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Activated carbon fibers from meltblown isotropic pitch fiber webs for vapor phase adsorption of volatile organic compounds

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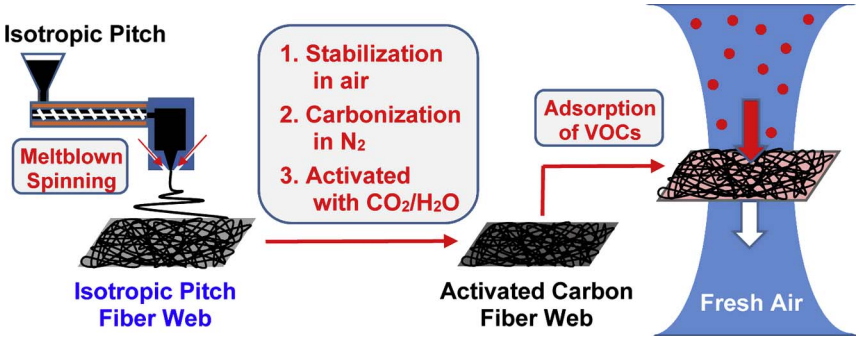
GRAPHICAL ABSTRACT



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ABSTRACT

Activated carbon fibers (ACFs) webs with potentially lower cost and high adsorption performance are fabricated from isotropic pitch by meltblowing process, stabilization in air, carbonization in N_2 and activation in CO_2/H_2O. The stabilization and activation conditions are studied and evaluated to prepare ACFs with high carbon yields and high surface area. The prepared ACFs are typical microporous adsorbents having a specific surface area of 1000–2000 m^2/g and a corresponding change in pore volume and pore size distribution. The selected ACF with a surface area of 1985 m^2/g has strong adsorption affinity for chloroform vapor and is superior to the commercial product Kynol® ACF-15 textile in terms of adsorption amount and adsorption rate due to its high specific surface area and fiber web form. Vapor phase adsorption of volatile organic compounds (VOCs) in a Thermogravimetric Analyzer demonstrates that this ACF can effectively remove different VOCs from air.

1. Introduction

Activated carbon fibers (ACFs) and their fabrics developed in the 1960s' offer a number of advantages over conventional adsorbents granular and powdered activated carbons as adsorbent materials. ACFs possess excellent adsorption properties for gas and liquid molecules, larger adsorption capacities, fast adsorption rate, and ease of regeneration [1–3]. Additionally, ACFs can be made into yarn, thread, fabric, felt/carpet, paper cloth and other shapes to optimize process designs for engineering use. Their ease of handling enables ACFs to be used in various devices for wide industrial, military, and public protection applications. So far, ACFs are used most successfully in the areas of chemical and biochemical adsorption and separation [4–6], air and water purification [7,8], catalysts or catalyst supports [9–11], masks [12,13], medical care purposes [14,15], etc.

However, the commercially available ACFs are very expensive,

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because of high cost of raw materials, fiber spinning and weaving, and subsequent thermal processes, as well as a huge weight losses during activation. As a result, ACFs currently remain a specialty product and as such has been limited for use in niche industrial applications, due to the high cost of fiber precursors and their associated processing costs.

In this study, ACFs with potentially lower cost and excellent performance are prepared by the following route: (1) the use of lower cost, high carbon content isotropic pitch as fiber precursor; (2) the use of patented and high-efficient meltblown fiber spinning method [16]; and (3) the spun fibers in the form of continuous webs are directly converted to ACFs without the need for additional fiber weaving. Since the ACF fiber processing is simple and efficient, the prepared ACF webs should have a potentially low cost compared to conventional ACF (e.g., melt-spun phenolic resin-based Kynol® ACF textiles). Thus, the fabrication conditions including fiber stabilization and activation are investigated in this paper to produce ACFs with high carbon yield and highly porous structure. The carbon yield, burn-off, surface area and pore development are characterized with thermogravimetric analysis (TGA), pore and surface area analysis and the adsorption of iodine and methylene blue. Vapor phase adsorption of different volatile organic compounds (VOCs) on the prepared ACFs is demonstrated in a TGA.

2. Experimental

2.1. Material

The isotropic pitch precursor used in this study was produced in China, and supplied by Professor Larry C. Wadsworth of University of Tennessee at Knoxville, The pitch had a softening point of ~230 °C. Mesophase pitch fiber prepared in the lab and a commercially available ACF textile Kynol® ACF-15 (Nippon Kynol) were used for comparative testing. Other chemicals including volatile organic compounds (VOCs) used as adsorbates were purchased from Fisher Scientific.

2.2. Meltblown fiber spinning

Continuous isotropic pitch fibers (in the form of fiber web) were spun with a patented, highly productive melt-blowing process which has a significant economic advantage over the more common procedure of melt spinning [16]. The spinning system comprises a pitch feeding device, extruder, exhaust vent, ballast pump, spinning pump, filter, spinneret head, air flow regulator, and temperature and pressure, and vacuum control system, etc. as shown in Fig. 1. The fibers are formed aerodynamically at speeds of several hundred meters per second and then slowed down to a few meter per second at the collection zone. The meltblown fiber spinning device comprises a spinneret head heated at 270 °C where exit orifices forms filaments, a venturi adjacent the capillaries that receives the filaments from the spinneret head, a diffuser downstream of the venturi that receives the filaments, one or more air exhaust ports that create in the diffuser an airflow having a direction normal to the direction of flow of the filaments. The number of filaments produced was 10 (adjustable from 1 to 1000). Fibers were collected on a moving belt in a web format. Production rate depended on the diameter, and desired matt thickness, etc. Typical cross section of the spun fibers is circular, with diameters from about 2–100 μm. The most spun fiber diameter is 7–15 μm.

2.3. Fabrication of ACF

The spun fibers were cut to a length of ~12 cm and placed on a stainless steel mesh and then placed in a tube furnace for thermal processing. The sample was first stabilized in air by heating the fibers with a heating rate of ~1 °C/min to 275 °C and then holding the temperature constant for 20 min. The stabilized fiber was carbonized to a given temperature at a heating rate of ~30 °C/min in a flowing N_2, and then activated for a given time with a mixed gas of CO_2/H_2O which

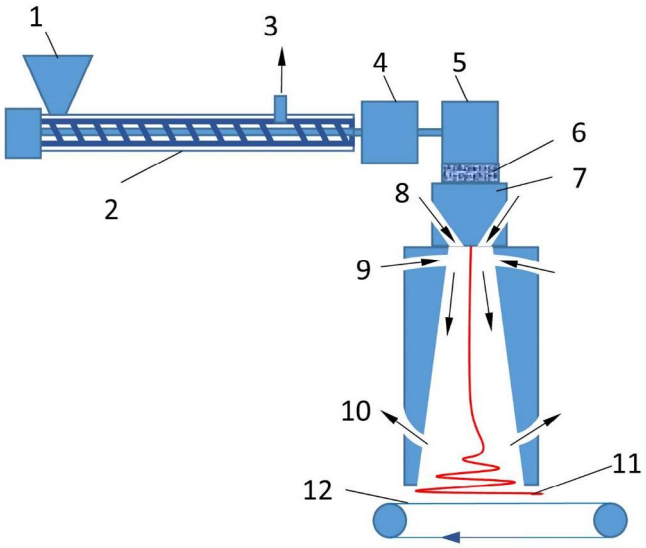

Fig. 1. A schematic diagram of a meltblown fiber spinning apparatus. 1-pitch feeding; 2-extruder; 3- exhaust vent; 4-ballast pump; 5-spinning pump; 6-filter; 7-spinneret head; 8-primary air stream; 9-secondary air stream; 10-vacuum exhaust; 11-spun fibers; 12-moving belt collector.

was generated by bubbling CO_2 through a liquid water reservoir at room temperature (~22 °C). After activation, the ACF was cooled down in a flowing N_2. The burn-off of carbonized fibers during activation and carbon yield of carbonized and activated fibers were calculated as follows:

$$\text{Burn-off(wt \%)} = \frac{\text{Weight loss during activation}}{\text{Weight of carbonized fibers}} \times 100 \quad (1)$$

$$\text{Carbon yield(wt\%)} = \frac{\text{Weight of carbonized or activated fibers}}{\text{Weight of spun fibers}} \times 100 \quad (2)$$

2.4. Adsorption of methylene blue (MB) and iodine

5–100 mg of fiber specimens in 50–200 mL of MB solution (40–50 mg/L of MB) were shaken at room temperature for 2 days to ensure the adsorption equilibrium is reached. The concentration of MB in each sample and the MB standards was measured with a UV spectrophotometer at 660 nm.

ASTM-D4607-94 (2006) standard was used as a guide for the testing and calculations of iodine values. Aqueous I_2/KI solutions with an iodine concentration of 0.05 N were used in adsorption experiments. The I_2 concentration was determined by $Na_2S_2O_3$ titration with a starch indicator.

2.5. Adsorption of vapors

Vapor phase adsorption experiments were carried out with a horizontal SDT Q600, TGA/DSC combo, TA Instruments. An impinger was used to generate a chemical vapor. 30 mL/min N_2 (or air) gas was used to bring chemical vapor into furnace chamber of the TGA from its reaction gas port. The concentration of vapor was diluted with 100 mL/min N_2 (or air) which entered the furnace from the balance and used as purge gas to protect the balance of the TGA. The schematic gas flow direction is shown in Fig. 2. The fiber specimen was cut to a small piece so that they can be loaded into ceramic sample cell in the center of the furnace chamber. Prior to adsorption testing, the sample was dried by heating it to 160 °C with a heating rate at 20 °C/min and a flowing N_2 (or air) at 100 + 30 mL/min. The sample was then cooled down to room temperature with the protection of 130 mL/min N_2 (or air). TGA