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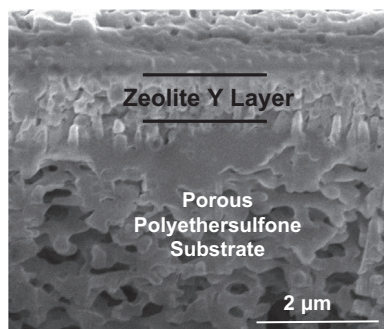
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Fabrication of zeolite/polymer multilayer composite membranes for carbon dioxide capture: Deposition of zeolite particles on polymer supports

Kartik Ramasubramanian^a, Michael A. Severance^b, Prabir K. Dutta^b, W.S. Winston Ho^{a,c,*}^a William G. Lowrie Department of Chemical and Biomolecular Engineering, The Ohio State University, 458 CBEC Building, 151 West Woodruff Avenue, Columbus, OH 43210-1350, USA^b Department of Chemistry and Biochemistry, The Ohio State University, 458 CBEC Building, 151 West Woodruff Avenue, Columbus, OH 43210-1350, USA^c Department of Materials Science and Engineering, The Ohio State University, 458 CBEC Building, 151 West Woodruff Avenue, Columbus, OH 43210-1350, USA

GRAPHICAL ABSTRACT



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ABSTRACT

Membranes, due to their smaller footprint and potentially lower energy consumption than the amine process, offer a promising route for post-combustion CO₂ capture. Zeolite Y based inorganic selective layers offer a favorable combination of CO₂ permeance and CO₂/N₂ selectivity, membrane properties crucial to the economics. For economic viability on large scale, we propose to use flexible and scalable polymer supports for inorganic selective layers. The work described in this paper developed a detailed protocol for depositing thin zeolite Y seed layers on polymer supports, the first step in the synthesis of a polycrystalline zeolite Y membrane. We also studied the effects of support surface morphology (pore size and surface porosity) on the quality of deposition and identified favorable supports for the deposition. Two different zeolite Y particles with nominal sizes of 200 nm and 40 nm were investigated. To obtain a complete coverage of zeolite particles on the support surface with minimum defects and in a reproducible manner, a vacuum-assisted dip-coating technique was developed. Images obtained using both digital camera and optical microscope showed the presence of color patterns on the deposited surface which suggested that the coverage was complete. Electron microscopy revealed that the particle packing was dense with some drying cracks. Layer thickness with the larger zeolite Y particles was close to 1 μm while that with the smaller particles was reduced to less than 0.5 μm. In order to reduce drying cracks for layers with smaller zeolite Y particles, thickness was reduced by lowering the dispersion concentration. Transport measurement was used as an additional technique to characterize these layers.

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* Corresponding author at: The Ohio State University, 458 CBEC Building, 151 West Woodruff Avenue, Columbus, OH 43210-1350, USA. Fax: +1 (614) 292 3769.

E-mail addresses: ramasubramanian.8@buckeyemail.osu.edu (K. Ramasubramanian), severance.18@buckeyemail.osu.edu (M.A. Severance), dutta@chemistry.ohio-state.edu (P.K. Dutta), ho.192@osu.edu (W.S.W. Ho).

Nomenclature

d	diameter (m)
J	steady-state flux (mole/m ² /s)
K	permeability of the porous media (m ²)
l	selective layer thickness
P	mixed-gas permeance (GPU, 1 GPU = 10 ⁻⁶ cm ³ (STP)/cm ² /s/cm-Hg; 3000 GPU = 10 ⁻⁶ mole/m ² /s/Pa = 10 ⁻¹ mole/m ² /s/bar)
p	pressure (bar)
Q	liquid flux (m ³ /m ² /s)
x	mole fraction on the feed side
y	mole fraction on the permeate or sweep side
z	position variable in the direction of flow through support (m)

Greek letters

α	membrane selectivity
μ	coating liquid viscosity (Pa·s)

Subscripts

c	capillary suction
f	feed side
i, j	species
l	liquid
p	pore
s	sweep or permeate side

1. Introduction

High performance membranes or selective barriers based on novel materials offer one of the few potential means of meeting the stringent economics of post-combustion CO₂ capture (PCC). For CO₂/N₂ separation in PCC, CO₂ permeates selectively through the membrane. In this context, it is useful to understand three quantities, CO₂ flux (J_i), CO₂ permeance (P_i) and CO₂/N₂ selectivity (α_{ij}) shown by Eqs. (1) and (2).

$$J_i = P_i(p_f \times x_i - p_s \times y_i) \quad (1)$$

$$\alpha_{ij} = \frac{P_i}{P_j} \quad (2)$$

where p_f is the feed pressure, p_s the permeate or sweep pressure (if a sweep gas is used to provide driving force), x the feed side mole fraction, and y the permeate side mole fraction. High P_{CO_2} (>3000 GPU) and high $\alpha_{\text{CO}_2/\text{N}_2}$ (>100) selectivity are important for economic viability as pointed out by recent studies [1–3].

Membranes usually consist of a thin selective layer (~100 nm to 1 μm in thickness) supported on a porous and relatively thick support for mechanical stability [4–7]. Polymeric membranes are flexible and scalable in the form of hollow-fiber and spiral-wound modules having high surface area/volume ratios. Although some polymer membranes based on polar ethylene oxide groups in the backbone have demonstrated high CO₂ permeance (>2000 GPU) [2,4], their performance is usually limited by the Robeson's selectivity–permeability trade-off [8]. Inorganic membranes on the other hand can potentially provide a higher selectivity at a comparable CO₂ permeance [5,9,10] but are plagued by irreproducibility due to defects and involve the use of thick, brittle, less scalable, less compact and more expensive inorganic supports [11,12]. The support cost issues, irreproducibility of synthesis and long synthesis times have hindered the development of inorganic membranes that barring a few examples [12], there has been minimal commercialization.

As a potential solution to the above problem and to harness the performance benefits of inorganic membranes, we studied appropriate porous polymer membranes as supports for promising zeolite-based inorganic layers. These porous membranes, generally made by phase inversion, have traditionally been used as selective layers in ultrafiltration, microfiltration and also as polymer membrane supports. Common polymers are polysulfone (PSf), polyethersulfone (PES), polyacrylonitrile, cellulose acetate, nylon, etc. Since such supports have not yet been studied for inorganic

selective layers or even inorganic particulate layers, our approach was to begin by developing a scalable method for depositing stable zeolite Y particulate layers on them with minimum defects. The specific goals of this work were as follows: (1) study and identify different support materials and support morphologies that will allow us to make defect-free zeolite Y particulate layers and (2) focus on fabricating thin zeolite Y seed layers of less than 1 μm in thickness. Subsequent growth of the seed layer into a selective polycrystalline zeolite membrane is not a part of this work. We recognize that the long times it takes for the hydrothermal synthesis of zeolites makes it incompatible with growing membranes on polymer supports, an issue we have addressed recently [13].

2. Background and rationale

2.1. Zeolite Y as membrane material

Commonly studied physical solid adsorbents for CO₂ include different types of zeolites and activated carbons while alkali earth metal oxides, supported amines, and hydrotalcite are examples of chemisorbents [14–18]. Physical adsorbents make use of van der Waals forces between gas molecules and adsorbent surface while chemisorbents make use of a specific chemical reaction, for instance, an acid–base type reaction in case of CO₂ and metal oxides or amines. Physical adsorbents generally show lower adsorption capacities but more favorable regeneration properties than chemisorbents. They also show acceptable to excellent adsorption and desorption kinetics. In case of post-combustion flue gas CO₂ capture, both CO₂ partial pressure (0.1–0.2 atm) and temperature (57 °C) are relatively low. Under these conditions, zeolites show an attractive trade-off between properties among physical adsorbents [14,15].

Zeolites are crystalline aluminosilicates with well-defined microporosity that has enabled their applications in a wide variety of fields like catalysis, detergency, and chemical sensing in addition to separations [19]. Depending upon the Si/Al ratio and the number of neutralizing cations inside the pores, zeolites can have different adsorptive properties, with the hydrophobicity increasing as the Si content increases [19,20]. The fixed pores of zeolites can also distinguish between molecules of different shapes and sizes. Traditionally, zeolites have been used in adsorption processes. Membranes are interesting from the point of view of using these materials in a continuous separation process.

Molecular permeation through a zeolite pore depends on the loading (determined by thermodynamics) in addition to the

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