



Fabrication of novel porous carbon membrane/sintered metal fibers composite for isopropanol adsorption



Yan Shao^{a,*}, Huanhao Chen^b, Yibiao Li^a, Xiaoou Ma^a

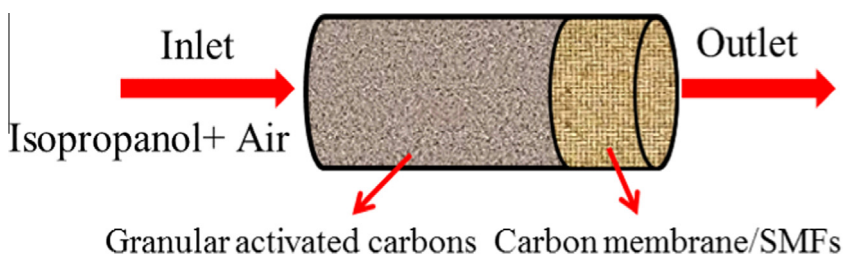
^a School of Chemical and Environmental Engineering, Wuyi University, Jiangmen 52920, PR China

^b Department of Chemical Engineering, University of Puerto Rico-Mayagüez Campus, Mayagüez, PR 00681-9000, USA

HIGHLIGHTS

- Novel carbon membrane/SMFs composite was designed and characterized.
- Structured fixed bed based carbon membrane materials was designed.
- Adsorption behavior of isopropanol in structured fixed bed was investigated.
- Mass transfer and bed utilization were enhanced by adding carbon membranes.

GRAPHICAL ABSTRACT



ARTICLE INFO

Article history:

Received 23 February 2015

Received in revised form 11 April 2015

Accepted 13 April 2015

Available online 20 April 2015

Keywords:

Carbon membrane
Sintered metal fibers
Structured fixed bed
Adsorption
Isopropanol

ABSTRACT

Novel porous thin-sheet carbon membrane/sintered metal steel fibers (SMFs) composites were developed by the dipping and carbonization process. These novel carbon membrane materials have been characterized by a series of techniques including thermal gravimetric analyses (TGA), X-ray powder diffraction (XRD), scanning electron microscopy (SEM), and N₂ adsorption–desorption method. Adsorption breakthrough curves of isopropanol in the structured fixed bed filled with both activated carbons in the inlet of bed and carbon membrane/SMFs composites in the outlet of bed were investigated, comparing with that in fixed bed containing only activated carbons with the same bed height. The experimental results indicated that the carbon membranes with different thickness from 748.1 nm to 1.27 μm were successfully fabricated on the surface of SMFs support. The length of unused bed (LUB) theory was also employed to study the breakthrough curves. The porous carbon membrane/SMFs composites filled in fixed bed show excellent bed utilization, demonstrating by a lowest LUB value (1.17 cm). The porous carbon membrane/SMFs composites as high efficiency adsorbents can offer a much higher mass transfer efficiency, contacting efficiency as well as adsorption rate.

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1. Introduction

Isopropanol as one of the most typical VOCs pollutants have some toxic effect on human health and environmental safety [1–4]. Hence, many technologies have been applied in removal of isopropanol from the air, such as adsorption [3] and catalytic

combustion [2,4]. Adsorption technology has been regarded as useful techniques for the removal of VOCs in air [3,5,6]. However, traditional fixed-bed adsorbers used in the adsorption process have some drawbacks such as low mass transfer and contacting efficiency, poor adsorption rates, and high bed pressure drop due to the use of adsorbent particulates [3]. In order to improve the fixed-bed adsorber performance, structured fixed-bed adsorbers have been widely used in the adsorption process due to their relatively higher mass transfer and contacting efficiency, lower bed pressure drop [3,6,7].

* Corresponding author. Tel.: +86 07503299397.

E-mail address: wuyuchem@126.com (Y. Shao).

Inorganic membrane materials as typical structured composites have widely been used in structured fixed-bed adsorbers due to their short diffusion path, high mechanical strength, high thermal and chemical stability, and high contacting efficiency [3,8–12]. Aguado et al. [8] prepared ZSM-5 catalytic membranes by liquid phase hydrothermal synthesis, and studied their applications in the removal of VOCs in air. Sintered metal fibers (SMFs) based zeolite membranes have been developed for purification of low-content VOCs gas-streams by Nikolajsen et al. [6]. Chen et al. [3,5] also prepared gradient porous ZSM-5 membrane/sintered stainless steel fibers by secondary growth process, and investigated their applications in the adsorption and catalytic oxidation of VOCs in air. It was found that the structured zeolite membranes can effectively improve the mass transfer and contacting efficiency, and adsorption rates. Structured sintered metal fiber (SMF) plates have been recognized as excellent support for inorganic membranes due to their open macro structure, high mass/heat transfer efficiency, high contacting efficiency, thermal and chemical stability [6,13,14].

Carbon membranes as one of the most promising materials have attracted increasing attention due to their outstanding advantages, such as high contacting efficiency, high thermal and chemical stability [9–12,15–19]. Therefore, carbon membranes have been widely used in the field of gas separations [9,12], adsorption, and catalysis [18]. Qin et al. [17] prepared a mesoporous carbon membrane by the organic sol-gel process, and the carbon membrane presents good permeation properties. Carbon membranes derived from poly (phthalazinone ether sulfone) for gas separation were also fabricated by Zhang and co-workers [12,20]. Tanaka and co-workers also developed microporous carbon membranes for pervaporation dehydration [19].

The main objectives of this study were to (1) investigate the fabrication of a novel porous microfibrillar-structured carbon membrane/sintered metal fibers composite; (2) study the application of these carbon membrane composites in structured fixed bed for isopropanol removal.

2. Experimental

2.1. Materials

Sintered metal fibers (SMFs) plates (diameter: 20 μm ; Porosity: 0.75 ± 0.05) used as support were supplied by Xian Fuerth Filter Company (China). Furfuryl alcohol was purchased from Shanghai Jinshan Ting New Chemical Reagent Factory (China). Powdered polyethylene glycol (PEG) samples of average molecular weights of 10,000, 6000 g/mol were obtained from Guangzhou Tianyi Fine Chemical Plant (China). Oxalic acid was purchased from Xilong Chemical Plant (China). Deionized water was used in all synthesis process. All of the chemical reagents used in this study were analytical grade.

Table 1
Synthetic conditions and sample names.

Samples	Polymerization time (h)	PFA/PEG: acetone mixture quality ratio (%)	Soaking time (min)	PFA:PEG ratio	Coating times
Carbon membrane/SMFs-1	7	50	5	1:1	1
Carbon membrane/SMFs-2	10	50	5	1:1	1
Carbon membrane/SMFs-3	14	50	5	1:1	1
Carbon membrane/SMFs-4	10	10	5	1:1	1
Carbon membrane/SMFs-5	10	100	5	1:1	1
Carbon membrane/SMFs-6	10	50	1	1:1	1
Carbon membrane/SMFs-7	10	50	10	1:1	1
Carbon membrane/SMFs-8	10	50	5	1:2	1
Carbon membrane/SMFs-9	10	50	5	1:6	1
Carbon membrane/SMFs-10	10	50	5	2:1	1
Carbon membrane/SMFs-11	10	50	10	1:1	3
Carbon membrane/SMFs-12	10	50	10	1:1	6
Carbon membrane/SMFs-13	10	50	10	1:6	3

2.2. Preparation of carbon membrane/sintered metal fibers composite

Firstly, SMFs support was calcined in air at 550 $^{\circ}\text{C}$ for 5 h to form an oxide layer on the stainless steel fibers surface. To prepare the poly(furfuryl alcohol) (PFA), oxalic acid (0.5 g) was added to furfuryl alcohol solution (100 mL) under continuous stirring at 80 $^{\circ}\text{C}$ for 7 h, 10 h and 14 h, respectively. This PFA/PEG/acetone precursor was prepared by combining PFA with a given average molecular weight of polyethylene glycol (PEG) in a certain percentage of weight ratio at 70 $^{\circ}\text{C}$ and subsequent dilution in acetone forming a stable mixture at 70 $^{\circ}\text{C}$ [21]. And then, the pretreated SMFs support was impregnated with the PFA/PEG/acetone precursor for 1 min, 5 min, and 10 min, respectively. After that, the resulting support was placed in an oven at 100 $^{\circ}\text{C}$ for 24 h. Finally, the polymer membranes were carbonized at 600 $^{\circ}\text{C}$ for 180 min using a heating rate of 2 $^{\circ}\text{C}/\text{min}$ and flowing nitrogen of 80 mL/min. In order to investigate the effect of coating times on the structure of the composites, the coating process and carbonization process were alternately repeated several times. The synthesis conditions of carbon membranes were summary in Table 1. Carbon membrane/SMFs composites were kept in a dryer to isolate them from moisture and dust before using in the adsorption experiments.

2.3. Characterization of carbon membrane/SMFs composites

The suitable carbonization temperature of carbon membrane/SMFs was determined by thermogravimetric analysis (TGA) using a SDT V3.0F (2960) instrument under nitrogen flow at a heating rate of 10 $^{\circ}\text{C}/\text{min}$ from 30 $^{\circ}\text{C}$ to 700 $^{\circ}\text{C}$. Carbon membrane/SMFs composite was tested by X-ray diffraction (XRD) on a Bruker D8 instrument using scanning angles between 10 $^{\circ}$ and 70 $^{\circ}$. N_2 adsorption/desorption isotherm of carbon membranes was gathered at 77 K using Micromeritics ASAP 2020 (Micromeritics Instrument Co., USA). All of the samples were degassed at 200 $^{\circ}\text{C}$ for 6 h. The BET surface area of carbon membrane/SMFs was calculated from adsorption branches using the BET (Brunauer–Emmett–Teller) method. And the pore size distribution of carbon membrane/SMFs composite was obtained via analyzing the adsorption isotherm using BJH (Barrett–Joyner–Halenda) and HK (Horvath–Kawazoe) method. The morphologies of carbon membranes were also observed by a FEI/Nova NanoSEM430 scanning electron microscopy (SEM).

2.4. Adsorption dynamics of isopropanol in structured fixed-bed

Adsorption dynamics of isopropanol in different fixed beds were performed using a continuous flow system including a stainless steel tube (20 mm i.d., 30 mm length). As can be seen in Fig. S1 in Supplementary Material, the structured fixed-bed was filled with granular activated carbons (GACs) (average particle

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