



Amine-impregnated porous nanofiber membranes for CO₂ capture

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ABSTRACT

Global warming is a worldwide issue, mainly caused by excessive emission of carbon dioxide (CO₂), thus, demands the development of robust and flexible sorbents with consider CO₂ capturing capacity. Herein, we report an easy and facile two-step fabrication of hierarchically structured and highly porous polystyrene (PS)/polyurethane (PU) electrospun nanofiber membrane impregnated with various amines for post-combustion CO₂ capture. Synthesized porous nanofiber membranes have highly flexible and strong mechanical characteristics. The comparative analysis of porous membranes functionalized with three functional amines shows that low molecular weight polyethyleneimine (PEI_L) impregnated porous nanofiber membranes offer relatively higher CO₂ adsorption (1.64 mmol/g) at 40 °C. In addition, optimized samples exhibit excellent regenerability, (≥ 90% of their original value) even after 19 adsorption-desorption cycles showing a stable and durable CO₂ capture performance. This work paves the way for developing electrospun nanofiber membranes capable of efficient CO₂ adsorption.

1. Introduction

Currently, global warming is the biggest alarming threat to environment and public health. One of the fundamental contributing source toward global warming is greenhouse effect. There are a number of gases, which cause greenhouse effect that leads to the problem of global warming, however, among them CO₂ is the leading contributor. Massive CO₂ emissions not only lead to global warming but also result in rise of sea level and environmental pollution [1,2]. That is why it is being highly demanded to take immediate efforts to reduce CO₂ concentration in the atmosphere and limit further emission of CO₂ in the atmosphere [3]. Therefore, new materials, techniques and technologies are being explored for developing effective, economic and scalable CO₂ capture and storage (CCS) technologies [4].

Ideal materials for CCS need to have highly porous structure, good mechanical strength and flexibility, sustainable and environment friendly fabrication process, besides that they should have feasibility for easier surface functionalization [5]. In this regard, electrospun polymeric nanofibers successfully qualify most of the demands to be ideal materials for CCS [6]. Owing to their free-standing nature, tailorable physical characteristics, easy and economical fabrication process,

electrospun nanofibers are gaining increasing attention of scientists in the recent years [7]. Moreover, in order to maximize their CCS capacity and selectivity, electrospun nanofibers can be functionalized with certain additives such as amines. Nowadays, amine functionalized porous materials (such as silica, carbon, nylon etc.) are being extensively examined to explore their potential capabilities for CCS owing to their effective CO₂ capturing quality and easy application method [5,6,8]. A variety of various amine groups have been thoroughly examined for CO₂ adsorption, however, polyethyleneimine (PEI) has been proved to be the most significant group of amine family because of its high number of functional sites result in outstanding affinity for CO₂ [9]. Whereas, very little work has been reported about the use of TEA for CO₂ adsorption [10]. These amines can essentially be applied by simply impregnating porous substrate in polymeric amine solutions, in-situ polymerization or grafting them on surface of the porous materials [11].

In the present work, amine functionalized polystyrene/polyurethane (PS/PU) nanofiber composite membrane has been developed. PS is commonly used polymer fiber with very high level of hydrophobicity and easily tailorable porous structure, thus, it has been chosen as primary substrate/support, because high porosity and water

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repellency are two primary demands for CO₂ adsorption. PU is another commonly used elastomeric fiber with excellent mechanical characteristics, and has been employed to overcome poor mechanical strength of PS nanofibers in pristine state. As-synthesized composite membrane was functionalized with various amines by impregnation method, and effect of various amine groups on CO₂ adsorption capacity of synthesized membrane was examined.

2. Experimental section

2.1. Materials

Polystyrene (PS, Mw = 350,000 g mol⁻¹) was supplied from Wako Co., Ltd., Japan, and polyurethane (PU, Mw = 180,000 g mol⁻¹) was procured from BASF Polyurethane Specialties Co., Ltd., China. Tetrahydrofuran (THF), dimethylformamide (DMF), ethanol, polyethyleneimine (PEI, Mn = 10,000 and 60,000) and triethylamine (TEA, Mn = 101.19) were obtained from Aladdin Chemical Co., Ltd., China. All chemicals were of analytical grade and used without further purification.

2.2. Solution preparation and membrane fabrication

Homogenous solutions of PS (15 wt%) and PU (7 wt%) were prepared by dissolving them into a mixture of DMF/THF (1/1) individually under vigorous magnetic stirring overnight. Prepared solutions were then electrospun using DXES-3 electrospinning machine (SOF Nanotechnology Co., China) at the feeding rate of 1 mL/h under an applied voltage of 30 kV. Nanofiber membrane was collected on a metallic roller revolving at 20 cm distance from the feeding needles. Constant temperature (25 ± 2 °C) and relative humidity (45 ± 3%) were maintained throughout the fabrication process. Resultant nanofiber membranes were dried under vacuum for 4 h to remove any residual solvent.

2.3. Impregnation of PS/PU composite membrane

Amine solutions were prepared by dissolving PEI and TEA in ethanol individually under vigorous magnetic stirring until transparent solutions were achieved. Composite membranes were then immersed in transparent solutions for 1 h under continuous stirring. Later on, the solution along with membrane was transferred into a rotary evaporator (PV 10 Basic Plus D, Wilmington, NC) and ethanol was evaporated from the solution under vacuum at 60 °C for ~1 h and the resultant membrane was finally dried at 50 °C for 5 h under vacuum. Subsequent impregnated nanofiber membranes were tagged PS/PU-PEI_L-Y, PS/PU-PEI_H-Y and PS/PU-TEA-Y, where L and H represent low and high molecular weight of PEI, and Y corresponds to the amine weight ratio relative to the weight of pristine membrane.

All the synthesized membranes were impregnated with four different weight ratios of amine polymers, weight ratios of amine to the weight of pristine membrane were 1/4, 1/2, 1/3 and 1/1. Subsequent PEI_L, PEI_H and TEA impregnated membranes were tagged as PS/PU-PEI_L-1, PS/PU-PEI_L-2, PS/PU-PEI_L-3, PS/PU-PEI_L-4, PS/PU-PEI_H-1, PS/PU-PEI_H-2, PS/PU-PEI_H-3, PS/PU-PEI_H-4, PS/PU-TEA-1, PS/PU-TEA-2, PS/PU-TEA-3, and PS/PU-TEA-4, respectively.

2.4. Structural characterization

Fiber surface morphological analysis of synthesized composite membranes was carried out using scanning electron microscope (SEM, TESCAN VEGA 3, TESCAN Ltd., Czech Republic). Fourier transform infrared (FTIR) spectrometer (Nicolet iS10, USA) was employed to observe the presence of amine groups and CO₂ molecules in the functionalized composite nanofiber membranes. Physical structure was examined by using an automatic adsorption system (ASAP 2020,

Micromeritics Co., USA). XQ-1C tensile tester (Shanghai New Fiber Instrument Co., Ltd., China) was involved for examining the mechanical characteristics.

2.5. CO₂ adsorption

The CO₂ capture performance, cycle performance and CO₂/N₂ selectivity of amine-impregnated nanofiber composite membranes was investigated by thermogravimetric analyzer (TGA, TA Instruments model Q600, USA). A flow of 200 mL/min for N₂ as well as CO₂ was maintained during the process. A sample weighing ~10 mg of composite membrane was placed on microbalance alumina sample cell. In order to remove any existing volatile impurities, sample was first exposed to 105 °C in N₂ environment. The temperature was then adjusted to 40 °C and pure dry CO₂ gas was introduced for 60 min to evaluate the CO₂ adsorption performance. Additionally, the CO₂ adsorption performance of optimized sample was also observed at 50 °C and 60 °C to examine influence of temperature on CO₂ capture performance. CO₂ desorption was carried under nitrogen environment for 40 min at 105 °C. CO₂ adsorption capacity in mmol/g was calculated during adsorption process. Multiple adsorption/desorption cycles were carried out to evaluate recyclability of the optimized samples. Moreover, in order to investigate the feasibility of capturing CO₂ in practical conditions, selectivity of optimized samples against CO₂ and N₂ was analyzed. Same procedure was repeated for N₂ adsorption. The selectivity of CO₂/N₂ was defined as $S = q_1/q_2$, where q_1 and q_2 are the adsorption capacities of CO₂ and N₂ in the adsorption process, respectively.

3. Results and discussions

Scheme 1 illustrates the schematic demonstration of amine functionalized PS/PU nanofiber membrane development process. Development of composite nanofiber membrane was carried out in two critical steps: (1) Fabrication of porous PS/PU nanofiber composite membrane with rough surface morphology, (2) functionalization of synthesized nanofiber composite membrane with various amines. Porous PS/PU nanofiber composite membrane was fabricated via electrospinning technique using two solvents having different degree of volatility. Owing to the difference in volatile nature of the two solvents, solvent evaporation process intensified resulting in much faster evaporation of solvent and simultaneous interaction with external non-solvent species such as air and moisture resulted into the formation of porous fiber structure. Moreover, successful surface functionalization of the as-synthesized PS/PU nanofiber composite membrane was carried out in rotary evaporator using impregnation technique.

Fig. 1a presents the morphological analysis of electrospun pristine PS/PU nanofiber membrane. It could be seen that fabricated membrane offered randomly oriented 3D fibrous structure. Since two polymers were electrospun side by side, therefore, two different ranges of fibers in the membrane were witnessed. PS fibers were highly porous and relatively thicker in diameter and ranged 2–3 μm. Whereas, PU nanofibers offered smooth and solid cylindrical shaped structure with average fiber diameter in the range of ~800–950 nm in the resultant PS/PU nanofiber membrane (A low magnification SEM image used for measuring fiber diameter has been provided in Fig. S1). This variance in physical structure and fiber diameter could be attributed to the difference in molecular weight of two polymers and use of two solvents with varied degree of volatile nature.

Porosity and specific surface area are the two most important characteristic features, which determine the CO₂ adsorption capacity of the substrate. Therefore, the hierarchical pore structure of membranes was examined via N₂ adsorption-desorption isotherms at 77 K (Fig. 1b). Typical type IV isotherm with an apparent adsorption hysteresis loop as classified by IUPAC was seen [12]. It was observed that a series of typical physical adsorption behaviors including monolayer adsorption, multilayer adsorption and capillary condensation phenomenon

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