous carbon with more stable performance can be obtained by hydrothermal methods (Hao et al., 2013; Wang et al., 2011; Regmi et al., 2012; Ding et al., 2013; Román et al., 2013). Román et al. (2013) prepared adsorbents from biomass by combining hydrothermal carbonization and activation. The hydrothermal carbons produced in this manner presented low BET surface areas of only about 30 m<sup>2</sup>/g, which increased up to 438 m<sup>2</sup>/g after CO<sub>2</sub> activation. It was reported that the initial hydrothermal treatment step allowed better control over the resulting porosity and that the activation step was crucial in the development of porosity in hydrothermal carbons. Furthermore, this method offered the ability to change surface functionalities and improved the final yield of the subsequent activation process. In the current study, the previously studied PG/C nanocomposite was further activated to develop a carbon pore structure coated onto PG. The influences of ZnCl<sub>2</sub> impregnation ratio and activation temperature on surface characteristics were then investigated. In addition, adsorption of MB onto the activated samples prepared under different conditions was compared.

## 1. Materials and methods

### 1.1. Raw materials

PG raw material was acquired from Jiangsu Province, China and sieved to 200 mesh. The chemical composition and texture properties were determined by chemical analysis and X-ray diffraction. The chemical composition of the clay was 57.01% SiO<sub>2</sub>, 8.78% Al<sub>2</sub>O<sub>3</sub>, 12.82% MgO, 4.28% Fe<sub>2</sub>O<sub>3</sub>, 0.08% FeO, and 17.03% others. All reagents (cellulose, ammonium iron sulfate hexahydrate (FeSO<sub>4</sub>(NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>·6H<sub>2</sub>O), hydrochloric acid (HCl), hydrofluoric acid (HF), anhydrous zinc chloride

(ZnCl<sub>2</sub>), and methylene blue (MB)) were of analytical grade and were bought from Sinopharm Group Co., Ltd (Shanghai, China) and used as received without further purification. Commercial activated carbon was obtained from the Institute of Coal Chemistry, Chinese Academy of Sciences. Only double-distilled water was used.

### 1.2. Preparation of palygorskite/carbon (PG/C) nanocomposite

The PG/C sample was prepared based on a previously reported method (Wu et al., 2011, 2014a,b). Cellulose (5 g) and FeSO<sub>4</sub>(NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>·6H<sub>2</sub>O (2.80 g) were dissolved in 72 mL double-distilled water. PG (2.5 g) was then added and the mixture was stirred for 2 hr at room temperature. The final suspension was transferred to a Teflon-lined stainless-steel autoclave (100 mL in total inner volume), sealed, and maintained at 220°C for 12 hr. The black product was centrifuged, washed with double-distilled water and ethanol several times, and then dried in an oven at 105°C for 8 hr.

### 1.3. Activation

The dried PG/C nanocomposites were passed through a 200-mesh sieve prior to chemical activation with ZnCl<sub>2</sub>. Appropriate amounts of PG/C powder and anhydrous ZnCl<sub>2</sub> were dispersed in water to obtain the desired impregnation mass ratio ZnCl<sub>2</sub>:PG/C composites. To remove moisture after impregnation, the samples were dried for 24 hr at 105°C in an oven and then heated for another hour at various temperatures under a controlled heating rate of 5°C/min and nitrogen atmosphere. To remove any residual organic and mineral matter, the sintered samples were washed with 1.2 mol/L HCl solution and then rinsed with hot distilled water until the pH value reached 7. Lastly, the samples were dried at 105°C.

The activation temperature was set at 450°C for experiments investigating the effect of varying impregnation ratios, whereas, the impregnation ratio was set at 1:1 for the experiments investigating the effect of varying activation temperatures. The samples were labeled as PG/C-Ax-y/z, where x stands for activation temperature, and y/z for impregnation weight ratio of ZnCl<sub>2</sub> and PG/C composites. The PG clay in the activated PG/C was removed by washing the composite in 40 wt.% HF solution at room temperature for 20 hr followed by several rounds of washing with double-distilled water and, lastly, drying at 105°C.

### 1.4. Characterization

Power X-ray diffraction analysis (XRD) was performed using an X-ray diffractometer with Cu anode operating at 40 kV and 100 mA (D/MAX2500V, Rigaku, Japan). Elemental analysis (EA) was conducted on an elemental analyzer (Vario EL c, Elementar, Germany). Thermal analysis (DSC-TG) curves of PG were recorded with a simultaneous thermal analyzer (Q2000, TA, USA). The sample was heated from room temperature to 700°C at 10°C/min under N<sub>2</sub> flow. Fourier transform infrared spectra of the samples were obtained in the range of 4000–400 cm<sup>-1</sup> in a FTIR spectrometer (Thermo, Madison, USA) using KBr pellets. The morphology and particle size of the products were observed with a field-emission scanning electron microscope (SEM, JSM-6700, JEOL, Japan). Brunauer–Emmett–Teller (BET) analysis

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