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Surface texture, chemistry and adsorption properties of acid blue 9 of hemp (*Cannabis sativa* L.) bast-based activated carbon fibers prepared by phosphoric acid activation

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ABSTRACT

Hemp (*Cannabis sativa* L.) bast was used to prepare activated carbon fibers by phosphoric acid activation at 400–600 °C. The pyrolysis process, textural and chemical properties for the samples were investigated by means of TG/DTA, SEM, cryogenic N₂ adsorption, FTIR and XPS. Dye adsorption on the resultant sample was also measured. The textural properties of the activated carbon fibers were found to be strongly dependent on the activation temperature. Activated carbon fibers exhibited narrow pore size distributions with maxima in the micropore and small mesopore regions. BET surface area, total pore volume, micropore volume and mesopore volume increased with the increase of activation temperature up to 450 °C and then decreased with further heating, and a sample with maximum surface area of 1142 m² g⁻¹ and total pore volume of 0.67 cm³ g⁻¹ was obtained. Phosphoric acid facilitated the conservation of porous structure, led to the creation of tremendous porosity, and resulted in various P-containing functional structures on the surface and in the bulk phase of the resultant samples. The adsorption of acid blue 9 on the sample could be favorably described by Langmuir isotherm, and the adsorption kinetics was found to be well fitted by the intraparticle diffusion model.

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1. Introduction

With the fast development of society and economy, the demand of renewable and environmental friendly energy and raw materials sources becomes urgent due to the pollution of environment, global warming and depletion of fossil oil reserves. Hemp (*Cannabis sativa* L.) is one of the most productive and useful plants known, and widely planted in China. It grows quickly without any great need of pesticides in most locations and climates with only moderate water and fertilizer requirements, becoming a valuable and environmentally friendly crop.

Nowadays, hemp is regarded to be of important industrial and economic value as a source of paper, textiles, building materials, food, medicine, paint, detergent, varnish, oil, ink, and fuel [1], especially in the new research field such as biodiesel production from hempseed oil and textiles production from hemp stems. As a result, the use of hemp generates an extensive amount of solid residues, which were set on fire under most situation and presents an environmental problem in terms of adequate disposal.

One of the effective alternative uses of agricultural waste biomass that has gained much attention over the last decade

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is the production of activated carbons by thermo-chemical conversion [2], and many agricultural wastes have been proved to be promising raw materials for the production of activated carbons [3]. Activated carbon fiber (ACFs) constitute a particular type of activated carbon with certain specific advantages such as extended specific surface area, high adsorption rates and capacities, microporous structure, special surface reactivity and ease of synthesis in form of cloths or felts [4]. Thus, ACFs exhibit a wide applicability in separation, purification and catalytic processes in comparison with powdered or granular activated carbons [5].

It is widely agreed that the pore structure and pore size distribution of an activated carbon is largely determined by the nature of the starting material [6]. Studies also have shown that it is easier to activate carbonaceous materials that contains a significant proportion of fibrous structure like cellulose and hemicellulose rather than those that has less fibrous structure but higher percentage of lignin in nature [7], because of a less robust and more labile nature of cellulose and hemicellulose as compared to lignin [8]. It is known that hemp stems consist of about 65% woody core fibers and 35% bast fibers, and the core fibers consist of 40–48% cellulose, 18–24% hemicellulose and 21–24% lignin, and the bast fibers are 57–77% cellulose, 9–14% hemicellulose and 5–9% lignin [9]. Obviously, the property of rich in cellulose and hemicellulose and low content of lignin for hemp bast together with its low price, abundant source and benefit for environment protection make it an attractive precursor for ACFs compared with core fibers.

Recently, Rosas et al. [10] has prepared ACFs by chemical activation of hemp fibers with phosphoric acid, and analyzed the influence of the activation temperature and the impregnation ratio on the porous structure and surface chemistry of ACFs, which presents a high oxidation resistance attributing to the existence of C–PO₃ and C–P, acting as a physical barrier, blocking the active carbon sites for the oxidation reaction. However, there are still few works on the preparation of ACFs derived from hemp bast. The main objectives of the present work were to prepare hemp bast-based ACFs by phosphoric acid activation under different activation temperatures, and to examine the surface texture, chemistry and dye adsorption properties of the final samples.

2. Materials and methods

2.1. Preparation of samples

The hemp bast was taken from the eight months of growth hemp with a stage of maturity at harvest of 90–120 days in Xishuangbanna Dai Autonomous Prefecture (E100°50', N21°95'), Yunnan Province in China. An anti-pulling decorticator was used to isolate the bast from hemp stem, and the hemp bast was stored in a special chamber with good ventilation, temperature of 18–23 °C and relative humidity of 40–65%. Clean hemp bast of about 1.5–2.0 m long was folded into about 10 cm long bundles, and each bundle was about 40 g in weight. Five bundles (ca. 200 ± 5 g) were impregnated with 4 L phosphoric acid aqueous solution of 2.5 mol L⁻¹ prepared by phosphoric acid (85 wt.%, A.R.). The mixture was

boiled at 110 °C for 6 h and then filtrated. Then the precursor was dried with vacuum dryer at 60 °C for 24 h. The impregnated hemp bast was placed in a tubular furnace and activated under continuous N₂ flow (50 mL min⁻¹). The activation temperature was reached at a heating rate of 5 °C min⁻¹ and maintained for 2 h, and then the system was cooled to room temperature in N₂ atmosphere. The resultant ACFs were washed with hot distilled water until the pH value of the washing solution reached 6–7, and then they were dried in an oven at 110 °C and weighted to determine the yield of the activation process (weight of activated carbon related to weight of raw material, in dry basis). Different activation temperatures within 400–600 °C in step of 50 °C were studied, and the ACFs were labeled as P×××, ××× represents the activation temperature in degree Celsius.

2.2. Characterization of samples

Thermogravimetric experiments were carried out with a TG/DTA thermal analyzer to investigate the pyrolysis process and activation mechanism. Two samples, raw hemp bast dried at 110 °C and hemp bast impregnated with H₃PO₄ were measured, and the weight loss was recorded in the range 0–1100 °C. About 10 mg of sample was heated from 0 to 1100 °C at a ramping rate of 10 °C min⁻¹ under flowing nitrogen at a rate of 50 mL min⁻¹ throughout the measurements.

The surface morphologies of the resultant samples were imaged by using a scanning electron microscope Model Cambridge S250Mk3.

Textural characteristics were determined by N₂ adsorption at –196 °C on automatic apparatus (ASAP 2020, Micromeritics), and the samples were degassed at 250 °C for 12 h under vacuum before the measurements. The specific surface areas (S_{BET}) were estimated by the Brunauer–Emmett–Teller (BET) equation [11], and the pore size distribution (PSDs) was estimated by the density functional theory (DFT) [12]. The total pore volume (V_{tot}) was calculated by the adsorption data at P/P₀ = 0.995. The micropore volume (V_{mic}), mesopore volume (V_{mes}) and macropore volume (V_{mac}) were obtained by applying DFT method.

The chemical characterization of functional groups in bulk phase and on surface of the samples was studied by a Fourier transform infrared spectrometer (FTIR-2000, Perkin–Elmer) in the range 4000–400 cm⁻¹, using pellets with samples dispersed in KBr.

The elemental contents and functional groups types on the surface of the samples were measured below 10⁻⁶ Pa using a Physical Electronics VG ESCALAB MK II XPS spectrophotometer with an achromatic MgKα X-ray source. The wide scan, C_{1s}, O_{1s} and P_{2p3} core level spectra of the samples were recorded at pass energy of 20 eV. The peak positions were corrected based on the most intense graphitic peak, which was taken as 284.6 eV. The spectra obtained were curve-fitted with the nonlinear least-square iterative technique based on the Gaussian function after baseline subtraction using Shirley's method [13].

2.3. Adsorption of acid blue 9

Acid blue 9 (CAS: 2650–18–2) was chosen as adsorbate, and the sample prepared at 450 °C was chosen as adsorbent. The

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