



Biomass-derived highly porous functional carbon fabricated by using a free-standing template for efficient removal of methylene blue



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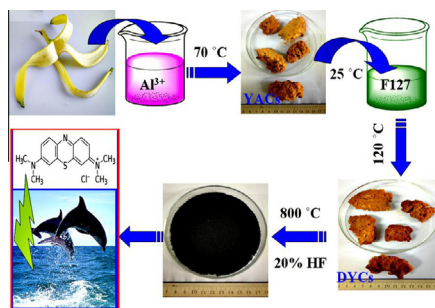
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HIGHLIGHTS

- Highly porous functional carbon material (HPFCM) was fabricated from banana peel.
- A free-standing template way was used for fabrication of HPFCM.
- Adsorption behavior of HPFCMs was examined by using methylene blue model dye.
- The dye adsorption capacity of as-obtained HPFCM is far more than activated carbon.
- The adsorption mechanism and kinetics were systematically investigated.

GRAPHICAL ABSTRACT



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ABSTRACT

Banana peel (BP), a biomass waste, was converted into a valuable highly porous functional carbon material (HPFCM) by a general chelate-assisted co-assembly process. The HPFCMs were fabricated by using Al(III)-based metal-organic framework-like as a free-standing template and commercial Pluronic F127 as a microstructure-directing agent. Several critical variables for fabrication including doses of Al(III) and F127, carbonization temperature had been optimized and the adsorption behavior of HPFCMs was examined by using methylene blue as dye model compound. The optimal adsorbent was validated as HPFCMs-5-1-800, and its equilibrium data were well fitted to the Langmuir isotherm model with a monolayer adsorption capacity of 385.12 mg g⁻¹ at ambient temperature. The surface physical properties of HPFCMs-5-1-800 were also exemplarily characterized. The findings revealed that the free-standing template is a potential route for preparation of HPFCM from waste BP.

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1. Introduction

Millions of tons of highly colored wastewater are discharged from different sources including plastic, textile, leather, cosmetics, paper-making, printing and dye manufacturing industries (Feng et al., 2012). The colored synthetic compounds of removal have been recently attracted tremendous attention due to they are

hazardous to human being and environments. Various treatments for the removal of dyes have been investigated, including adsorption, chemical coagulation, photodegradation, biodegradation, active sludge, etc. (Zhuang et al., 2009). Among them, adsorption on porous carbons is one of the most efficient processes for dye removal and decoloration (Zhuang et al., 2009) because of large specific surface areas, pore volumes, chemical inertness, and good mechanical stability of carbon. Specifically, activated carbon (AC) can treat dyes in wastewater that show difficulty in biodegradation, e.g., azo dyes (Chatterjee et al., 2011). However, the practical applications of AC are restricted because of micropore sizes (Zhuang et al., 2009). Thus, the development of porous carbon materials

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(PCMs) with higher surface areas and larger pore volumes are considerably important for efficient application in dye removal. Extensive methods have been employed to fabricate various PCMs, including hard and soft templating or activation methods (Fechler et al., 2013; Lee et al., 2006). Traditional inorganic materials zeolite and silica are known templates for casting porous carbon. However, this method is costly, complicated and always involves in highly toxic substances, which hinders its application in large scale production.

Recently, metal–organic frameworks (MOFs) with tunable pore size and functionality (Eddaoudi et al., 2002) have been demonstrated as novel potential templates for PCMs synthesis. MOFs can allow the entry and polymerization of soft carbon precursors inside the pores, similar to mesoporous silica and zeolites (Pachfule et al., 2012). Isoreticular zeolitic imidazolate frameworks (Pachfule et al., 2012), MOF-5 (Liu et al., 2008), Al-based porous coordination polymer (Radhakrishnan et al., 2011), zeolitic imidazolate framework-8 (Jiang et al., 2011), and isoreticular metal organic frameworks (Yang et al., 2012) have been successfully used as templates for fabricating PCMs using furfuryl alcohol (FA) as the carbon precursor. It is noteworthy that the Al-based coordination polymer exhibits a three-dimensional framework, which has been used as a very promising precursor for preparing high surface area and large pore volume nanoporous carbon (Radhakrishnan et al., 2011; Hu et al., 2012). In spite of this strategy shows an effective way to produce porous carbon, there are still some aspects should be noticed. The most obvious part is that all the works are based on MOFs, which are subject to costly, and time consuming building-up processes. It's very much worth noting that *N,N'*-dimethylformamide (DMF) and *N,N'*-diethylformamide (DEF) are often used in preparing MOFs. These solvents are toxic and teratogenic reagents that need special actions to dealing with. Thus the application of this method in industry is limited, and the super-high surface areas of these materials drop drastically when the processes are scaled up (Hu et al., 2012).

Carbon materials fabricated from waste biomass have shown promising applications as sorption materials, biochemicals, and others (Hu et al., 2010; He et al., 2013). Lately, various low-cost adsorbents, such as crude biomass, chemically modified biomaterials and some industrial wastes were desirably investigated in order to provide a competitive substitute for AC in purifying the colored wastewater (Mahmoud et al., 2012; Piccin et al., 2012). To date, there is still no general and satisfactory process for the large-scale production of valuable carbon materials from crude biomass. Banana peel (BP), a common agricultural waste, represents 40% of the total weight of fresh banana. It is composed of biopolymers in plant cell walls such as celluloses, hemicelluloses, pectins, lignins and proteins (Lv et al., 2012). Some investigations had reported BP as an economical and selective sorbent for the adsorption of heavy metal ions from aqueous solution (Liu et al., 2012; Lv et al., 2012), which is mainly attributed to the metal ions can easily bind to carboxyl and hydroxyl groups on the surface of pores.

In order to make waste BP into high-value-added product and highly porous carbon materials, the viability of BP as an efficient carbon precursor for fabrication of highly porous functional carbon materials (HPFCMs) was investigated in this work. HPFCMs were fabricated via using Al-based metal–organic framework-like (denoted as Al-based MOF-like) as a free-standing template, and Pluronic F127 (i.e., PEO–PPO–PEO) as a structure-directing agent. The significant influences of various treatment ways, structural functional and surface chemistry of the prepared adsorbent were evaluated. The adsorption equilibrium, the adsorption isotherms and kinetics for methylene blue were further inspected.

2. Methods

2.1. Materials and chemicals

Crude BP was obtained from a local fruit market in Xi'an, China and thoroughly washed with distilled water before use. The commercial triblock copolymers, Pluronic F127 (poly(ethylene oxide)-block-poly(propylene oxide)-block-poly(ethylene oxide), PEO₁₀₆-PPO₇₀PEO₁₀₆, MW 12600) was purchased from Sigma–Aldrich Corp. Al(NO₃)₃·9H₂O, KOH, NaOH, KNO₃, HF, CH₃COOH, NaCl, HCl and ethanol were purchased from the Sinopharm Chemicals Co., Ltd. (Shanghai, China). Methylene blue (MB) was purchased from Shanghai Chemical Corp. MB stock solutions of 1000 mg L⁻¹ was prepared and further diluted to the required concentrations for testing the adsorption capacity and investigating the adsorption process. All chemicals were of analytical grade and used without further purification. Ultrapure water (18.2 MΩ cm) was produced by a Millipore purification system (USA) and used to prepare all aqueous solutions.

2.2. Fabrication of highly porous functional carbon materials

The fabrication process of the HPFCMs is shown in Fig. 1. Typically, about 1.6 kg BP fragments completely submerged in a certain concentration of Al(NO₃)₃·9H₂O aqueous solution (2 L) for a week at 70 °C. In this process, the –COOH and –OH on the surface of the BP pores could be coordinated easily with absorbed aluminum ions with the gradual evaporation of solvent and a type of yellow Al-based metal–organic framework-like complexes (denoted as Al-based MOF-like complexes, i.e. YACs) were obtained after drying at 70 °C. Subsequently, YACs were submerged in an ethanol solution of F127 for 24 h at 30 °C, and then held for 24 h at 120 °C for thermo-polymerization, and finally translated into deep yellow Al-based composites (denoted as DYCs) with solvent evaporation induced self-assembly way. The DYCs were carbonized at 800 °C at a heating rate of 1 °C min⁻¹ for 3 h under N₂ atmosphere. The resultant black monoliths were treated with 20 wt% HF for 24 h to remove the Al species, washed with deionized water and anhydrous ethanol (at least each 4 times), dried at 100 °C for 24 h and HPFCMs were obtained. The as-synthesized samples were designated “HPFCMs-*a-b-c*,” where “*a*”, “*b*”, and “*c*” indicate the mass ratio of Al-based MOF-like complexes to F127, the concentration of Al(NO₃)₃·9H₂O solution (M), and the carbonization temperature (°C), respectively. To compare the adsorptive property, other carbon materials were fabricated by direct carbonization of BP (denoted as DCBP) and chemical activation of BP using 1.0 M KOH as the activating agent under the same carbonization conditions (800 °C in N₂).

2.3. Adsorption equilibrium studies

Batch adsorption experiments were carried out in a set of 250 mL conical flasks containing 0.10 g HPFCMs-5-1-800 adsorbent and 150 mL MB aqueous solutions with various initial concentrations (50, 100, 150, 200, 250, 300 and 350 mg L⁻¹). The flasks were agitated in a water-bath shaker at 30 °C and shaking speed of 120 rpm for 8 h. After above treatments, the samples were centrifuged to minimize interference of carbon fines with the analysis, then 1 mL of the supernatants was diluted to a suitable concentration and the absorbance of the supernatants at 665 nm (Liang et al., 2012) was measured using a double beam TU-1901 UV–visible station (Beijing Puxitong Analytical Ltd., China). Each experiment was carried out in triplicate under identical conditions and an average value was employed. The amount (q_e , mg g⁻¹) of

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