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Textural and morphological study of activated carbon fibers prepared from kenaf

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Abstract

Activated carbon fibers (ACFs) are relatively novel fibrous adsorbents that show important advantages with respect to conventional activated carbons. In this work the influence of the preparation conditions on the fractal dimension and porous texture of ACFs prepared from kenaf by physical activation has been investigated. The kenaf fibers were first carbonized, the sample showing optimal textural properties (i.e., that carbonized at 400 °C for half an hour) being used as precursor for the activation treatment. This sample was subsequently activated between 500 and 700 °C for 2 and 4 h. All samples were characterized by N_2 adsorption at −196 °C, mercury porosimetry and scanning electronic microscopy (SEM). The adsorption data reveal that, in general, the textural parameters increase markedly with temperature and time. On the other hand, the mercury porosimetry experiments suggest that the macropore volume varies slightly in all the temperature and treatment time interval. The fractal dimension (D) of the samples has been calculated for micropores by applying the Frenkel–Hansey–Hill (FHH) equation, whereas data from the mercury porosimetries were used to determine the fractal dimension corresponding to macropores.The variation pattern of D points out that, as temperature rises, the thermal treatment and the presence of activating agent favor the porous development of the samples. Finally, the SEM images suggest the occurrence of interconnection and opening of pores as well as of detachments of the external layers of the pores. As a consequence more homogeneous pores with smoother surfaces are observed.

Keywords: Kenaf; Activated carbon fibers; Porous texture; SEM images; Fractal dimension

1. Introduction

Porous materials are usually heterogeneous both structurally and energetically. Among porous solids used in industry, activated carbons are the most complex ones. On the other hand, they are the most versatile due to their extremely high surface area and micropore volume [1]. Activated carbon fibers (ACFs) are relatively novel fibrous adsorbents produced for example from pith, cellulose, lignocellulose, phenol resin and polyacrylonitrile [2–7]. ACFs show important advantages with respect to conventional activated carbons. Among these advantages it is worth noting their smaller fiber diameter (which minimizes diffusion limitations and allows rapid adsorption/desorption), more concentrated pore size distribution, and excellent adsorption capacity at low concentrations of adsorbates. Their main inconvenience lays on the difficulty of choosing adequate activating agents and activation conditions that are required in order to maintain the fibrous morphology of the raw material.

The adsorption capacity of ACFs depends on many factors, such as raw materials, activation process, pore

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structure and surface functionalities [8–10]. Surface roughness is an important factor that influences the adsorption properties of carbonaceous adsorbents. Fractal dimension is a measure of roughness of a surface. The use of the fractal concept is becoming very popular as a tool to characterize the texture of complex materials, such as porous solids [11]. The fractal properties of these porous systems have been determined by means of several techniques such as gas adsorption [12–19], mercury porosimetry [20–27] and small-angle X-ray scattering (SAXS) [28]. These methods should be treated as complementary, because the data obtained from analyses of adsorption isotherms and pore size distribution curves can detect irregularities of different parts of the surface.

Kenaf is an herbaceous annual plant that belongs to the family of *Malvaceae*. Kenaf posseses two kinds of fiber that may be explored to produce ACFs. The long fiber supposes approximately 30% of the total volume of the plant whereas the short fiber represents the remaining 70%. The aim of this work is to study the influence of the preparation conditions on the fractal dimension and porous texture of ACFs prepared from long fibers of kenaf by physical activation using carbon dioxide as the activating agent.

2. Experimental

2.1. Preparation of the samples

The precursor used for the production of ACFs was kenaf. For the production of the ACFs a Termolab tubular furnace equipped with Eurotherm 904 temperature controllers and a 1 m long tubular ceramic insert was used. The temperature within the furnace was first calibrated and the length and position of the constant temperature hot zone determined. About 1.5 g of kenaf fiber were placed in a 10 cm stainless steel boat with perforated ends to facilitate gas flow and positioned in the center of the constant temperature zone. The carbonization of the samples was carried out at 300 and 400 °C. The heating rate was 5 °C min^{-1} under a constant N_2 flow of 85 cm^3 min^{-1}. The isothermal carbonization time was 0.5 or 1 h (see Table 1). The carbonized sample showing optimal characteristics in terms of porosity and surface area was selected as the precursor for the preparation of the activated fibers. The activation temperatures were 500, 600 and 700 °C. A flow of activating agent (carbon dioxide) equal to 85 cm^3 min^{-1} was used, the heating rate being 5 °C min^{-1}. The isothermal activation time was 2 or 4 h (see Table 2).

Table 1
Nomenclature and preparation conditions of the different carbonized samples

Sample	Carbonization temperature (°C)	Carbonization time (h)
CF3-0.5	300	0.5
CF4-0.5	400	0.5
CF3-1	300	1
CF4-1	400	1

Table 2
Nomenclature, preparation conditions and burn-off of the different activated samples

Sample	Carbonization temperature (°C)	Activation temperature (°C)	Activation time (h)	Burn-off (%)
ACF5-2	400	500	2	8
ACF5-4	400	500	4	8
ACF6-2	400	600	2	14
ACF6-4	400	600	4	15
ACF7-2	400	700	2	32
ACF7-4	400	700	4	70

Once the isothermal activation time had finished the activating agent flow was replaced by a N_2 flow and the furnace was cooled to a final temperature of 50 °C. Next, the sample was removed from the oven and kept in a tightly closed container.

2.2. Characterization of the samples

The samples were texturally characterized by gas adsorption (N_2, −196 °C), mercury porosimetry and scanning electron microscopy (SEM). The samples were always first oven dried at 120 °C for 24 h. Adsorption isotherms of N_2 (purity ⩾ 99.998%) at −196 °C were collected using an adsorption apparatus (Autosorb-1, Quantachrome). Approximately 0.15 g of sample were used in each adsorption experiment. Adsorbents were placed in a glass container and degassed at 10^{-3} Torr at 120 °C overnight prior to the adsorption measurements. A Quantachrome porosimeter, Autoscan-60, was used to obtain the mercury intrusion curves. The values of surface tension and contact angle used in the computational program of the porosimeter were 0.480 N m^{-1} and 140°, respectively. The sample morphology was observed using a scanning electron microscope (SEM; model S-3600N, Hitachi, Japan). The specimens for SEM observation were prepared by depositing the fibers onto specimen-stubs with conductive double-sticky copper tapes, and then sputter-coating (model Polaron SC7640, Quorum Technologies Ltd, UK) the fiber surfaces with Au–Pd to prevent electrical charging during examination. Imaging was done in the high vacuum mode at an accelerating voltage of 20 kV, using secondary electrons.

The fractal dimension (D) is often used as an index of roughness or irregularity of the surface. Recently, several researchers reported the fractal dimension of activated carbon [29–32]. In this study, the fractal dimension was determined by applying the following Frenkel–Hansey–Hill (FHH) equation [33] to the N_2 adsorption data [34,35]:

$$\frac{q}{q_e} = K\left[\ln\frac{P_0}{P}\right]^{(D-3)} \quad (1)$$

where q is the amount adsorbed at equilibrium pressure, P; q_e is the amount adsorbed filling micropore volume; P_0 is saturated pressure; K is a constant; D is the fractal dimension. Thus, the plot of ln q versus ln(ln(P_0/P)) shows linear