Enhanced CO$_2$/N$_2$ Separation of Mixed-Matrix Membranes Containing Hierarchical UiO-66 Synthesized from Hard Templates

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ABSTRACT: The metal–organic framework (MOF) has attracted widespread attention due to its high porosity, adjustable framework, and tailorability. However, a high diffusion barrier hinders the fast permeation of guest molecules in the framework. The introduction of a hierarchy in the MOF represents an effective protocol for reducing guest diffusion resistance. In this work, a polystyrene sphere was utilized as a hard template to prepare hierarchical UiO-66. Compared with bulk UiO-66, UiO-66 contained a hierarchical pore structure, which established fast diffusion pathways for guest molecules; moreover, a hard template facilitated more sufficient exposure of coordinatively unsaturated Zr–oxo sites in the framework, which was conducive to preferential CO$_2$ adsorption. Prepared MMMs with a loading of 10 wt % H-Uio-66 exhibited an ideal CO$_2$/N$_2$ selectivity of 43.48, with CO$_2$ permeability reaching 809.26 barrer, which well exceeded the 2008 Robeson upper bound and the majority of MOF-based MMMs, showing promising prospects for efficient CO$_2$ capture from flue gas.

INTRODUCTION

Excessive CO$_2$ emission represents the main cause of global warming so that energy-efficient CO$_2$ capture has become indispensable. Membrane separation has been considered to be a promising technique for energy-efficient CO$_2$ capture. Among them, mixed-matrix membranes (MMMs), which combine the advantages of polymer membranes and inorganic fillers, have been developed for energy-efficient CO$_2$ capture. Ascribed to its high porosity, adjustable framework, and tailorable functionality to elucidate the interplay between the functional group types and CO$_2$ adsorption capacities. Experimental results indicated that its CO$_2$ adsorption capacity was comparable with those of other benchmark MOF materials like MOF-74 and Cu-BTC. Cho et al. investigated the impact of ligand defects in the UiO-66 framework on the CO$_2$ separation performance of obtained poly(ethylene glycol) diacrylate (PEGDA) MMMs. Under optimized conditions, the CO$_2$ permeability was 4 times higher than that of the pure PEGDA membrane, with comparable CO$_2$/N$_2$ selectivity.

It should be noted that the pore sizes of MOF materials commonly fall in the microregime (<2 nm), thereby exerting higher diffusion resistance. It therefore became indispensable to establish multiscale facilitated CO$_2$ diffusion pathways. Hierarchical MOF (H-MOF), which is composed of interconnected multiscale pore channels, offers a possible solution to this issue.

Zhou et al. employed a metal–ligand-fragment coassembly strategy to introduce additional thermal/chemical stability and strong affinity toward CO$_2$. Usman et al. synthesized versatile UiO-66 with distinct functionality to elucidate the interplay between the functional group types and CO$_2$ adsorption capacities. Experimental results indicated that its CO$_2$ adsorption capacity was comparable with those of other benchmark MOF materials like MOF-74 and Cu-BTC. Cho et al. investigated the impact of ligand defects in the UiO-66 framework on the CO$_2$ separation performance of obtained poly(ethylene glycol) diacrylate (PEGDA) MMMs. Under optimized conditions, the CO$_2$ permeability was 4 times higher than that of the pure PEGDA membrane, with comparable CO$_2$/N$_2$ selectivity.

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mesopores in (N)-PCN-125, showing a higher CO₂ adsorption capacity than “perfect” PCN-125. Gu et al.⁰ constructed Zr-based H-MOF through a salt-aided nanoeulsion-guided assembly. The obtained dendritic mesoporous UiO-66 was exemplified to provide an ideal platform for a coenzyme-dependent cascade enzymatic reaction, realizing the effective heterogeneous regeneration of expensive nicotinamide adenine dinucleotide.

It should be noted that, in spite of the tremendous advances that have been achieved, the facile preparation of H-MOFs remains a grand challenge to date for the following reasons: (1) The stability of H-MOF is lower compared with that of its pristine counterpart. (2) The crystallinity and framework integrity of H-MOF are commonly lower than those of pristine MOF, posing a great challenge to its size-sieving ability. (3) The process is complex due to the involvement of special additives such as amphiphilic molecules and modulators.

Both soft- and hard-template methods have been used in preparing H-MOF materials. In terms of the soft-template method, its compatibility with a solvent is essential for the formation of stable micellar templates with specific sizes and shapes (e.g., spheric and 1D cylindrical pores). Nonetheless, common surfactants tend to dissolve rather than assemble into micelles in organic solvents where MOF materials are usually synthesized, resulting in the restricted selection of solvents. In this regard, the hard-template method is more effective in synthesizing H-MOF materials. It should be noted that, in spite of many efforts devoted to suppression of the nucleation and growth of MOF materials, using a hard template may lead to the formation of undesired precipitates in the bulk solution because of weak interactions between the MOF precursor and template; in addition, harsh post-treatment steps, such as acidic or basic etching, are often required to remove the hard template, resulting in the partial loss of structural integrity. In this study, we pioneered the preparation of hierarchical Uio-66 (H-Uio-66) as a filler of the Pebax 2533 membrane for efficient CO₂ capture (Figure 1). Polystyrene (PS) has been widely used as the hard template of hierarchical porous materials like zeolites. Because PS is highly uniform in size, the textural properties of H-Uio-66 can be accurately controlled; moreover, because PS is soluble in N,N-dimethylformamide, the removal of hard templates conventionally conducted under acidic or alkaline conditions could be effectively avoided, which is advantageous to maintaining the structural integrity of Uio-66. Compared with Pebax 1657, Pebax 2533 had better permeability at the expense of lower selectivity, which, however, could be overcome by adding H-Uio-66 with CO₂-facilitated transport pathways. In addition, regulation of the Uio-66 pore microenvironment has been proven to have a profound effect on its adsorption performance. Our research indicated that missing-linker defects were simultaneously introduced in the Uio-66 framework during templating, resulting in unprecedented CO₂ permeability (809.26 barrer) and CO₂/N₂ selectivity (43.48) for H-Uio-66 MMMs, showing great promise for practical CO₂ capture from flue gas.

### EXPERIMENTAL SECTION

Reagents and Materials. All reagents used in this work were purchased from commercial vendors and used as received, unless otherwise noted. Potassium persulfate (K₂S₂O₈, 99%, Macklin), styrene (99%, Macklin), sodium hydroxide pellets (NaOH, 96%, Macklin), poly(vinylpyrrolidone) (PVP, Mₐ = 10000/58000/220000, 99%, Macklin), zirconium tetrachloride (ZrCl₄, 99.9%, Macklin), 2-aminothephrthalic acid (NH₂-BDC, 99%, Macklin), Pebax 2533 [containing 80 wt % poly(tetramethylene oxide) (PTMO) and 20 wt % polyamide-12 (PA-12), Arkema], formic acid (FA, 99.5%, Tianjin Kemiu), N,N-dimethylformamide (DMF, C₅H₈NO, 99.8%, Tianjin Kemiu), N,N-dimethylacetamide (DMAC, C₅H₁₀NO, 99%, Macklin), methanol (CH₃OH, 99.5%, Tianjin Kemiu), ethanol (C₂H₅OH, 99.7%, Tianjin Kemiu), and isopropyl alcohol (C₃H₇OH, 99.5%, Tianjin Kemiu) were used as received without further purification. Porous α-Al₅O₃ disks with a pore size of 70 nm, a diameter of 18 mm, and a thickness of 1 mm were purchased from Fraunhofer IKT, Germany.

Preparation of PS Nanospheres. Monodispersed PS nanospheres with different sizes (PS-a, ~250 nm; PS-b, ~150 nm; PS-c, ~80 nm) were synthesized according to reported literature with slight modification. Taking the synthesis of PS-a as an example, 13 g of styrene was first washed thoroughly with 5 mL of a 10 wt % NaOH solution and deionized (DI) water to remove the stabilizer. Subsequently, washed styrene was added to a 250 mL round-bottom flask containing 0.5 g of PVP (Mₐ = 10000) dissolved in 115 mL of water. The mixture
was fluxed at 95 °C under magnetic stirring for 30 min. In the next step, 15 mL of an aqueous solution containing 0.1 g of K$_2$SO$_4$ was added to the flask to initiate the polymerization reaction. After being stirred for 24 h at 95 °C, the mixture was cooled to room temperature. The obtained milk-like product was sequentially centrifuged, washed with DI water five times, and ultimately dried at 50 °C overnight before further use. PS-b and PS-c were prepared in the same manner except that the molecular weights of PVP were 58000 and 220000, respectively.

**Preparation of H-UiO-66.** H-UiO-66 was prepared through in situ nanocasting. H-UiO-S(x,y) was named H-UiO-S(x,y), with x and y denoting the sizes of the templated pores and crystals, respectively. For the synthesis of H-UiO-S(250,350), 0.059 g of ZrCl$_4$ was dissolved in a solution consisting of 3.5 mL of FA and 8 mL of H$_2$O. Afterward, 3 g of PS-a was ground and added to the precursor solution, followed by ultrasonication for 20 min. In the next step, 20 mL of ethanol containing 0.045 g of NH$_2$BDC was added to the precursor solution and maintained at room temperature under stirring for 48 h. The obtained white powders were centrifuged, dried in air, and immersed into DMF for 5 days to remove PS. Finally, the obtained yellow powders were washed with DMF and ethanol three times and ultimately dried at 70 °C overnight before further use. PS nanospheres with different sizes were employed to prepare H-UiO-66 with varying template size using the same procedure as that for H-UiO-S(250,350). The obtained products were named H-UiO-S(150,500) and H-UiO-S(90,650), respectively.

**Synthesis of Bulk UiO-66 Crystals.** For comparison, bulk UiO-66 crystals (denoted as B-UiO-66) was synthesized with a similar method except in the absence of a PS template in this process.

**Fabrication of UiO-66 MMMs.** H-UiO-66 MMMs were named H-UiO-M(x,y), with x and y denoting the sizes of the templated pores and H-UiO-66 filler loading. The H-UiO-66 filler loading is defined in the following equation:

$$\text{H-UiO-S loading (wt %)} = \frac{m_{\text{H-UiO-S}}}{m_{\text{Pebax}} + m_{\text{H-UiO-S}}} \times 100\%$$

H-UiO-M was fabricated by spin-coating, followed by solvent evaporation. For the synthesis of H-UiO-M(250,10 wt %), 0.028 g of H-UiO-S(250,350) was added to 4.75 g of DMAc with sonication for 2 h to obtain a homogeneous H-UiO-S suspension. Subsequently, 0.25 g of Pebax 2533 particles was slowly added to the solution under stirring at 70 °C for 2 h. MMMs were fabricated by spin-coating the mixed solution on a porous α-Al$_2$O$_3$ support at 3000 rpm for 60 s. Finally, the obtained H-UiO-M(250,10 wt %) was dried at 70 °C overnight to remove residual solvents. The preparation of B-UiO-66 MMMs was the same as that of H-UiO-M MMMs, except that B-UiO-66 was used as a filler. The prepared sample is denoted as B-UiO-M(x), where x represents the filler loading.

**Gas Permeation Test.** H-UiO-66 and B-UiO-66 MMMs were placed in a membrane module and sealed with O-rings. The volumetric flow rates of both the feed and permeate sides were kept at 50 mL min$^{-1}$, while the permeate side was swept with helium. The pressure difference in the feed and permeate sides was kept at 1 bar. A calibrated gas chromatograph (7890B, Agilent) was used to analyze the composition of the permeate mixed gases. The separation factor (SF) $\alpha_{A/B}$ was calculated from the quotient of the molar fractions of the components (A and B) in the feed and permeate sides using the following equation:

$$\alpha_{A/B} = \frac{X_{A,\text{perm}}/X_{B,\text{perm}}}{X_{A,\text{feed}}/X_{B,\text{feed}}}$$

The ideal selectivity, i.e., $\alpha_{A/B}$ (ideal), was defined as the single gas permeance ratio of gases A and B:

$$\alpha_{A/B}(\text{ideal}) = \frac{P_A(\text{permeance})}{P_B(\text{permeance})}$$

The gas permeability ($P_i$ mol·m$^{-1}$·s$^{-1}$·Pa$^{-1}$) was expressed as the flux of component i per unit area ($J_i$ mol·m$^{-2}$·s$^{-1}$) multiplied by the effective thickness of the MOF membrane ($L_i$ m) and divided by its driving force, i.e., partial pressure differences ($\Delta P_i$ Pa) between the upstream and downstream sides through the membrane:

$$P(\text{permeability}) = P_i(\text{permeance}) \times L = \frac{J_i}{\Delta P_i}$$

**Characterization.** X-ray diffraction (XRD) patterns were collected on a Rigaku SmartLab diffractometer with Cu Kα radiation ($\lambda = 0.15418$ nm) at 45 kV and 200 mA. Thermogravimetric analysis (TGA) results were obtained on a NETZSCH (TG 209) thermal analyzer under an air or N$_2$ atmosphere. Scanning electron microscopy (SEM) images were evaluated by a FlexSEM 1000 instrument (Hitachi Co.) at accelerating voltages of 10 and 15 kV. Physical adsorption analysis was obtained on a Mike ASAP 2020 Plus analyzer.

**RESULTS AND DISCUSSION**

**Preparation of the H-MOF Filler.** PS nanoparticles with sizes of 80, 150, and 250 nm could be obtained by varying the

![Figure 2. Morphology of PS and H-UiO-66 fillers. SEM images of PS: (a) 250 nm; (b) 150 nm; (c) 80 nm. SEM and TEM images of H-UiO-66: (d and h) H-UiO-S(250,350); (e and i) H-UiO-S(150,500); (f and i) H-UiO-S(80,650).](https://doi.org/10.1021/acs.iecr.4c01130)
molecular weight of the added emulsifier (PVP). Fourier transform infrared (FT-IR) spectra (Figure 3b) showed vibration peaks of $-\text{C}==\text{C}-$ on the aromatic ring at 1492 and 1452 cm$^{-1}$ and vibration peaks of $=\text{C}==\text{H}$ at 758 and 700 cm$^{-1}$. As depicted in Figure 2a–c, all PS nanoparticles possessed a uniform size and high sphericity, which is beneficial for creating uniform macropores in Zr-MOF crystals. Our results showed that the molecular weight of the emulsifier had a significant impact on the particle size of the obtained PS nanoparticles. With increasing molecular weight of the added PVP, the size of the obtained PS nanoparticles became smaller. When the molecular weight of PVP was increased to 220000, the size of the obtained PS nanoparticles was reduced to 80 nm.

H-UiO-66 was prepared through in situ nanocasting. H-UiO-66 was named H-UiO-S($x$,$y$), with $x$ and $y$ denoting the sizes of the templated pores and crystals, respectively. Our study revealed that $\pi-\pi$ interaction between the precursor solution and PS surface enabled preferential UiO-66 nucleation and crystallization on the PS surface, resulting in the formation of UiO-66@PS. With increasing molecular weight of the added PVP, the size of the obtained PS nanoparticles became smaller. When the molecular weight of PVP was increased to 220000, the size of the obtained PS nanoparticles was reduced to 80 nm.

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Figure 3. Textural characterizations of H-UiO-66 fillers: (a) XRD patterns; (b) FT-IR spectra of H-UiO-66, B-UiO-66, and PS; (c) N$_2$ adsorption/desorption isotherms (77 K) of H-UiO-66 and B-UiO-66; (d) TGA curves of H-UiO-S(250,350).
The Brunauer–Emmett–Teller surface areas of H-UiO-S(250,350), H-UiO-S(150,500), and H-UiO-S(80,650) were calculated to be 723, 607, and 653 m$^2$·g$^{-1}$, respectively, which were close to that of B-UiO-66 (684 m$^2$·g$^{-1}$), demonstrating that the structural integrity of UiO-66 was well maintained during templating.

Of particular note, we found that the introduction of PS nanoparticles during the nucleation and crystallization of H-UiO-66 led to more missing-linker defects in the framework. The introduction of linker deficiencies would further expose coordinatively unsaturated sites, enhancing the affinity interaction between H-UiO-66 and CO$_2$; on the other hand, the absence of linkers would enlarge the micropore size of H-UiO-66, which is conducive to fast permeation of CO$_2$ in the membrane. TGA (Figure 3d) was further conducted to evaluate linker vacancies based on the method developed by Lillerud et al. (shown in SI-4 and SI-5). Our results indicated that the missing-linker number per the Zr$_6$O$_4$(OH)$_4$ node of H-UiO-S(250,350) was 2.31, which was 4.8 times higher than that of B-UiO-66 (0.48). Simultaneously, the DFT model (Figure S2) revealed that, in addition to native 0.57-nm-sized micropores, the obtained H-UiO-66 crystals further exhibited micropores ranging between 0.8 and 1.5 nm, which further confirmed the micro/macroporous hierarchical structure. Based on previous reports, increasing both the missing-linker number and micropore size was beneficial for enhancing the CO$_2$ separation efficiency. 29,30 The gas adsorption properties of H-UiO-S(250,350) were investigated. Figure 5e depicts the CO$_2$ and N$_2$ adsorption isotherms at 25 °C and 1 bar. It was observed that the adsorption capacity of CO$_2$ on H-UiO-S(250,350) reached 48.9 cm$^3$·g$^{-1}$ STP, while the N$_2$ adsorption capacity was only 4.1 cm$^3$·g$^{-1}$ STP. Furthermore, the ideal adsorbed solution theory (IAST) adsorption selectivity was calculated, and it was found that the CO$_2$/N$_2$ (50:50) IAST selectivity of H-UiO-S(250,350) reached 23.16 under ambient conditions, which was higher than that of B-UiO-66 (20.29).

Preparation of H-MOF MMMs. As mentioned in previous work, the introduction of a hierarchy in the UiO-66 framework not only opens up a rapid diffusion pathway for CO$_2$ but also renders more sufficient exposure of coordinatively unsaturated open metal sites toward higher CO$_2$/N$_2$ adsorption selectivity, making it an ideal filler for a CO$_2$-philic Pebax 2533 membrane. The area of the prepared H-UiO-66/Pebax 2533 MMMs was 2.5 cm$^2$.

Initially, the influence of the H-UiO-66 content on the membrane morphology was investigated. As shown in Figure 4a–c, the surface of the obtained MMMs appeared uniform and smooth; moreover, the H-UiO-66 filler was uniformly distributed in the Pebax 2533 matrix with no agglomeration at a loading of no higher than 10 wt % (Figure S5); however, severe agglomeration (Figure 4d) inevitably occurred upon further increasing the filler loading to 12 wt %. Cross-sectional SEM images (Figures 4e and S6) indicated that the thickness of H-UiO-M(250,350) reached 4.2 μm with a clearly visible boundary between the membrane and underlying porous α-Al$_2$O$_3$ substrate. Relevant XRD patterns (Figure 4f) showed...
behaviors of the obtained H-UiO-66 MMMs were evaluated in the membrane.

In addition, SEM and TEM images of the mixture prepared from H-UiO-66 crystals and Pebax 2533 polymer are shown in S1-7 (Figure S7a,d). Our results indicated that H-UiO-66 crystals in the Pebax 2533/DMAc mixture had a high affinity with a polymer matrix (Figure S7b,c). TEM images (Figure S7e,f) showed that H-UiO-66 seeds were coated with polymers, and Pebax 2533 had penetrated into their macro-pores.

Gas Permeation Test. Initially, the gas permeation behaviors of the obtained H-UiO-66 MMMs were evaluated with a Wicke–Kallenbach apparatus under ambient conditions. The volumetric flow rates of H$_2$, CO$_2$, N$_2$, and CH$_4$ through H-UiO-M(250,10 wt %), B-UiO-66 MMMs (10 wt %), and the pure Pebax 2533 membrane were first evaluated. As shown in Figure 5a, single gases exhibited similar permeation behavior in all of the above membranes. Among them, CO$_2$ exhibited the highest permeability. A more detailed comparison of the CO$_2$ permeability in H-UiO-M(250,10 wt %), B-UiO-M(10 wt %), and a pure polymer membrane implied that the CO$_2$ permeability of B-UiO-M(10 wt %) increased by 95.64% (from 330.76 to 647.09 barrer) because UiO-66 had a higher affinity with CO$_2$. In comparison, the CO$_2$ permeability of H-UiO-M(250,10 wt %) further increased to 828.49 barrer, which was 2.5 times higher than that of the pure Pebax 2533 membrane. Obviously, the introduction of a hierarchy in the H-UiO-66 framework significantly contributed to fast permeation of CO$_2$ in the membrane.

Subsequently, the separation performances of equimolar CO$_2$/N$_2$ gas mixtures on H-Ui-66 MMMs, B-UiO-66 MMMs, and the pure Pebax-2533 membrane were evaluated. As shown in Figure 5b,c and Table S12, the pure Pebax membrane exhibited a CO$_2$/N$_2$ SF of 21.64 and a CO$_2$ permeability of 339.42 barrer, while for B-UiO-M(10 wt %), its CO$_2$/N$_2$ SF reached 30.69 with a CO$_2$ permeability of 659.41 Barrer, which were 41.82% and 94.28% higher than those of the pure Pebax 2533 membrane. In the case of H-UiO-M(250,10 wt %), the CO$_2$/N$_2$ SF and CO$_2$ permeability further increased to 43.48 and 809.26 barrer, respectively, surpassing the 2008 Robeson upper bound for practical CO$_2$ capture from flue gas. Obviously, compared with B-UiO-66 MMMs, the CO$_2$/N$_2$ selectivity and CO$_2$ permeability of H-UiO-M(250,10 wt %) were positively promoted by the improvement of the missing-linker defects number and the construction of a hierarchical structure, respectively. In addition, performance tests were performed under conditions simulating flue gas (V$_{CO_2}$/V$_{N_2}$ = 15:85). The SF of H-UiO-M(10 wt %) reached 28.67 with a CO$_2$ permeability of 163.69 barrer, while the SF of the pure Pebax polymer membrane was reduced to 6.79 with a CO$_2$ permeability of 113.50 barrer. Therefore, H-UiO-M (10 wt %) maintained a superior performance in terms of flue gas separation. Ding et al. concluded that increasing the membrane selectivity was beneficial for reducing the investment cost and CO$_2$ avoidance cost, while increasing the CO$_2$ permeability further improved the separation efficiency and reduced the operation cost. Therefore, H-UiO-M(250,10 wt %) showed promising potential for CO$_2$ capture from flue gas.

Finally, the effect of filler loading on the CO$_2$ permeability and the CO$_2$/N$_2$ selectivity of the obtained MMMs was
investigated (Figure 5d). When the H-Uio-S(250,350) loading was increased from 0 to 10 wt %, the CO₂ permeability significantly increased (from 339.42 to 809.26 barrer), in contrast to only a slight increment of the N₂ permeability (from 15.70 to 19.20 barrer), resulting in a remarkably enhanced CO₂/N₂ SF (from 21.64 to 43.48). However, further increasing the H-Uio-S(250,350) loading to 12 wt % led to undesired filler agglomeration and enlarged interfacial defects in the membrane, resulting in a sharply reduced CO₂/N₂ SF of H-Uio-M(250,12 wt %).

CONCLUSIONS
In this study, we used a hard-template etching strategy to prepare H-Uio-66 with different macropore sizes. π−π stacking between NH₂-BDC ligands in the UiO-66 framework and PS templates was proven to be the key factor for obtaining Uio-66-PS composites, followed by the formation of H-Uio-66 after DMF etching; moreover, hard templates facilitated more sufficient exposure of coordinatively unsaturated Zr₆-oxo sites in the framework, providing CO₂-facilitated and low-resistant diffusion pathways for H-Uio-66-based MMMs. Gas permeation results showed that the CO₂/N₂ separation performance of the obtained MMMs increased with increasing H-Uio-66 loading and reached a plateau at a H-Uio-66 loading of 10 wt %, which well exceeded the 2008 Robeson upper bound. The above protocol is anticipated to bring new insights to practical applications in CO₂ separation from flue gas.

ASSOCIATED CONTENT
Supporting Information
The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acs.iecr.4c01130.

Additional experimental details, gas permeation test details, detailed characterizations of H-Uio-66 seeds and H-Uio-66 MMMs, and the detailed gas separation performance of H-Uio-66 MMMs (PDF)

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