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Tubular activated carbons made from cotton stalk for dynamic adsorption of airborne toluene

Lijuan Hu^a, Ying Peng^a, Feng Wu^a, Shanzhi Peng^a, Jinjun Li^{a,*}, Zizheng Liu^{b,**}^aSchool of Resource and Environmental Science, Hubei Key Laboratory of Biomass-Resources, Wuhan University, Wuhan 430079, China^bSchool of Civil Engineering, Wuhan University, Wuhan 430072, China

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ABSTRACT

Monolithic tubular activated carbons were prepared from cotton stalks by chemical activation using phosphorous acid and zinc chloride and referred to as AC-P and AC-Z, respectively. These tubular carbons can be stacked into a honeycomb-like configuration, which has potential in the emission control of volatile organic compounds. Dynamic adsorption of toluene on the tubular activated carbons was tested. AC-P had a specific surface area of 1256 m²/g, 73.4% of which was contributed by mesopores; while AC-Z had a specific surface area of 795 m²/g, 60% of which was contributed by micropores. FTIR spectra revealed that AC-Z possessed less oxygen-containing polar groups than AC-P, which contributed to its higher hydrophobicity. TG analysis also suggested that less water was adsorbed on AC-Z. At a flow rate of 150 mL/min and toluene concentration of 1000 ppm, the dynamic toluene adsorption capacity of AC-P was as high as 328 mg/g under dry conditions owing to its high specific surface area; however, it decreased to only 94 mg/g under wet conditions (80% relative humidity, 25 °C) due to its hydrophilicity. In contrast, AC-Z having high hydrophobicity showed a dynamic toluene adsorption capacity of 258 mg/g under dry conditions, which remained at 221 mg/g under wet conditions.

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1. Introduction

Some volatile organic compounds (VOCs) are detrimental for both human health and the environment [1]. They play important roles in the formation of photochemical smog and toxic byproducts and may lead to the destruction of the ozone layer as well as global warming [2,3]. In recent years, regulations have been established in many countries to limit VOC emissions. Therefore, VOC abatement technologies have aroused much more attention than before. Adsorption is one of the most widely used technologies and versatile activated carbons were proved to be effective adsorbents for VOC removal [4–6], due to their developed microporous structures, high surface areas, favorable pore sizes and good chemical stability. When dealing with industrial gaseous organic emissions, traditional adsorption beds composed of granular active carbons have significant drawbacks, such as high pressure drops in the adsorbent bed, particle entrainment, and channeling [7–9]. These can be overcome by using monolithic honeycomb-like activated carbons, which are often produced by extruding powder active carbons along with binders and fillers [10–12]. On the other hand,

the extruded honeycomb-like activated carbons are often limited in mechanical strength [13], and the binders and fillers, such as silicate, alumina and titania, could also cause decrease in thermal conductivities. Poor thermal conductivity may lead to local accumulation of adsorption heat, increasing the risk of VOC ignitions and accidental fires [14].

Agricultural and forestry residues have been given special emphasis as raw materials for the preparation of activated carbon because they are renewable, highly available, and their utilization reduces the potential environmental pollution [15]. Many agricultural and forestry wastes, such as wood [16–18], lotus stalks [19,20], acorn shell [21], sugarcane bagasse [22,23], spent coffee grounds [24], and tea waste [25,26], have been used as raw materials for the preparation of activated carbons. Generally, biomass-derived activated carbons are in the form of particles or powders. Recently we prepared monolithic activated carbons having a tubular structure that resembles bamboo stems using shrubby bamboos as raw material [13]. The small tubes have high mechanical stability, inherited from bamboo. These carbon tubes can be stacked into a honeycomb-like form that has potential in the treatment of waste streams.

In this work, we aimed to synthesize monolithic activated carbons using cotton stalks as raw material for VOC adsorption. Cotton is widely planted and most of its biomass is residue that, if not properly handled, could cause environmental problems. For

* Corresponding authors.

** Corresponding author at: School of Civil Engineering, Wuhan University, Wuhan 430072, China.

E-mail addresses: lijinjun@whu.edu.cn (J. Li), lzz2015@whu.edu.cn (Z. Liu).<http://dx.doi.org/10.1016/j.jtice.2017.07.029>

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example, burning these solid wastes leads to serious air pollution [27,28]. It would therefore be meaningful to transform cotton stalks to value-added products. Most of the cotton stems and branches have diameters similar to shrubby bamboo (*ca.* 1 cm), and carbonization could lead to their diameter shrinking to the mm scale. We found that such carbonized products could also be made into tubular structures to be used in air decontamination.

2. Materials and methods

2.1. Chemicals and raw materials

Phosphoric acid (H_3PO_4 , 85%), zinc chloride (ZnCl_2), hydrochloric acid (HCl, 37%) and toluene were purchased from Sinopharm Chemical Reagent Company Limited (Shanghai, China). All the chemicals were of analytical grade and used as received. Cotton stalk was collected from Chibi, Hubei province. The proximate analysis of the cotton stalk were made by ASTM standard test methods [18,29], the chemical composition was measured by the National Renewable Energy Laboratory method [30], and the data are shown in Table S1. Before use, the cotton stalks were dried at 120 °C for 24 h, stems with diameters of *ca.* 0.6 cm were selected, peeled, and sawed to sections about 5 cm in length before use.

2.2. Preparation of tubular activated carbons

Cotton stems were activated using H_3PO_4 and ZnCl_2 , obtaining products referred to AC-P and AC-Z, respectively. The preparation procedures were similar to that reported recently [13].

AC-P: a typical cotton section (*ca.* 5 cm in length and *ca.* 1.2 g in weight) was immersed in 20 mL of 80% H_3PO_4 solution for 12 h, dried in an oven at 120 °C, and then placed in a quartz tube reactor in an electric tubular furnace. The quartz reactor was purged with high-purity nitrogen at a flow rate of 60 mL/min and heated at 200 °C for 40 min and then at 400 °C for 1 h at a heating rate of 10 °C/min. The activated sample was repeatedly washed with boiling distilled water until a neutral pH of the washings was achieved, and then dried at 120 °C overnight. The heart-center of the stem was loose and soft, and it was easily removed after activation, obtaining a tubular product denoted as AC-P.

AC-Z: a typical cotton section (*ca.* 5 cm in length and *ca.* 1.2 g in weight) was immersed in 20 mL of 60% ZnCl_2 solution for 12 h, dried at 120 °C, and then treated at 600 °C for 1 h at a heating rate of 10 °C/min in a 60 mL/min nitrogen flow. The activated sample was immersed in 50 mL of 2 M HCl for 5 h, and then washed repeatedly with copious amounts of boiling distilled water until a neutral pH of the washings was achieved, and then dried at 120 °C overnight. The heart-center was then eliminated, obtaining a tubular product denoted as AC-Z.

2.3. Characterization of the prepared activated carbons

The nitrogen adsorption-desorption isotherms were obtained on V-sorb 2800P porosity analyzer (Gold APP, China). The specific surface area was obtained through the Brunauer-Emmett-Teller (BET) method. Micropore volume and surface area were estimated by the t-plot method. The micropore size distribution was calculated by Horvath-Kawazoe analysis.

The morphology observations of the activated carbons were carried out on a QUANTA200 scanning electron microscope (FEI, Netherlands). Images were obtained at an excitation energy of 20 kV.

The Fourier transform infrared spectrums in the range 4000–400 cm^{-1} were recorded using a Nicolet 5700 FTIR spectrometer (Thermo, USA) at room temperature.

Thermogravimetric (TG) analyses were conducted on a TG-DTA 6300 thermal analyzer (Seiko, Japan). The samples were heated at 10 °C/min from room temperature to 800 °C under an air flow of 30 mL/min.

2.4. Dynamic adsorption of toluene

Dynamic toluene adsorption onto AC-P and AC-Z was tested in a continuous-flow reactor, and a schematic diagram of the setup for dynamic adsorption test was shown in one of our previous work [13]. Tubular carbons (weight of 300 mg, stacked length of *ca.* 8 cm) were treated under vacuum at 180 °C overnight, and then transferred to a U-shaped glass tubular reactor with an inner diameter of 5 mm (Fig. S1), which was placed in a thermostatic water bath at 25 °C. A 150 or 100 mL/min of air flow containing 1000 ppm of toluene vapor was passed through the reactor. The toluene vapor was generated using a method reported in literature [13], which involved rate-controlled injection of liquid toluene into a preheated air flow (140 °C) by a TJ-1A micro-injection pump (Baoding Longer Precision Pump Company Limited, China), producing a concentration-stable toluene vapor. The inlet toluene concentration (C_0) and the outlet one (C) were monitored on an online GC7806 gas chromatograph (Wenling Instrument, China) equipped with a six-way injection valve and a hydrogen flame ionization detector. In order to evaluate the dynamic adsorption performances under wet conditions (with a relative humidity of 80%), water vapor was generated by continuously injecting liquid water into a preheated air flow using a micro-injection pump [13]. The dynamic adsorption capacity (Q) of the adsorbent was calculated from the breakthrough curves according to Eq. (1) [31–33]:

$$Q = \frac{F \times t_Q}{W} \quad (1)$$

The time t_Q was estimated according to Eq. (2):

$$t_Q = \int \left(1 - \frac{C}{C_0}\right) dt - t_D \quad (2)$$

where F was the mass flow of adsorbate vapor (mg/min), W was the amount of the adsorbent loaded in the reactor (g), C and C_0 were the outlet and inlet adsorbate concentration (ppm), respectively, and t_D was the dead time of the system (min), which represented the time required by the adsorbate to travel from the vapor generator to the detector in the absence of adsorbent.

2.5. Temperature programmed desorption of toluene

The temperature programmed desorption of toluene was tested on a self-built set-up, which consisted of a U-shaped quartz reactor placed inside an electrical furnace and connected to a thermal conductivity detector (TCD). The activated carbon placed in the reactor was firstly pretreated at 300 °C in a nitrogen flow of 50 mL/min. After being cooled to room temperature, the gas flow was switched to 50 mL/min of nitrogen containing 5000 ppm toluene, and maintained 2 h to achieve toluene adsorption onto the activated carbons. The gas flow was then switched to 50 mL/min of nitrogen and the temperatures were raised to 400 °C at a heating rate of 10 °C/min to desorb the adsorbed toluene. The desorbed toluene in nitrogen was continuously monitored by the TCD, and the signals were recorded during the temperature rising from 50 to 400 °C.

3. Results and discussion

3.1. Characterization of the adsorbents

The photos of the carbon-based materials and the stacked configurations are shown in Fig. 1. After activation, the diameters of

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