Fabrication of highly (110)-Oriented ZIF-8 membrane at low temperature using nanosheet seed layer

Chenhuan Zhang, Jiahui Yan, Taotao Ji, Dongying Du, Yanwei Sun, Liangliang Liu, Xiongfu Zhang, Yi Liu

State Key Laboratory of Fine Chemicals, PSU-DUT Joint Centre for Energy Research, School of Chemical Engineering, Dalian University of Technology, Ganjingzi District, Dalian, 116024, China

Abstract

Preferred orientation represents a main concern for microstructure control and performance optimization of MOF membranes. To date, it remains a challenging task to precisely control preferred orientation of ZIF-8 membrane. Herein, we prepared highly (110)-oriented ZIF-8 membrane by oriented secondary growth. Among various elements, preparation of uniform ZIF-8 nanosheet seeds through thermal conversion of identically shaped ZIF-L precursors, spin-coating-assisted oriented deposition of (110)-oriented ZIF-8 seed layer, and controlled in-plane secondary growth at low temperature were found crucial for obtaining well-intergrown (110)-oriented ZIF-8 membranes. To date, it remains a challenging task to precisely control preferred orientation of ZIF-8 membrane. Herein, we prepared highly (110)-oriented ZIF-8 membrane by oriented secondary growth. Among various elements, preparation of uniform ZIF-8 nanosheet seeds through thermal conversion of identically shaped ZIF-L precursors, spin-coating-assisted oriented deposition of (110)-oriented ZIF-8 seed layer, and controlled in-plane secondary growth at low temperature were found crucial for obtaining well-intergrown (110)-oriented ZIF-8 membrane whose $H_2/CO_2$ ideal selectivity exceeded $H_2/N_2$ and $H_2/CH_4$ under ambient conditions, owing to preferential $CO_2$ adsorption capacity of not only PEI but also ZIF-8 nanosheets compared with their bulk counterparts. Our research highlighted the significance of preferred orientation regulation in tailoring the gas permeation behavior of MOF membranes.

1. Introduction

Membrane-based technology has offered unexampled opportunities for efficient separation [1,2] considering its advantages over other separation technologies like low energy consumption, nearly-zero pollutant discharge, low capital cost, small footprint, and easy operation. Metal-organic framework (MOF) [3-6], as a burgeoning class of hybrid material comprising metal ions/clusters bridged by organic linkers, has been considered as ideal membrane candidates for separation because of its adjustable composition and structure, tailorable pore size, and diverse functionality. Among them, ZIF-8 membrane [7,8] has aroused widespread attention for the potential applications in hydrogen purification and olefin/paraffin separation owing to its appropriate pore size, exceptional chemical stability, thermal stability, and relative ease of fabrication.

In recent decades, noticeable development has been made in the preparation of high quality ZIF-8 membranes enabling efficient gas separation. For instance, Xing and Pan et al. prepared PDMS layer-coated ZIF-8 membranes on commercial ceramic tubes to realize efficient $C_2H_6/C_3H_8$ separation under high pressure [9]. The PDMS layer effectively blocked the inter-crystalline defects and suppressed lattice flexibility of the ZIF-8 membrane, resulting in significantly improved separation factor for $C_3H_8/C_2H_6$ gas mixture at the expense of a slight loss in $C_2H_6$ permeance. Yao et al. prepared ZIF-8@CNF composite membrane with excellent $CO_2/N_2$ and $CO_2/CH_4$ selectivity through combining in-situ growth with vacuum filtration [10]. He et al. combined ZIF-8@GO flake-like composite fillers with the ultrasound-assisted pre-Zn(II)-doping strategy to prepare Pebax-based MMMs showing concurrently enhanced $CO_2$ permeability and $CO_2/N_2$ selectivity [11]. Wang et al. obtained alumina hollow fiber-supported ZIF-8 membrane exhibiting superior $H_2/N_2$ selectivity by secondary growth. Maintaining appropriate sodium formate content and using thick-walled Teflon-liner were found crucial in warranting the well-intergrowth of relatively thin ZIF-8 membranes [12]. Wang et al. proposed an in-situ current-driven synthesis approach [13,14] to prepare ZIF-8 membrane with suppressed lattice flexibility and enhanced $C_3H_8/C_2H_6$ selectivity. Agrawal et al. developed a rapid heat treatment (RHT) strategy [15] to increase the lattice stiffness of ZIF-8 membrane which exhibited remarkably enhanced selectivity towards $CO_2/CH_4$ and $CO_2/N_2$ gas pairs. Jeong et al. prepared ZIF-7-8 membrane via...
corresponding mixed ligands [16], which led to effective pore aperture reduction and separation performance improvement. Tsapatsis et al. [17] proposed a vapor-phase-ligand-treatment (VPLT) strategy to partially replace 2-methylimidazole (Hmim) ligands with 2-amino-benzimidazole (2abIm), which concurrently reduced the effective pore aperture and facilitated the preferential adsorption of CO$_2$, resulting in improved CO$_2$/N$_2$ and CO$_2$/CH$_4$ selectivity.

It is worth noting, however, majority of previous studies mainly focused on tailoring structural properties (e.g., framework flexibility) of ZIF-8 membranes on a microscopic scale. In contrast, precise microstructural control (like preferred orientation) of ZIF-8 membranes remains largely unexplored to date except Caro’s early work on the preparation of preferentially (100)-oriented ZIF-8 membrane [18]. More recently, Eddaoudi et al. reported the preparation of preferentially (110)-oriented, ultrathin ZIF-8 membrane showing unprecedented and unique CH$_4$–n–C$_4$H$_{10}$ mixture permeation behavior via the liquid-phase epitaxy (LPE) method [19]. In the same manner, Valadez Sánchez et al. prepared monolithic (110)-oriented ZIF-8 SURMOF membrane support on Au thin layer-modified porous α-Al$_2$O$_3$ substrate via LPE layer-by-layer deposition. Prepared ZIF-8 membrane showed certain selectivity towards ethene/ethane gas mixture [20]. The preferred orientation of molecular sieve membranes has been proven to exert profound effects on their separation performances due to ordered alignment of pore channels, decrease of intercrystalline defects and therefore, considerable reduction in diffusion resistance [4,21–24]. Therefore, it is meaningful to prepare highly oriented ZIF-8 membranes to achieve unique permeation properties (Fig. S1). Compared with in situ growth protocol [25,26], secondary growth enabled more accurate regulation over preferred orientation [22,27,28] due to the effective separation of nucleation and epitaxial growth processes.

Among various factors affecting microstructure and functionality of MOF membranes, MOF seeds have proven to be especially important. So far as preferred orientation control is concerned, uniform MOF nanosheets (NSs) are considered as ideal seed candidates because of the ease of deposition, better thickness control, and versatile functionality. Taking into account of potential benefits derived from MOF NS seeds, in this study, we prepared highly (110)-oriented ZIF-8 membrane using uniform ZIF-8 NS seeds derived from identically shaped ZIF-L precursors [29–31], which not only facilitated the deposition of oriented seed layers, but also led to enhanced CO$_2$ capacity compared with their bulk counterparts. Moreover, carrying out secondary growth at low temperature was confirmed crucial for the elimination of grain boundary space while maintaining the optimized orientation inherited from ZIF-8 seed layer (Fig. 1). As far as we know, this work represented the first report on the fabrication of highly (110)-oriented ZIF-8 membrane via secondary growth method. Gas separation results showed that H$_2$/CO$_2$ ideal selectivity of obtained ZIF-8 membrane exceeded H$_2$/N$_2$ and H$_2$/CH$_4$ gas pairs under ambient conditions, which has been rarely reported in previous studies.

2. Experimental section

2.1. Reagents and materials

Zinc nitrate hexahydrate (Zn(NO$_3$)$_2$·6H$_2$O, 99%, Xiya Reagent), Zinc acetate (Zn(CH$_3$COO)$_2$, AR, Macklin), 2-methylimidazole (Hmim, 98%, Macklin), N,N-dimethylformamide (DMF, 99.8%, Kemiou), methanol (CH$_3$OH, 99.5%, Macklin), ethanol (C$_2$H$_5$OH, 99.7%, Tianjin Kemiou), and ethylene imine polymer (PEI, Mw = 1800, 99%, Aladdin) were used as received without further purification. α-Al$_2$O$_3$ substrates were obtained from Fraunhofer IKTS, Germany.

![Fig. 1. Schematic illustration of preparing highly (110)-oriented ZIF-8 membrane.](image-url)
2.2. Preparation of leaf-shaped ZIF-L NSs

Uniform leaf-shaped ZIF-L NSs were synthesized as reported in the literature [32]. Initially, 1.30 g Hmim and 0.59 g Zn(NO$_3$)$_2$⋅6H$_2$O were dissolved in 40 mL deionized water via continuous stirring. Subsequently, above homogeneous aqueous solutions were blended under stirring and then stirred under ambient conditions for 4 h, which led to the formation of white precipitates. The above precipitates (i.e., ZIF-L NSs) were collected via repeated centrifugation (6000 rpm, 5 min), washed with DI water for several times, and dried overnight before further use.

2.3. Synthesis of ZIF-8 NSs

ZIF-8 NSs with leaf-like morphology were prepared as reported in the literature with slight modification [30, 31]. Initially, 150.0 mg of leaf-shaped ZIF-L crystals were uniformly dispersed in 144 mL DMF and 48 mL ethanol, and then sonicated for 5 min. Subsequently, the above solution was subjected to solvothermal treatment at 70 °C for 30 h. The above precipitates (i.e., ZIF-8 NSs) were isolated via repeated centrifugation (6000 rpm, 5 min), washed with DI water for several times, and dried overnight before further use.

2.4. Synthesis of ZIF-8 microcrystals and nanocrystals

ZIF-8 microcrystals and nanocrystals were prepared as reported in the literature with slight modification [33, 34]. For the synthesis of ZIF-8 microcrystals, initially, 1.58 g Hmim and 1.05 g Zn(CH$_3$COO)$_2$ were dissolved in 120 mL methanol, respectively. Subsequently, above homogeneous solutions were blended under stirring for 5–10 min and then kept at room temperature for 24 h to form white precipitates. The above precipitates were collected via repeated centrifugation (8000 rpm, 10 min), washed with methanol for several times, and dried overnight before further use. For the synthesis of ZIF-8 nanocrystals, initially, 1.62 g Hmim and 1.47 g Zn(NO$_3$)$_2$⋅6H$_2$O were dissolved in 100 mL methanol, respectively. Subsequently, above homogeneous solutions were blended under stirring for 5–10 min and then kept at room temperature for 24 h to form white precipitates. The above precipitates were collected via repeated centrifugation (12000 rpm, 20 min), washed with methanol for several times, and dried overnight before further use.

2.5. Oriented deposition of the ZIF-8 seed layer

Oriented seed layer was obtained via spin-coating. Initially, we prepared ZIF-8 seed suspension by adding 0.025 g PEI (Mw = 1800) and 0.010 g of ZIF-8 NSs into 5 g methanol. Subsequently, the above suspension was stirred at room temperature overnight after sonication for 10 min. In the next step, 0.1 mL of prepared ZIF-8 seed suspension was dropped on the surface of dry α-Al$_2$O$_3$ disk, and then spin-coating was conducted at 3000 rpm for 60 s. In the end, ZIF-8 seed layer-coated substrate was dried overnight before further use.

2.6. Secondary growth of highly (110)-oriented ZIF-8 seed layer

Initially, 2.27 g Hmim and 0.11 g Zn(NO$_3$)$_2$⋅6H$_2$O were dissolved in 20 mL deionized (DI) water under ice-water bath, respectively [35]. Subsequently, metal source aqueous solution was added in Hmim aqueous solution under continuous stirring. After further stirring under...
ice-water bath for 20 min, obtained precursor solution was poured in a 50 mL autoclave containing vertically placed ZIF-8 seed layer-coated α-Al₂O₃ substrate. The syntheses were conducted at ~5 °C for 12 h by keeping the autoclave in a refrigerator. After reaction, the substrate was taken out, gently rinsed with deionized (DI) water and dried under ambient conditions overnight. For the secondary growth at 30, 60, 90 and 120 °C, the same synthesis strategy was used except for the reaction temperature.

2.7. Preparation of ZIF-8 membrane with nano-sized seeds

Procedure for the preparation of ZIF-8 membrane with nano-sized seeds was follow: First, the ZIF-8 seed suspension was prepared by adding 0.025 g PEI (Mw = 1800) and 0.015 g ZIF-8 nanocrystals in 5 g methanol. After sonication for 10 min, the above suspension was further stirred at room temperature overnight. Subsequently, 0.2 mL of the above ZIF-8 seed suspension was dropped on the α-Al₂O₃ disk, followed by spin-coating at 3000 rpm for 60 s. In the next step, ZIF-8 seed layer-coated substrate was dried overnight before further use. Finally, secondary growth was conducted following the same procedure as depicted in section 2.6.

2.8. Gas permeation test

During the implementation of permeation test, volumetric flow rate on both feed and permeate sides was maintained at 50 mL/min; meanwhile, the pressure difference of feed side and permeate side was maintained at 1 bar. Gas phase composition was analyzed by 7890B GC, Agilent. The separation factor of binary mixture permeation (α_A/B) could be described as follows:

\[ \alpha_{A/B} = \frac{y_A/y_B}{x_A/x_B} \]

\( x \) represented volume fractions of different components (A, B) in the feed side, and \( y \) corresponded to volume fractions in permeate side.

2.9. Characterization

X-ray diffraction (XRD) patterns were collected on Rigaku SmartLab diffractometer. Thermogravimetric analysis (TGA) results were obtained on NETZSCH (TG 209) thermal analyzer under air or N₂ atmosphere. Scanning electron microscopy (SEM) images were evaluated by FlexSEM 1000 instrument (Hitachi Co.). Physical adsorption analysis was obtained on Mike ASAP 2020 Plus.

3. Results and discussion

3.1. Synthesis of ZIF-8 NSs

The first step was preparation of ZIF-8 NS seeds. In a recent study, Oh et al. [30] reported that leaf-shaped ZIF-L NSs could be transformed to ZIF-8 NSs preserving identical morphology through simple thermal treatment. Motivated and inspired by this, herein we attempted to prepare ZIF-8 NSs following the same route. A solution-based route for the preparation of uniform ZIF-L NSs has been well established in a previous study [32]. As shown in Fig. 2a and b, prepared ZIF-L NSs with the pore size of 0.34 nm in the (110) direction exhibited special leaf-like appearance with a general crystal size of 5 × 2 μm and a thickness of ca. 150 nm. Subsequently, as affirmed by SEM images and XRD patterns (Fig. 2c-e), prepared ZIF-L NSs were transformed into more thermodynamically stable ZIF-8 NSs maintaining the same morphology through solvothermal treatment in DMF-ethanol binary solvent.

In view of potential impacts of ZIF-8 seeds on gas permeation properties of prepared membrane, herein physical adsorption analysis was performed on prepared ZIF-8 NSs. In parallel, 70 nm-sized ZIF-8 NSs were...
nanocrystals (defined as ZIF-8$_\alpha$) and 4 μm-sized ZIF-8 microcrystals (defined as ZIF-8$_\beta$) were prepared further for comparison [33,34] (Fig. S2). CO$_2$ and N$_2$ adsorption isotherms of ZIF-8 NSs, ZIF-8$_\alpha$ and ZIF-8$_\beta$ measured at 298 K were shown in Fig. 2f and Fig. S3. It was found that on the one hand, all these samples showed similar N$_2$ adsorption capacity; on the other hand, ZIF-8 NSs exhibited considerably higher CO$_2$ adsorption capacity (32.91 cm$^3$ g$^{-1}$) than both ZIF-8$_\alpha$ and ZIF-8$_\beta$, which was potentially advantageous for improving the H$_2$/CO$_2$ separation selectivity of corresponding membranes. Besides, the pore volume and BET surface area of prepared ZIF-8 NSs reached 0.66 cm$^3$/g and 1304 m$^2$/g, respectively, which was comparable with other reported ZIF-8 particles [8,36,37] (shown in Table S1), thereby indicating that prepared ZIF-8 NSs were of high quality. In addition, their thermal stability was further evaluated by TGA. As shown in Fig. S4, prepared ZIF-8 NSs exhibited excellent thermal stability under both N$_2$ and air atmospheres, which was ideally suited for operation under harsh conditions.

3.2. Oriented deposition of ZIF-8 seed layer

The second step involved oriented deposition of ZIF-8 seed layer via spin-coating. It was observed that after spin-coating under optimized experimental conditions (Fig. S5), the porous α-Al$_2$O$_3$ substrate was fully covered with uniform ZIF-8 NSs (Fig. 3a). Cross-sectional SEM image further showed that the thickness of seed layer reached ~800 nm (Fig. 3b). Furthermore, the XRD results (Fig. 4) indicated that obtained ZIF-8 seed layer was dominantly (110)-oriented.

3.3. Secondary growth of highly (110)-oriented seed layer

Subsequently, secondary growth was carried out to seal intercrystalline gaps between neighboring seeds on the precondition of preserving preferred orientation inherited from the seed layer. Aqueous solution approach has been confirmed to be an excellent strategy for synthesizing high performance ZIF-8 membranes since Hmim could be easily deprotonated to achieve better intergrowth [35]. Previous researches indicated that high performance ZIF-8 membranes could be obtained in a wide range of reaction temperatures [35,38,39]. By referring to the strategy proposed by Lai et al. [35], initially secondary growth was conducted at reaction temperatures of 30, 60, 90 and 120 °C, respectively. Nevertheless, XRD results indicated that all ZIF-8 membranes prepared at these temperatures were randomly oriented (Fig. S6). SEM images further showed that not only the membrane surface became rough but also massive twin crystals were aggregated on the surface of membrane (Fig. S7).

Fortunately, our results showed that undesired twin growth could be effectively suppressed by carrying out secondary growth at low reaction temperature (~5 °C). SEM images (Fig. 3c and d) indicated that well-intergrown, twin-free membrane displaying a thickness of 1.1 μm had been emerged on porous α-Al$_2$O$_3$ substrate under optimized reaction conditions. Corresponding XRD pattern exhibited remarkable diffraction peaks corresponding to (110) and (220) planes of ZIF-8 crystallites, which convincingly demonstrated the dominance of (110)-preferred orientation inherited from the oriented seed layer (Fig. 4). This can be ascribed to reduced nucleation and growth rate of ZIF-8 crystalline grains in the synthetic solution and therefore, effective suppression of undesired twin growth accompanying with a substantial reduction in reaction temperature. As far as we know, this work represented the first report on the fabrication of highly (110)-oriented ZIF-8 membrane via secondary growth method. Moreover, considering higher CO$_2$ adsorption capacity of ZIF-8 NS seeds compared with their bulk counterparts, it is expected that prepared (110)-oriented ZIF-8 membrane may exhibit superior H$_2$/CO$_2$ selectivity.

3.4. Evaluation of gas separation performance

Finally, single gas permeation tests and mixed binary gas separation tests were operated under ambient conditions to evaluate permeation behavior of pure gas (such as H$_2$, CO$_2$, N$_2$ and CH$_4$) and equimolar gas mixtures (H$_2$/CO$_2$, H$_2$/N$_2$ and H$_2$/CH$_4$) through prepared (110)-

![Fig. 4. XRD patterns of simulated ZIF-8, (110)-oriented seed layer and (110)-oriented membrane, respectively. Note: * represented diffraction peaks of porous α-Al$_2$O$_3$ substrate.](image)
Fig. 5. (a) Single-gas and equimolar binary gas mixture permeation results under ambient conditions; (b) single-gas and equimolar binary gas mixture permeation results at 150 °C; (c) H₂ permeance, CH₄ permeance and ideal selectivity of equimolar H₂/CH₄ gas pair on prepared ZIF-8 membrane as a function of operation temperature; (d) evaluation of equimolar H₂/CH₄ gas mixture separation performance at different operating temperatures; (e) H₂ permeance, CO₂ permeance and ideal selectivity of equimolar H₂/CO₂ gas pair on prepared ZIF-8 membrane as a function of operation temperature; (f) evaluation of equimolar H₂/CO₂ gas mixture separation performance at different operating temperatures; (g) long-term stability of (110)-oriented ZIF-8 membrane towards equimolar H₂/CO₂ gas mixture separation under ambient conditions; and (h) long-term stability of (110)-oriented ZIF-8 membrane towards equimolar H₂/CH₄ gas mixture separation at 150 °C.
oriented ZIF-8 membrane. The relationship between kinetic diameters and permeance of gas molecules was shown in Fig. 5a. It was observed that the permeance of H₂ strongly exceeded CH₄, N₂ and CO₂; moreover, the ideal selectivity of H₂/CO₂, H₂/N₂ and H₂/CH₄ gas pairs reached 12.9, 7.7 and 11.7, respectively (Fig. 5a), which was higher than their Knudsen diffusion coefficients (i.e., 4.7, 3.7 and 2.8), thereby illustrating the dominance of molecular sieving mechanism and the existence of few grain-boundary defects.

It should be noted that the permeance of gas molecules was not inversely proportional to their kinetic diameters, which was contradictory to common experimental observations. Particularly, the ideal selectivity of H₂/CO₂ equimolar gas pair on highly (110)-oriented ZIF-8 membrane exceeded H₂/N₂ and H₂/CH₄ gas pairs under ambient conditions. To verify whether the ZIF-8 seeds exerted influence on the H₂/CO₂ selectivity, herein ZIF-8 membrane was further prepared from nano-sized ZIF-8 seeds while keeping other synthetic conditions unchanged (shown in Figs. S8 and S9). The ideal H₂/CO₂, H₂/N₂ and H₂/CH₄ selectivity of prepared ZIF-8 membrane reached 10.0, 5.5 and 10.5 at room temperature. We noticed that the ideal H₂/CO₂ selectivity was lower than not only the ideal H₂/CH₄ selectivity of the same membrane, but also the ideal H₂/CO₂ selectivity of prepared (110)-oriented ZIF-8 membrane. We therefore concluded that the increased H₂/CO₂ selectivity could be due to preferential CO₂ adsorption capacity of not only PEI but also ZIF-8 nanosheets, which led to certain reduction in CO₂ diffusion coefficients and therefore, enhanced ideal selectivity of H₂/CO₂ within the framework of ZIF-8 membrane [40].

Subsequently, the evaluation of the separation performance of prepared (110)-oriented membrane at different operating temperatures was conducted. It was found that permeance and SF of binary gas mixture strongly relied on the operating temperature. By raising the operation temperature gradually from 30 to 150°C, H₂ permeance and SF of equimolar H₂/CO₂, H₂/N₂ and H₂/CH₄ steadily increased; simultaneously, CO₂, N₂ and CH₄ permeances were slightly decreased (as shown in Fig. 5b-f and Fig. S10), which could be explicated by adsorption–diffusion model: Higher operating temperature led to lower adsorption capacity of CO₂, N₂ and CH₄ in the pores of ZIF-8, which inevitably blocked the diffusion of highly mobile and rarely adsorbed H₂. Since a higher free microporous volume for the diffusion of H₂ was established at higher operating temperature, both H₂ permeance and SF of equimolar H₂/CO₂, H₂/N₂ and H₂/CH₄ mixtures were concurrently enhanced [41,42]. To the best of our knowledge, the H₂/CO₂ selectivity of our ZIF-8 membrane was the highest in comparison with previously published results (Table S2).

Furthermore, long-term stability of prepared (110)-oriented ZIF-8 membrane was investigated under ambient conditions. Gas permeation test results revealed that both SF of equimolar H₂/CO₂ gas mixture and permeance of H₂ showed no discernible degradation within 20 h (shown in Fig. 5g), which vividly confirmed reliable long-term operation stability. Furthermore, its long-term thermal stability was evaluated at 150°C and 1 bar. Gas permeation test results revealed that both SF of equimolar H₂/CH₄ and permeance of H₂ remained unchanged within 50 h (shown in Fig. 5h), which was indicative of its excellent thermal stability.

4. Conclusions

To sum up, in this study we prepared highly (110)-oriented ZIF-8 membrane through combining oriented deposition of ZIF-8 NSs with controlled in-plane secondary growth. It was found that the following elements were vital for guaranteeing the formation of the ZIF-8 membrane with desired microstructure: 1) Preparation of uniform ZIF-8 NS seeds exposing (110)-dominant facets through simple solvothermal treatment of identically shaped ZIF-L precursors. 2) Carrying out secondary growth at low temperature for the suppression of undesired twin growth. Gas permeation results indicated that H₂/CO₂ ideal selectivity of prepared (110)-oriented ZIF-8 membrane exceeded H₂/N₂ and H₂/CH₄ gas pairs under ambient conditions, owing to preferential CO₂ adsorption capacity of not only PEI but also ZIF-8 nanosheets compared with their bulk counterparts. Our research highlighted the significance of preferred orientation regulation in tailoring the gas permeation behavior of MOF membranes.

Author statement

Chenhan Zhang: completed the main experiments and the analysis of relevant experimental results. Jiahui Yan: assisted in drawing the schematic illustration and the test of gas separation performance. Taotao Ji and Dongying Du: assisted in drawing the crystal structure of ZIF-8. Yanwei Sun: assisted in the test and analysis of gas separation performance. Liangliang Liu: assisted in morphological characterization and the analysis of relevant experimental results. Xiongfu Zhang: helped with the analysis of gas permeation results. Yi Liu: proposed the major idea, projected relevant experiments, and jointly wrote the manuscript.

Declaration of competing interest

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Appendix A. Supplementary data

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