



Effects of steam activation on the pore structure and surface chemistry of activated carbon derived from bamboo waste



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ABSTRACT

The effects of steam activation on the pore structure evolution and surface chemistry of activated carbon (AC) obtained from bamboo waste were investigated. Nitrogen adsorption–desorption isotherms revealed that higher steam activation temperatures and/or times promoted the creation of new micropores and widened the existing micropores, consequently decreasing the surface area and total pore volume. Optimum conditions included an activation temperature of 850 °C, activation time of 120 min, and steam flush generated from deionized water of 0.2 cm³ min⁻¹. Under these conditions, AC with a BET surface area of 1210 m² g⁻¹ and total pore volume of 0.542 cm³ g⁻¹ was obtained. Changes in surface chemistry were determined through Boehm titration, pH measurement, Fourier transform infrared spectroscopy (FTIR), and X-ray photoelectron spectroscopy (XPS). Results revealed the presence of a large number of basic groups on the surface of the pyrolyzed char and AC. Steam activation did not affect the species of oxygen-containing groups but changed the contents of these species when compared with pyrolyzed char. Scanning electron microscopy was used to observe the surface morphology of the products. AC obtained under optimum conditions showed a monolayer adsorption capacity of 330 mg g⁻¹ for methylene blue (MB), which demonstrates its excellent potential for MB adsorption applications.

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1. Introduction

Adsorption is a superior approach for removing heavy metals, organic matter, and dyes from wastewaters [1,2]. Activated carbons (ACs) are well-known adsorbents with excellent adsorption abilities for various pollutants. There is a general agreement that microporous ACs are favorable in small molecule adsorption, while mesoporous ACs are very suitable in large molecule adsorption [3]. Suitable textural properties, including a large surface area and proper pore size distribution, are also required for ACs to perform well in a particular application [4]. Besides, the surface chemistry of ACs, especially their surface oxygen-containing groups, has been proven to have significant impacts on the selectivity of ACs serving as adsorbents [5]. Therefore, ACs with identical textural properties but produced from different methods may show very different adsorption capacities because of differences in their surface chemistry [6]. The unique adsorption capacity of ACs is influenced by their textural characteristics and surface chemistry. As such,

investigations of the textural characteristics and surface chemistry of ACs has attracted considerable attention.

Organic wastes, such as palm shell, coconut shell [7], grape seed [8], rice straw [2] and corn cob [9], are often used to produce ACs because the use of these wastes reduces production costs and contributes to disposal problems. Bamboo, a tropical plant, is widely distributed in China and has been largely used as a construction material for scaffoldings. Conversion of bamboo scaffolding waste into ACs has attracted tremendous research attention [10].

Several research works have recently been published on producing bamboo-based ACs. Hameed et al. [11], for example, prepared AC from bamboo by physicochemical activation using potassium hydroxide and carbon dioxide as activating agents. González et al. [12,13] used bamboo to prepare ACs by steam activation and potassium hydroxide activation. Liu et al. [14] prepared bamboo-based ACs by microwave-induced phosphoric acid activation. Ip et al. [15] produced large-surface area bamboo-derived AC by phosphoric acid activation. Mui et al. [16] prepared a series of bamboo-based ACs using HCl, HNO₃, and H₂SO₄ as activating agents. Physical activation, especially steam activation, conforms to the need for cleaner production, and the product can be widely used in many fields, such as in pharmaceutical and food industries. Therefore, in production, physical activation is more favorable than chemical activation.

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However, only one report used steam as an activating agent among the works described in this study, and no report has yet been published on the influence of activation parameters on the evolution of textural characteristics of bamboo-based AC produced by steam activation. ACs prepared from various carbonaceous precursors via steam activation has been investigated by many researchers, however, when the effect of steam on the evolution of porosity is analyzed, some of the results appear to be contradictory. Guillermo et al. [17] have reported that steam activation can produce ACs with narrower and more extensive micropores than CO₂ activation, whereas Eomán et al. [18] have concluded that the carbons activated by steam present wider pore size distribution and more obvious macropores. In order to produce bamboo-based AC with desired pore size via steam activation, the knowledge on the evolution of porosity under different processing parameters is of great importance. X-ray photoelectron spectroscopy (XPS) is widely known as a surface-sensitive quantitative spectroscopic technique that can measure the elemental composition and chemical states of elements present in a material. However, no XPS information on AC derived from bamboo by steam activation can be found in the literature.

In view of the above facts, the main purpose of the present study is to bring plain understanding to the evolution of surface area and porosity of AC produced from bamboo waste with varying steam activation parameters as well as changes in surface chemistry from the bamboo precursor to the pyrolyzed char and to AC systematically. Textural properties were explored by N₂ adsorption–desorption. Surface chemistry was analyzed using Boehm titration, pH measurement, Fourier-transform infrared spectroscopy (FTIR), and XPS analyses. In addition, the surface morphologies of the materials were analyzed using scanning electron microscopy (SEM). The cationic dye methylene blue (MB) was chosen as a model compound to evaluate the adsorption capacity of the AC prepared under optimum conditions.

2. Material and methods

2.1. Materials

Waste bamboo materials from a construction site in Xiamen were used to prepare ACs. The materials were dried in an air oven at 50 °C for 8 h to remove most of the moisture and then chipped to small pieces of about 20 mm × 10 mm × 5 mm. The average proximate analyses of the bamboo precursor were measured as 71.7, 26.6 and 1.7% for volatile matters, fixed carbon and ash on dry basis, respectively. The average ultimate analyses were 47.1, 6.5, 45.8 and 0.6% for C, H, O and N contents, respectively.

2.2. Preparation of activated carbon

This work adopts the established two-step method for preparing AC. Both pyrolysis and activation were carried out in a quartz tube reactor (450 mm height × 50 mm inner diameter) placed in a vertical tube furnace.

For pyrolysis, about 100 g of bamboo chips was placed on the air distributor of the reactor. The furnace temperature was then increased and held at 450 °C for 60 min. About 15 g of the resulting char was then activated at 550–850 °C for 60–150 min under a steam flush of 0.2 cm³ min⁻¹ generated from deionized water in a heated tube. The activating molar flow rate per unit weight of char was calculated to be 0.045 mol g⁻¹ h⁻¹. The same heating rate (i.e., 5 °C min⁻¹) was used in the pyrolysis and activation process. Nitrogen, as the protecting gas, was flushed into the reactor at a rate of 500 mL min⁻¹ throughout pyrolysis and activation. The resulting ACs were stored in desiccators for further analysis and

characterization. The percent yields after pyrolysis or activation were obtained by determining weight differences. Activation burn-off was calculated as:

$$\text{Burn-off}(\%) = \frac{w_c - w_a}{w_c} \quad (1)$$

Where w_c and w_a are the masses of carbons before and after activation, respectively, on a dry-ash-free basis.

The nomenclature used for pyrolyzed char and ACs obtained in this study are as follows: C denotes char, followed by the pyrolysis temperature and time, i.e., C450-60 refers to char pyrolyzed at 450 °C for 60 min. Similarly, AC is followed by the activation temperature and time, i.e., AC850-60 refers to AC activated at 850 °C for 60 min.

2.3. Characterization of materials

Ultimate analysis of carbon, hydrogen, and nitrogen in the materials was performed using a Vario MAX elemental analyzer. The oxygen content was obtained by difference of carbon, hydrogen and nitrogen.

Textural properties were determined using N₂ adsorption–desorption isotherms at –196 °C with a Micromeritic ASAP 2020. Prior to measurement, the sample was degassed under vacuum at 350 °C for 4 h. The surface area was measured using the Brunauer–Emmet–Teller (BET) equation obtained from the nitrogen adsorption data. Total pore volume (V_{tot}) was calculated from the nitrogen adsorbed at a relative pressure of 0.99. The micropore volume (V_{mic}) and pore size distribution were calculated by nonlocal density functional theory (NLDFT) model for carbon slit-shaped pores from the measured isotherms. The median pore width of slit-shaped micropores (D_p) was estimated by the Horvath–Kawazoe (HK) method [19].

Boehm titration was performed to determine total acidic and basic groups. Total acidic and basic groups were neutralized with solutions of 0.05 mol L⁻¹ NaOH and 0.1 mol L⁻¹ HCl, respectively. pH was measured according to the Chinese national standards GB/T 12496.7–1999.

FTIR spectra of the samples were obtained using a NicoletTM iSTM10 FTIR spectrometer. Oven-dried samples were mixed with potassium bromide in an agate mortar, and the resulting mixtures were pressed into pellets for analysis. The spectra were recorded from 4000 cm⁻¹ to 400 cm⁻¹, and 16 scans were taken with a resolution of 4 cm⁻¹.

XPS analysis was carried out at normal emission using a Kratos AXIS ULTRADLD spectrometer equipped with a monochromatic Al K α x-ray source (1486.6 eV). The pass energy was set to 160 eV for the survey spectrum and 20 eV for the high-resolution spectrum. The base pressure of the analysis chamber was about 2×10^{-9} torr. The binding energy was calibrated at 284.8 eV for the C1s level. The energy steps of the survey and high-resolution spectra were 1 and 0.01 eV, respectively. All samples were analyzed under identical conditions.

Micromorphological characteristics of the materials were determined by SEM using a Hitachi S-4800.

2.4. MB adsorption

Around 0.1 g of AC was added to a series of 25 mL of MB solution in 100 mL Erlenmeyer flasks with initial concentrations ranging from 150 mg L⁻¹ to 1500 mg L⁻¹. The MB solutions were prepared without pH adjustment. The flasks were shaken at 25 °C for 24 h until equilibrium was achieved. The concentration of MB remaining in solution was measured using a UV-1100 spectrophotometer at 665 nm. The equilibrium adsorption quantity (q_e [mg g⁻¹]) was calculated as follows: $(2)q_e = \frac{(C_0 - C_e)V}{M}$

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