# Supplementary Information

# One-dimensional Material as Nano-scaffold and Pseudo-seed for Facilitated Growth of Ultrathin, Mechanically Reinforced Molecular Sieving Membranes

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#### **Materials and Methods**

All chemicals were used as received. Anopore aluminium oxide (AAO, GE Healthcare Life Sciences; diameter: 25 mm, pore size: 0.1 µm) membranes, with annular polypropylene ring were used as support. The polyethersulfone (PES) membrane with a diameter of 47 mm and a pore size of 0.03 µm was purchased from Sterlitech Corporation, USA. Carbon nanotubes, (multi-walled O.D. × I.D. × L 10-20 nm × 5-10 nm × 0.5-200 µm, purity  $\geq$ 95%) were purchased from Sigma Aldrich Australia. Dopamine hydrochloride (98 %, Sigma-Aldrich) and tris(hydroxymethyl)aminomethane (Tris, ACS reagent,  $\geq$ 99.8%, Sigma-Aldrich Australia) were used for CNT coating layer modification. Zinc nitrate hexahydrate (Zn(NO<sub>3</sub>)<sub>2</sub>.6H<sub>2</sub>O, 98%) and 2-methylimidazole (Hmim, C<sub>4</sub>H<sub>6</sub>N<sub>2</sub>, 99%) supplied by Sigma-Aldrich Australia were used for ZIF-8 preparation. Methanol (absolute) was purchased from Merck, Australia. The water used for the experiments was purified with a water purification system (Milli-Q integral water purification system, Merck Millipore) with a resistivity of 18.2 MΩ/cm.

*Polydopamine modification of CNTs:* CNTs were modified with polydopamine by an ethanolmediated oxidative dopamine polymerization process as reported previously.<sup>[1]</sup> Briefly, 10 mg of CNTs was dispersed in a solution of water (15 mL) and ethanol (20 mL) under sonication, followed by the addition of 40 mg of dopamine. 10 mL of Tris aqueous solution (25 mM) was then added with magnetic stirring. After 2 h of reaction at room temperature, the modified CNTs were collected from the solution by centrifugation (5000 rpm for 5 min) and washed with deionized water three times. The modified CNTs were dispersed in 200 mL fresh deionized water as a coating solution.

Preparation of ZIF-8/CNTs membrane on porous AAO disk: The above coating solution was sonicated for 60 min to yield a stable and uniformly dispersed nanotubes. 3 mL of the solution

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was then deposited onto the AAO porous membranes by vacuum filtration. Contra-diffusion method was conducted to fabricate the ultrathin ZIF-8/CNT membranes.<sup>[2]</sup> The CNTs deposited supports were mounted on a homemade setup, where the zinc nitrate solution and Hmim solution were separated by the supporting membrane. The zinc nitrate solution, prepared by dissolving 0.293 g of  $Zn(NO_3)_2.6H_2O$  I n 20 mL of methanol, was added to the CNTs deposited side of the support and Hmim solution, prepared by adding 0.649 g of Hmim in 20 mL of methanol, was immediately added to the other side of the support. The designed Hmim:  $Zn^{2+}$  molar ratios in the system was 8 and was kept constant throughout the study. After crystallization at room temperature ( $20 \pm 2$  °C) for up to 60 min, the obtained membranes were thoroughly rinsed with methanol and dried at 50 °C for 6 h. For comparison, ZIF-8 membranes were also prepared on a bare AAO and on a pristine CNTs deposited AAO, following the same preparation method described above for 60 min growth.

### Characterization

Powder X-ray diffraction (XRD) patterns were recorded at room temperature using a Miniflex 600 diffractometer (Rigaku, Japan) with Cu K $\alpha$  radiation (15 mA and 40 kV) at a scan rate of 2° min<sup>-1</sup> and a step size of 0.02°. Scanning electron microscopy (SEM; FEI Nova NanoSEM 450) was used for imaging the surface and cross-sectional morphologies of membranes. Transmission electron microscopy (TEM) images were taken by a FEI Tecnai G2 T20 operated at an accelerating voltage of 200 kV. Fourier Transform Infrared (FTIR) spectra of the samples were taken by an attenuated total reflectance (ATR) FTIR (PerkinElmer, U.S.A.) at an average of 20 scans with a resolution of 4 cm<sup>-1</sup>.

The single gas permeation of hybrid membranes was measured using the pressure rise method,<sup>[3]</sup> as described in detail in our previous study.<sup>[4]</sup> The membrane samples were attached to a porous

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stainless steel holder using a vacuum sealant (Torr seal, Varian). The gas permeation tests were performed at room temperature ( $20 \pm 2 \, ^{\circ}$ C) on H<sub>2</sub>, CO<sub>2</sub>, N<sub>2</sub>, and CH<sub>4</sub>. The pressure rise of the permeating gas was measured using a pressure transducer (MKS 628B Baratron). For each single gas measurement, the respective gas was fed to one side (feed) of the permeation cell while the other side (permeate) of which was under vacuum. Since the feed side was at ambient pressure, a pressure difference of 1 atm was maintained between the permeate side and the feed side during permeation measurements.



**Figure S1.** (a) Photos of water dispersibility and the corresponding TEM images of CNTs (I) and polydopamine-coated CNTs (II), XRD patterns (b) and FTIR spectrum (c) of CNTs (I) and polydopamine-coated CNTs (II), (f) schematic illustration of the coated CNT and the chemical structure of polydopamine.

In high resolution transmission electron microscopy (HRTEM) images (Figure S1a, ESI), a layer of lower contrast, surrounding the CNTs can be easily identified, demonstrating a conformal PDA coating on CNTs. Fourier transform infrared spectroscopy (FTIR) analysis further confirmed that PDA was successfully coated on CNTs (Figure S1c, ESI). Upon coating with PDA, new absorption bands at around 1633 and 3438 cm<sup>-1</sup> appear, which are assigned to the aromatic rings and catechol –OH groups, respectively,<sup>[1]</sup> confirming the successful deposition of PDA on the surface of the CNTs. For the minimal changes in the intrinsic properties of CNTs, specifically its flexibility and diameters, an extremely thin coating layer (< 2 nm) of PDA was formed on the CNTs. The introduction of thin PDA coating on the CNTs made no difference in the X-ray diffraction patterns (XRD) (Figure S1b, ESI) as compared with the pristine CNTs.



**Figure S2.** SEM (a, b) and optical (c) images of pristine (a, c1-c3) and modified (b, c4-c6) CNTs-deposited on AAO. Detailed experimental: deposited pristine CNTs from (c1, c4) 1 mL (c2, c5) 3 mL and (c3, c6) 6 mL mother solution. Pristine CNTs mother solution: 10 mg CNTs in 200 mL DDI water.



**Figure S3.** SEM images of ZIF-8 film prepared on (a, b, c) bare AAO and on (d, e, f) AAO deposited with pristine CNTs. Zinc side: b, e; Hmim side: c, f.

Distinct ZIF-8 nanocrystals and crystal islets are notable on both sides and within the support if the support surface was not deposited with the modified CNTs (Figure S3a-c, ESI). In fact, due to the uninterrupted fast contra-diffusion of metal ions and ligand molecules through the large pores of the support and the week interaction between the species and the support, a non-continuous ZIF-8 layer is formed. The situation is similar when the pristine CNTs are attempted (Figure S3d-f, ESI). Filtration of non-modified CNTs suspension usually results in a deposition of huge agglomerates and bundles on the substrate as a result of their poor aqueous dispersibility and high tendency to bundle up.<sup>[5]</sup> Subsequently, such a non-uniform deposition does not effectively contribute to the formation of a continuous MOF film; as seen in Figure S3d of ESI, scattered crystals along with islands composed of a mixture of CNTs and ZIF-8 crystals is observable. It is also important to note that ZIF-8 and CNTs are phase-separated as opposed to the fully embedded CNTs in ZIF-8 when PDA-coated CNTs are used (Figure S3e, ESI).



**Figure S4.** SEM images of bare AAO (a), as-prepared samples with insufficient (b, c), and excess (d, e, f) deposition of modified CNTs on AAO before (b, d) and after (e, f) contradiffusion synthesis. The inset in f is a high magnification cross-sectional view. Detailed experimental: deposited CNTs from (b) 1 mL and (d) 6 mL mother solution respectively.

When the coverage of PDA-coated CNTs on the AAO is insufficient (see the experimental in ESI), only non-continuous and imperfect growth of ZIF-8 on the support can be obtained (Figure S4, ESI). Meanwhile, using an excess volume of the suspension (see the experimental in ESI) results in a membrane with rough surface morphology after the contra-diffusion synthesis (Figure S4, ESI). However, high-magnification top view and cross-sectional SEM images (Figure S4, ESI) reveal that the rough morphology at the top is indeed composed of an underlying compact and defect-free intercalating layer. The excessive volume of the suspension results in a multi-layer CNTs network, which by generating a nanoporous structure with high surface area per unit volume provides an ideal medium for ZIF-8 crystallization. However, the

crystallization rate on the CNTs closer to the surface of AAO is much faster due to the presence of higher ligand concentration near the surface than that of further away the surface.<sup>[2]</sup> Subsequently, owing to the self-limiting crystal growth mechanism in the contra-diffusion process,<sup>[6]</sup> the underlying CNT network is sealed prior to further growth across the CNTs film, leaving an ultrathin continuous and compact ZIF-8/CNT membrane with a rough and larger size ZIF-8/ CNTs composite on the surface. This mechanism is further confirmed by the fact that increasing synthesis time affects neither the membrane morphology nor its thickness. This nanoscale surface morphology can provide a better contact between the feed gas and the hybrid membrane, potentially leading to a higher gas permeation through the membrane.<sup>[7]</sup>

A long reaction time was noted for the preparation of ZIF-8 layer on the supports directly modified with PDA and the reported MOF membranes had thicknesses in the rage of tens of microns,<sup>[8]</sup> adversely affecting gas flow through the membranes. In our study The formation of considerably thinner (more than two order of magnitude) membrane in a shorter synthesis time (1 h. versus 1 day in reference <sup>[8]</sup>) is attributed to the considerably larger nucleation area available on the PDA-coated CNTs deposited support resulted from the subsequent larger PDA content, considering the high surface area of the deposited CNTs as compared to the corresponding bare support, as well as to the self-terminating contra-diffusion synthesis process. These results explicitly demonstrate the essential role of PDA-coated CNTs hybrid and that a sufficient and uniform coverage of nanotubes on the support is required for the formation of ultrathin and compact ZIF-8 layer on support.



**Figure S5.** SEM images (a, b) and XRD pattern (c) of the ZIF-8/CNTs membrane after sonication for 2 h.



**Figure S6.** Optical image of the free standing ZIF-8/CNT hybrid membrane floated in the sodium hydroxide solution (a) and SEM images of Cross-sectional view (b, c) and surface edge (d) of the corresponding free standing membrane.

The supported membrane is soaked in a weak sodium hydroxide solution (NaOH). The base solution selectively etched and removed the AAO support and an ultrathin free standing ZIF-8/CNT hybrid membrane floated in the solution (Figure S6, ESI). This experimental observation not only validates the high mechanical stability of the hybrid membrane, it also suggest that the

method explained here can lead to a versatile route for the development of ultrathin free-standing MOF/CNTs hybrid membranes. Carbon nanotubes (CNTs), having low wettability, low thermal expansion coefficient (CTE) and high gas adsorption selectivity,<sup>[9]</sup> are considered as an ideal reinforcing material and the resulting free-standing MOF hybrid should be able to retain good thermal and mechanical stability under practical applications. An elaborate study on the resultant free-standing membranes is currently under way.



**Figure S7.** Single gas permeances of several gases through the ZIF-8/CNT membrane at 20 °C and different feed pressures. The plot shows that viscous flow (permeance depends on pressure) is absent, indicating the hybrid membrane is well-intergrown and free of cracks and defects.



Figure S8. FTIR ATR spectra of the AAO support deposited with modified CNTs, the ZIF-

8/CNTs membranes as a function of synthesis time, and ZIF-8 powder.



**Figure S9.** SEM images of (a, b) bare PES, (c, d) PES with deposited modified CNTs (6 mL of mother solution) and (e, f) as prepared membrane after contra-diffusion synthesis (1 h).



**Figure S10.** (a) XRD patterns and (b) FTIR spectra of pristine PES, supported ZIF-8/CNT membrane and ZIF-8.

**Table S1.** Single gas permeances and ideal selectivities for the ZIF-8@CNTs-t (t: crystallization time (min), hybrid membranes at 20 <sup>o</sup>C and 1 bar. E shows the sample prepared with an excess use of CNTs (6mL of the mother solution).

| Sample ID | Permeance              |                 | Selectivit                      | ty                             |                                 |              |  |
|-----------|------------------------|-----------------|---------------------------------|--------------------------------|---------------------------------|--------------|--|
|           | (10 <sup>-7</sup> mol/ |                 |                                 |                                |                                 |              |  |
|           | m <sup>2</sup> .s.Pa)  |                 |                                 |                                |                                 |              |  |
|           | H <sub>2</sub>         | CO <sub>2</sub> | H <sub>2</sub> /CO <sub>2</sub> | H <sub>2</sub> /N <sub>2</sub> | H <sub>2</sub> /CH <sub>4</sub> | $H_2/C_3H_8$ | C <sub>3</sub> H <sub>6</sub> /C <sub>3</sub> H <sub>8</sub> |
| Bare AAO  | 1267                   | 998             | 1.3                             | 1.5                            | 1.5                             | 3.1          | 1.1  |
| AAO-CNTs  | 1343                   | 1002            | 1.3                             | 1.4                            | 1.2                             | 2.8          | 1  |
| AAO- ZIF- | 954                    | 381             | 2.5                             | 2.2                            | 2.5                             | 3.2          | 1.1  |
| 8/CNTs-5  |                        |                 |                                 |                                |                                 |              |  |
| AAO- ZIF- | 560                    | 181             | 3.7                             | 3.8                            | 4.1                             | 5.2          | 1.3  |

| 8/CNTs-30   |     |      |      |      |      |       |      |
|-------------|-----|------|------|------|------|-------|------|
| AAO- ZIF-   | 287 | 20.5 | 14   | 18   | 35   | 950.1 | 16.7 |
| 8/CNTs-60   |     |      |      |      |      |       |      |
| AAO- ZIF-   | 304 | 18.6 | 16.3 | 16.5 | 30.3 | 916   | 16.1 |
| 8/CNTs-60-E |     |      |      |      |      |       |      |

Owing to its large diameter (~ 100 nm) straight channels, AAO support shows very high and relatively close permeances for all the gases, indicating its minimal effect on the overall membrane separation properties. Surprisingly, an increase in the permeances of all gases was noticed upon homogeneous deposition of modified nanotubes. For example, H<sub>2</sub> permeance of blank AAO increased from 1267 to  $1343 \times 10^{-7}$  mol m<sup>-2</sup> s<sup>-1</sup> Pa<sup>-1</sup> when a moderate amount (See materials and methods in ESI) of the nanotubes were deposited on its surface. The possible reason is that AAO porous channels were modified by dopamine oligomers dissolved from the polydopamine coating layer in water during the formation of modified CNTs layer via vacuum filtration. The modified AAO channel surfaces favorably interact with gas molecules, slightly enhancing gas permeances.

A decrease in the permeance of CO<sub>2</sub> is observed for ZIF-8 membranes prepared with an excess use of CNTs (ZIF-8/CNT-E; materials and methods in ESI). Surprisingly, the membrane also shows an enhanced H<sub>2</sub> permeance ( $304 \ (\pm 8.717) \times 10^{-7} \ \text{mol} \ \text{m}^{-2} \ \text{s}^{-1} \ \text{Pa}^{-1}$ ) and greater H<sub>2</sub> selectivity over CO<sub>2</sub> ( $16.3 \pm 0.87$ ), which could be attributed to the resultant new nano-scale rough surface morphology (Figure S4e,f), which can allow better contact between the ZIF-8 crystals and highly mobile H<sub>2</sub> molecules.<sup>[7]</sup> The outstanding hydrogen permselectivity in this study is due to the ultrathin ZIF-8 layer (~200 nm). Another possible reason for the clear cut-off between  $H_2$  and larger gases can be the presence of the CNTs network in the matrix of ZIF-8. It was previously shown that the presence of carbonaceous materials such as GO can effectively slow down the permeance of larger gases through the ZIF-8 membrane by reducing the nonselective intercrystalline defect pathways and also by its possible constriction effect on the flexibility of ZIF-8 lattices, thus restricting larger molecules to go into the pores.<sup>[10]</sup>

**Tables S2.** Comparison of the synthesis parameters (time and temperature) and gas permeation properties of the ZIF-8/CNTs hybrid membrane in this work with other ZIF-8 membranes from the recent literature.

| Membrane/              | MOF       | Synthes | H <sub>2</sub>         | Selectiv          | Selectivity      |                  |                               | Ref  |
|------------------------|-----------|---------|------------------------|-------------------|------------------|------------------|-------------------------------|------|
| Growth                 | Thickness | is T    | Permeance              | H <sub>2</sub> /C | H <sub>2</sub> / | H <sub>2</sub> / | H <sub>2</sub> /              |      |
| Facilitator            | (µm)      | (°C)/t  | (10 <sup>-7</sup> mol/ | O <sub>2</sub>    | N <sub>2</sub>   | CH <sub>4</sub>  | C <sub>3</sub> H <sub>8</sub> |      |
|                        |           | (h)     | m <sup>2</sup> .s.Pa)  |                   |                  |                  |                               |      |
| ZIF-8/APTES            | ~ 2       | 150/5   | 573                    | 17.05             | 15.4             | _                | _                             | [11] |
| functionalized         |           |         |                        |                   |                  |                  |                               |      |
| α-alumina              |           |         |                        |                   |                  |                  |                               |      |
| particles              |           |         |                        |                   |                  |                  |                               |      |
| ZIF-8/PDA-             | 30        | 85/24   | 266                    | 8.8               | 15.4             | 24.6             | 442.5                         | [12] |
| coated surface         |           |         |                        |                   |                  |                  |                               |      |
| ZIF-8/APTES            | Less than | RT/5    | 201                    | 7.04              | 7.8              | 8.6              | _                             | [7]  |
| functionalized         | 1         |         |                        |                   |                  |                  |                               |      |
| TiO <sub>2</sub> layer |           |         |                        |                   |                  |                  |                               |      |

| ZIF-8/Nil           | 16  | RT/24  | 126.2                 | _                 | 3.7              | _     | _     | [2]  |
|---------------------|-----|--------|-----------------------|-------------------|------------------|-------|-------|------|
| ZIF-8/EDA           | 0.2 | RT/3   | 20.5                  | 12.8              | 9.7              | _     | _     | [13] |
| modified            |     | min    |                       |                   |                  |       |       |      |
| surface             |     |        |                       |                   |                  |       |       |      |
| ZIF-8/EDA           | ~2  | RT/2   | 7.5                   | 5.1               | 8.3              | 9.1   | 833   | [4]  |
| modified            |     |        |                       |                   |                  |       |       |      |
| surface             |     |        |                       |                   |                  |       |       |      |
| ZIF-8/ZnO           | 6   | 100/5  | 1.04                  | 7.6               | 12.3             | 12.7  | _     | [14] |
| nanorods            |     |        |                       |                   |                  |       |       |      |
| ZIF-8/PDA-          | 20  | 85/24  | 2.17                  | 10.3              | 17.6             | 34.8  | 905.1 | [8]  |
| coated surface      |     |        |                       |                   |                  |       |       |      |
| ZIF-8@GO/           | 20  | 85/16  | 1.45                  | 22.4              | 102.8            | 198.3 | 5870. | [10] |
| PDA-coated          |     |        |                       |                   |                  |       | 1     |      |
| surface             |     |        |                       |                   |                  |       |       |      |
| ZIF-8/ZnAl-         | 20  | 100/12 | 1.4                   | 4.2 <sup>a)</sup> | 10 <sup>a)</sup> | 12.1  | _     | [15] |
| CO <sub>3</sub> LDH |     |        |                       |                   |                  |       |       |      |
| buffer layers       |     |        |                       |                   |                  |       |       |      |
| ZIF-8/Nil           | 0.9 | 30/9   | 1.23                  | _                 | _                | _     | 370   | [6]  |
|                     |     |        |                       |                   |                  |       |       |      |
| ZIF-8/ 2D ZIF-      | 0.1 | RT/3   | 0.546                 | 1.6               | 11.1             | 11.2  | 405   | [16] |
| 8/GO hybrid         |     |        |                       |                   |                  |       |       |      |
| nanosheets          |     |        |                       |                   |                  |       |       |      |
| ZIF-8/ZIF-L         | 3.5 | RT/6   | ~ 6000 <sup>(b)</sup> | ~ 4.5             | ~ 6.5            | ~ 6   | _     | [17] |

| seed crystals |           |      | Barrer <sup>(c)</sup> |    |    |    |     |      |
|---------------|-----------|------|-----------------------|----|----|----|-----|------|
| ZIF-8/PDA-    | Less than | RT/1 | 287                   | 14 | 18 | 35 | 950 | This |
| modified      | 0.2       |      |                       |    |    |    |     | work |
| CNTs          |           |      |                       |    |    |    |     |      |

<sup>a)</sup>Mixture separation factor; <sup>b)</sup> Permeability; <sup>c)</sup> Barrer =  $3.348 \times 10^{-16}$  mol m m<sup>-2</sup> Pa<sup>-1</sup> s<sup>-1</sup>.

 Table S3. Single gas permeances and ideal selectivities of three ZIF-8/CNT-60 membrane

 samples tested at 25 °C and 1 bar.

| Membrane | Permeance                                    |                 | H <sub>2</sub> / CO <sub>2</sub> | Average     | Standard deviation |  |  |
|----------|--|-----------------|----------------------------------|-------------|--------------------|--|--|
|          | (10 <sup>-7</sup> mol/ m <sup>2</sup> .s.Pa) |                 | selectivity                      | selectivity | of selectivity     |  |  |
|          | H <sub>2</sub>                               | CO <sub>2</sub> |                                  |             |                    |  |  |
|          |  |                 |                                  |             |                    |  |  |
| M1       | 295  | 19.5            | 15.1                             |             |                    |  |  |
| M2       | 280  | 21.7            | 12.9                             | 14          | 1.1                |  |  |
| M3       | 286  | 20.3            | 14.1                             |             |                    |  |  |

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