Applied Surface Science 299 (2014) 86–91

Contents lists available at ScienceDirect

Applied Surface Science

journal homepage: www.elsevier.com/locate/apsusc

Preparation and characterization of activated carbon fiber (ACF) from cotton woven waste

Jieying Zheng, Quanlin Zhao, Zhengfang Ye*

Department of Environmental Engineering, Peking University, The Key Laboratory of Water and Sediment Sciences, Ministry of Education, Beijing 100871, China

ARTICLE INFO

Article history:
Received 28 October 2013
Received in revised form
18 December 2013
Accepted 30 January 2014
Available online 6 February 2014

Keywords:
Cotton woven waste
Activated carbon fiber
N2 adsorption test
XPS

ABSTRACT

In this study, the activated carbon fibers (ACFs) were prepared using cotton woven waste as precursor. The cotton woven waste was first partly dissolved by 80% phosphoric acid and then was pre-soaked in 7.5% diammonium hydrogen phosphate solution. Finally, carbonization and activation were proceeded to get ACF. The optimum preparation conditions, including carbonization temperature, carbonization time, activation temperature and activation time, were chosen by orthogonal design. Nitrogen adsorption/desorption test was conducted to characterize the prepared ACF's pore structure. Fourier transform infrared spectroscopy (FTIR) analysis, X-ray photoelectron spectroscopy (XPS) and environmental scanning electron microscope (ESEM) were employed to characterize its chemical properties and morphology. Adsorption of oilfield wastewater was used to evaluate its adsorption properties. The results show that the prepared ACF is in the form of fiber, with the sectional diameters of 11.7 × 2.6 μm and the surface area of 789 m^2/g. XPS results show that carbon concentration of the prepared ACF is higher than that of the commercial ACF. When the prepared ACF dosage is 6 g/L, over 80% of COD and over 70% of chrominance can be removed after 24 h of adsorption at 18 °C.

© 2014 Elsevier B.V. All rights reserved.

Introduction

Activated carbon fiber (ACF) is considered a kind of promising adsorption material, which can be widely used for treatment of organic wastewater [1–4] and the recovery of noble metals or metal adsorption [5,6]. It shows excellent adsorption performances because of its nano-structure, abundant micrometer porosity, high specific surface area and narrow pore size distribution. Currently, the major precursors for producing ACF are rayon, acrylic, polyacrylonitrile (PAN), novoloid (novolac resin) and viscose rayon fibers, etc. ACF can be made from them by pretreatment and subsequent carbonization and activation [7]. In the traditional technologies, the high cost of precursor hinders its large-scale application [8]. So it is essential to find a cheap substitute material for ACF production.

In recent years, great amount of cotton woven waste are produced, which need to be recycled or disposed effectively. Such researches have been focused on landfill, incineration, composting and recycling [9,10]. Since cotton woven waste is composed of cotton fiber, it may be a potential substitute material for precursor to produce ACF [11]. In our present work, we investigated the feasibility of cotton woven waste to be used as precursor for production of ACF. Phosphoric acid was used to dissolve cotton woven waste partly to slightly loose the fibrils bundled together, which makes its character closer to that of individual cotton fibrils.

The aim of the present work was to prepare ACF using cotton woven waste as precursor. The pore structure of the prepared ACFs were evaluated by nitrogen adsorption/desorption test. Fourier Transform Infrared (FTIR) spectroscopy analysis and X-ray photoelectron spectroscopy (XPS) were conducted to determine its chemical properties. Environmental scanning electron microscope (ESEM) was used to observe its surface morphology. Furthermore, its adsorption efficiency for treatment of oilfield wastewater was also evaluated, with commercial ACF as comparison.

Experimental

Materials

In this study, cotton woven waste was collected from tailor's shop. It was cut to produce pieces of size 5 × 2.5 cm, washed several times with deionized water, and then dried at 80 °C for 24 h. High purity (99.999%) nitrogen gas and carbon dioxide gas (90%) were purchased from Hengyuantong Gas Co. Ltd. (Beijing, China). Other chemicals were analytical grade. A commercial ACF (BEGF 1000) provided by Sutong Carbon Fiber Corporation (Jingsu Province, China) was used as reference, with the surface area of 950 m^2/g.

* Corresponding author. Tel.: +86 10 62755914; fax: +86 10 62756526.
E-mail addresses: zhengfangye@163.com, yezhengfangiee@163.com (Z. Ye).

0169-4332/$ – see front matter © 2014 Elsevier B.V. All rights reserved.
http://dx.doi.org/10.1016/j.apsusc.2014.01.190

Preparation of ACF

The pre-weighed amount of cotton woven (CW) was put into phosphoric acid (80%) solution with the solid loading of 10% (w/v), and stirred for 5 min. After 1 h aging, it was taken out and washed by distilled water several times to remove residual solution. The pretreated cotton woven (PCW) was dried in an oven at 80 °C for 24 h, then put out and cooled under ambient temperature for 12 h. The cooled PCW was soaked in $(NH_4)_2HPO_4$ solution (7.5%) at room temperature for 3 h and then squeezed and dried at 80 °C for 24 h. After cooling to ambient temperature, it was transferred to an alumina boat and put into a muffle furnace. The N_2 gas was used as protective gas with the flow rate of 600 mL/min. The temperature raised from 25 °C to 500–800 °C with the rate of 5 °C/min and was kept for 30–60 min to proceed carbonization. Then it was heated to 700–800 °C with the rate of 10 °C/min and kept for 30–60 min to process activation under CO_2 atmosphere with the flow rate of 0.5 L/min. After the temperature decreased to 300 °C under N_2 flow naturally, the product was removed from the furnace and cooled to ambient temperature. Then it was washed by distilled water until a neutral pH was reached. Finally it was dried at 80 °C for 24 h to get ACF.

Characterization of ACFs

Nitrogen adsorption measurement

Nitrogen adsorption/desorption test was conducted on an ASAP 2010 gas sorption analyzer (Micromeritics, USA). The N_2 apparent surface area was calculated using the BET equation in the relative pressure range of 0.07–0.20. The total pore volume was estimated from the amount of N_2 adsorbed at $p/p_0 = 0.99$.

Iodine value measurement

Iodine value of the prepared ACF was determined according to Chinese standard GB/T 12496.8-1999. An amount of 0.5 g dried ACF was weighed and transferred to a 100 mL iodine flask equipped with a ground glass stopper. Then 10 mL HCl (10 wt%) was added to fully wet the sample. The mixture was heated to boil and maintained for 30 s. After cooling to room temperature, 50.0 mL iodine standard solution (0.1 mol/L) was put into the flask. The flask was stoppered and the contents were vigorously shaken for 15 min. Then the mixture was quickly filtered into a beaker. 10.0 mL filtrate was pipeted into a 250 mL iodine flask and 100 mL water was added. The standardized $Na_2S_2O_3$ solution (0.1 mol/L) was used to titrate until the solution turned to pale yellow. Then 2 mL starch indicator solution was added and the titration was continued until the solution became colorless. The iodine value (mg/g) can be calculated according to the following equation:

$$\text{iodine value} = \frac{5 \times (10c_1 - 1.2c_2V_2) \times 127}{m} \times D \quad (1)$$

where c_1 (mol/L) and c_2 (mol/L) are the concentrations of iodine standard solution and sodium thiosulfate standard solution, respectively. V_2 (mL) is the consumed volume of sodium thiosulfate standard solution and m (g) is the mass of ACF. D is the correction coefficient.

FTIR analysis

The surface functional groups were investigated by FTIR analysis using a Magna-IR750 spectrometer (NICOLET, USA), with 128 scans and a resolution of 8 cm^{-1}.

XPS analysis

XPS analysis was conducted using an Ultra Axis X-ray photoelectron spectrometer (SHIMADZU, Japan) equipped with an achromatic Al $K\alpha$ X-Ray source. The pressure during the analysis was 6.70×10^{-8} Pa and the take off angle was 90°. Survey scans in the range 0–1000 eV with step size 1 eV and high-resolution scans with step size 0.1 eV for C 1s was recorded. The binding energies were corrected by referencing to the hydrocarbon component at 284.8 eV.

Table 1
Factors and levels of orthogonal design.

Level	Factor			
	(*A*) Carbonization temperature (°C)	(*B*) Carbonization time (min)	(*C*) Activation temperature (°C)	(*D*) Activation time (min)
1	500	30	700	30
2	600	60	800	60
3	700			
4	800			

ESEM analysis

The surface morphology of the prepared ACFs was observed by a Quanta 200FEG environmental scanning electron microscope (FEI, USA).The accelerating voltage was 20 kV and the pressure of the chamber was 60 Pa.

Results and discussion

Optimization of ACF's preparation conditions

Orthogonal design was utilized to optimize the preparation conditions of ACF. $L_8(4^1 \times 2^4)$ orthogonal design (Table 1) was used to optimize the preparation conditions, such as carbonization temperature (A), carbonization time (B), activation temperature (C) and activation time (D). The adsorption properties of the prepared ACFs were evaluated by measuring iodine value, which was determined according to Chinese Standard GB/T 12496.8-1999.

Table 2 lists the iodine values and range analysis. It can be seen that the sum of iodine value K_{C2} (3978.8 mg/g) is bigger than K_{C1} (2941.9 mg/g). For other factors, $K_{A3} > K_{A4} > K_{A2} > K_{A1}$, $K_{B1} > K_{B2}$, $K_{D1} > K_{D2}$. Thus the optimum preparation conditions are as followed: carbonization temperature 700 °C, carbonization time 30 min, activation temperature 800 °C, activation time 30 min. Under this condition, the iodine value of the prepared ACF is 1146.0 mg/g.

F-test was employed to evaluate the influence of the four factors. Since carbonization time has little influence on the result, it is treated as error term. As can be seen in Table 3, activation temperature (with 72.75% contribution) is the most significant factor ($F_C = 35.24 > F_{0.05}(1, 2) = 18.51$). Activation time, carbonization temperature and carbonization time have less influence, respectively.

Table 2
$L_8(4^1 \times 2^4)$ orthogonal test results.

Sample no.	*A* (°C)	*B* (min)	*C* (°C)	*D* (min)	*E*	Iodine value (mg/g)
1	500	30	700	30	1	721.6
2	500	60	800	60	2	917.7
3	600	30	700	60	2	680.6
4	600	60	800	30	1	999.4
5	700	30	800	30	2	1146.0
6	700	60	700	60	1	700.1
7	800	30	800	60	1	915.7
8	800	60	700	30	2	839.6
K_1 (mg/g)	1639.3	3463.9	2941.9	3706.6	3336.8	
K_2 (mg/g)	1680.0	3456.8	3978.8	3214.1	3583.9	
K_3 (mg/g)	1846.1					
K_4 (mg/g)	1755.3					
R	206.8	7.1	1036.9	492.5	247.1	

Download English Version:

https://daneshyari.com/en/article/5361902

Download Persian Version:

https://daneshyari.com/article/5361902

Daneshyari.com