

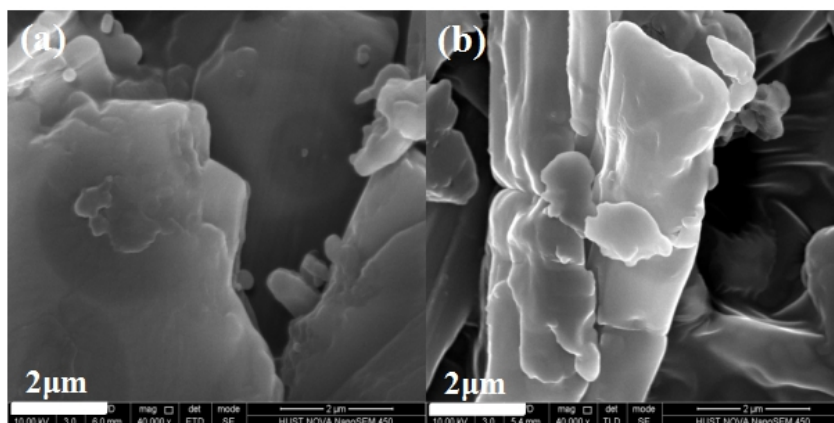
## Supporting Information

### **Effects of Synthetic Methodology on Microporous Organic Hyper-Cross-Linked Polymers with Respect to Structural Porosity, Gas Uptake Performance and Fluorescence Properties**

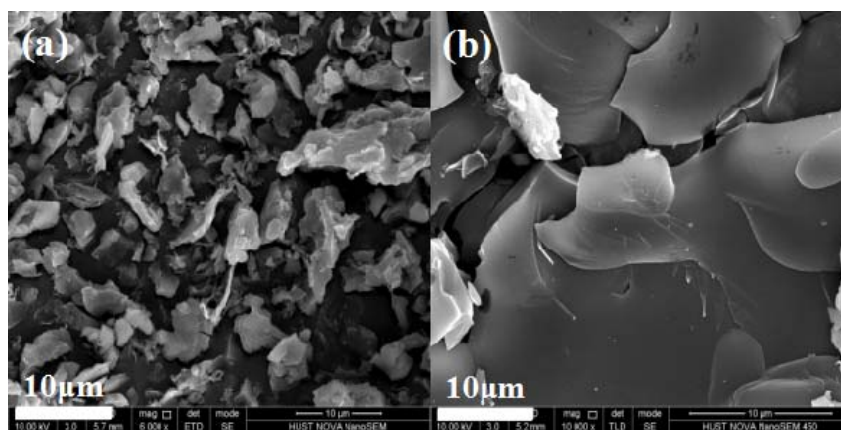
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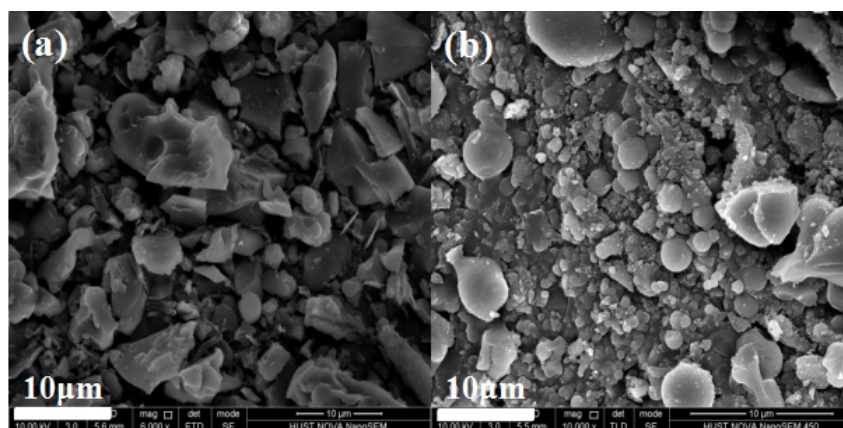
\*E-mail [bien.tan@mail.hust.edu.cn](mailto:bien.tan@mail.hust.edu.cn); Tel +86-27-87558172; Fax +86-27-87543632 (B. T.).



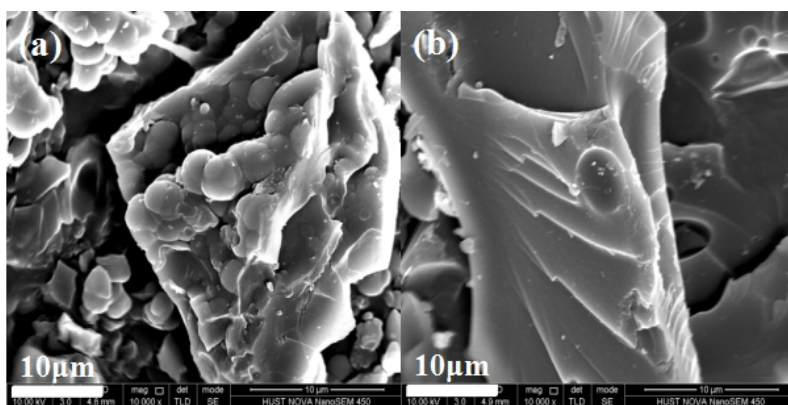
**Figure S1.** SEM images of (a) 1,4-diphenylbenzene, and (b) 1,3-diphenylbenzene at the same magnification (scale bars 2 μm).



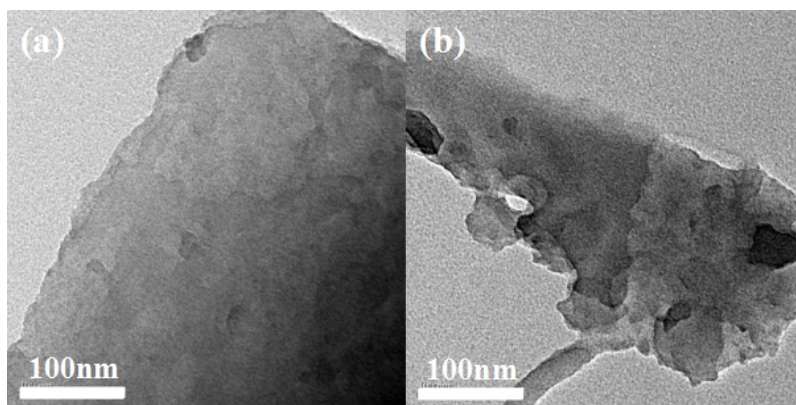
**Figure S2.** SEM images of (a) polymer 1, and (b) polymer 2 at the same magnification (scale bars 10 μm).



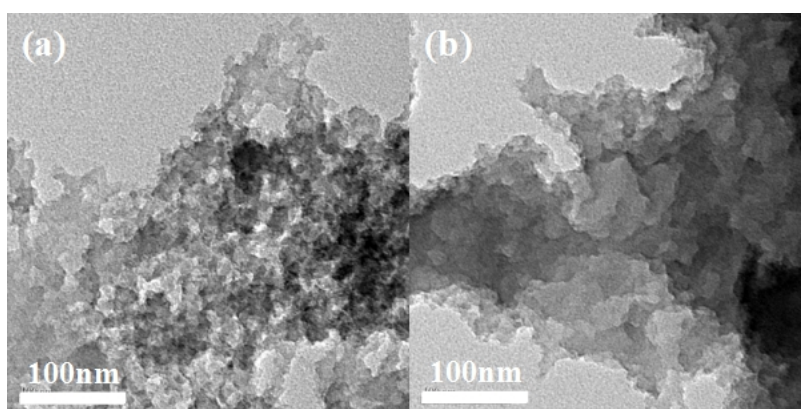
**Figure S3.** SEM images of (a) polymer 3, and (b) polymer 4 at the same magnification (scale bars 10 μm).



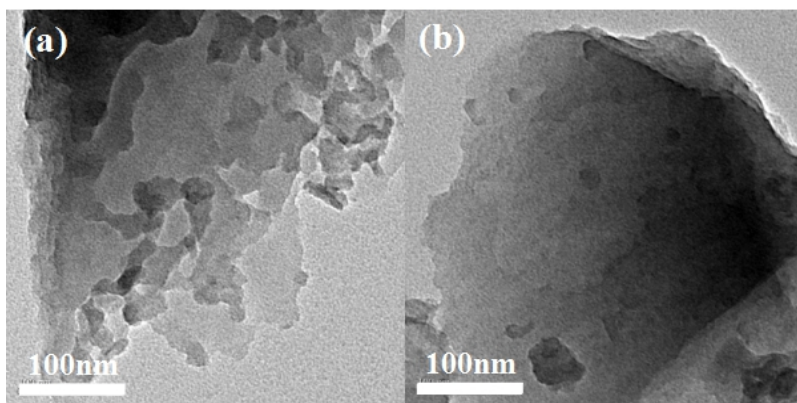
**Figure S4.** SEM images of (a) polymer 5, and (b) polymer 6 at the same magnification (scale bars 10 μm).



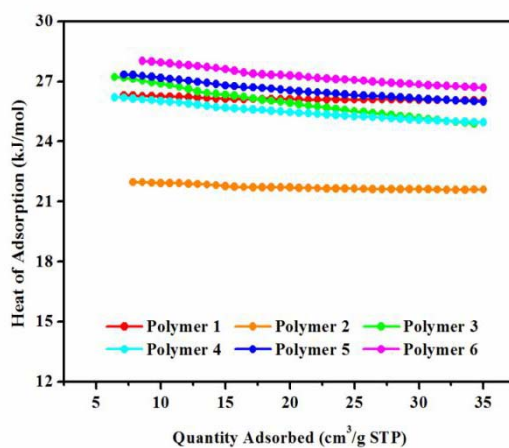
**Figure S5.** TEM images of (a) polymer 1, and (b) polymer 2 at the same magnification (scale bars 100 nm).



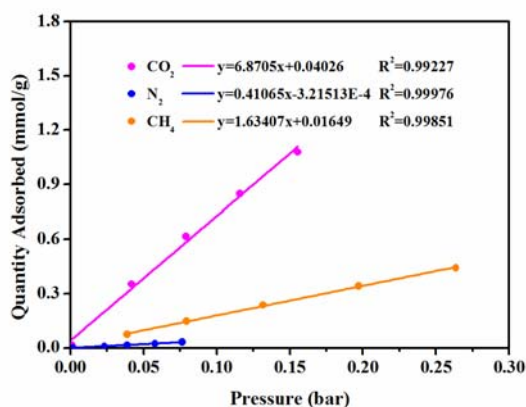
**Figure S6.** TEM images of (a) polymer 3, and (b) polymer 4 at the same magnification (scale bars 100 nm).



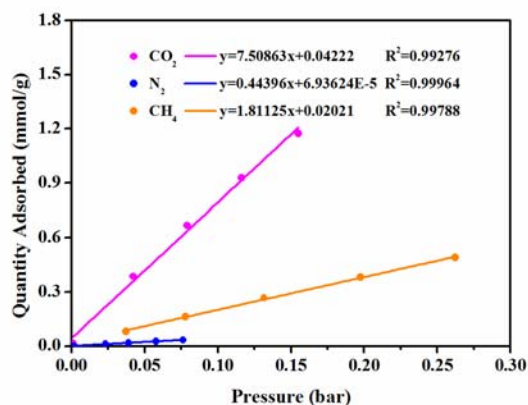
**Figure S7.** TEM images of (a) polymer 5, and (b) polymer 6 at the same magnification (scale bars 100 nm).



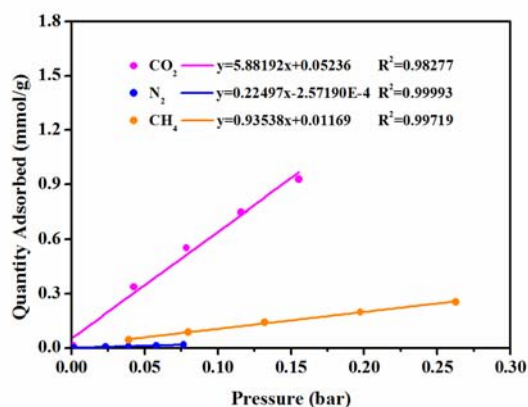
**Figure S8.** Isosteric heat of adsorption for CO<sub>2</sub> at different CO<sub>2</sub> loadings.



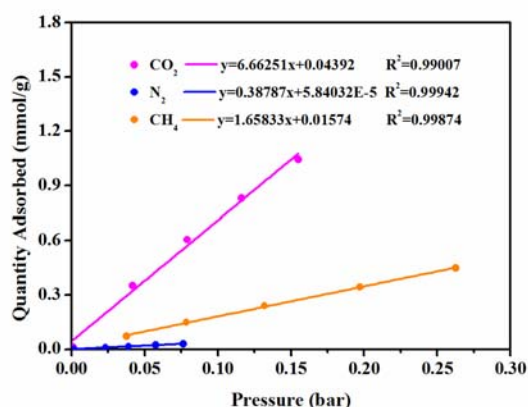
**Figure S9.** Adsorption selectivity of CO<sub>2</sub>/N<sub>2</sub> and CO<sub>2</sub>/CH<sub>4</sub> for polymer 1 calculated using Henry's law initial slope method according to the adsorption isotherms of CO<sub>2</sub> (pink), N<sub>2</sub> (blue), and CH<sub>4</sub> (orange) at 273.15 K and a low pressure coverage of less than 0.3 bar.



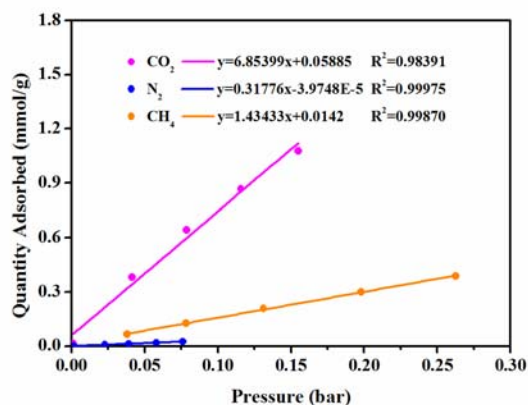
**Figure S10.** Adsorption selectivity of CO<sub>2</sub>/N<sub>2</sub> and CO<sub>2</sub>/CH<sub>4</sub> for polymer **2** calculated using Henry's law initial slope method according to the adsorption isotherms of CO<sub>2</sub> (pink), N<sub>2</sub> (blue), and CH<sub>4</sub> (orange) at 273.15 K and a low pressure coverage of less than 0.3 bar.



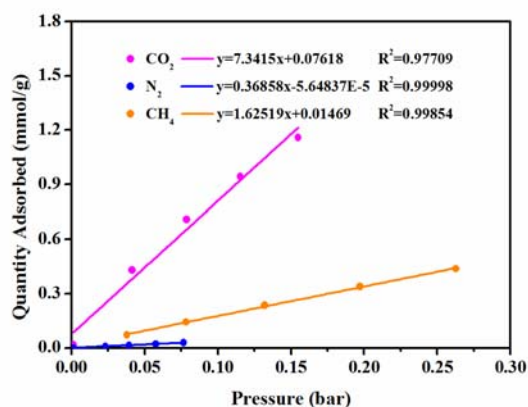
**Figure S11.** Adsorption selectivity of CO<sub>2</sub>/N<sub>2</sub> and CO<sub>2</sub>/CH<sub>4</sub> for polymer **3** calculated using Henry's law initial slope method according to the adsorption isotherms of CO<sub>2</sub> (pink), N<sub>2</sub> (blue), and CH<sub>4</sub> (orange) at 273.15 K and a low pressure coverage of less than 0.3 bar.



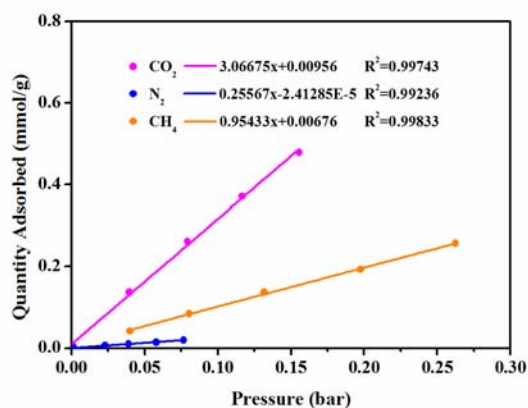
**Figure S12.** Adsorption selectivity of CO<sub>2</sub>/N<sub>2</sub> and CO<sub>2</sub>/CH<sub>4</sub> for polymer **4** calculated using Henry's law initial slope method according to the adsorption isotherms of CO<sub>2</sub> (pink), N<sub>2</sub> (blue), and CH<sub>4</sub> (orange) at 273.15 K and a low pressure coverage of less than 0.3 bar.



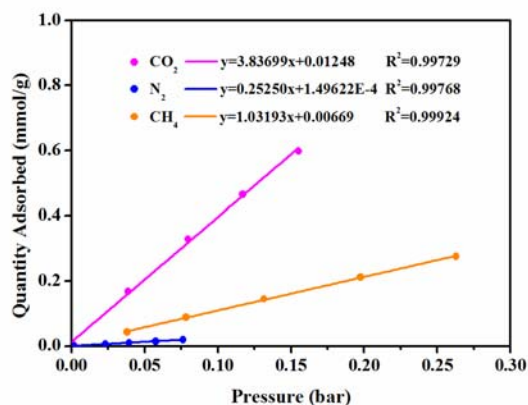
**Figure S13.** Adsorption selectivity of CO<sub>2</sub>/N<sub>2</sub> and CO<sub>2</sub>/CH<sub>4</sub> for polymer **5** calculated using Henry's law initial slope method according to the adsorption isotherms of CO<sub>2</sub> (pink), N<sub>2</sub> (blue), and CH<sub>4</sub> (orange) at 273.15 K and a low pressure coverage of less than 0.3 bar.



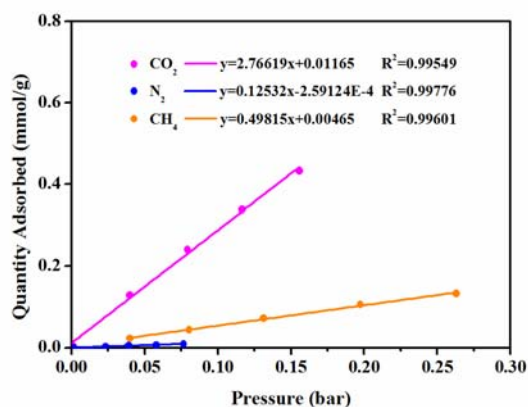
**Figure S14.** Adsorption selectivity of CO<sub>2</sub>/N<sub>2</sub> and CO<sub>2</sub>/CH<sub>4</sub> for polymer **6** calculated using Henry's law initial slope method according to the adsorption isotherms of CO<sub>2</sub> (pink), N<sub>2</sub> (blue), and CH<sub>4</sub> (orange) at 273.15 K and a low pressure coverage of less than 0.3 bar.



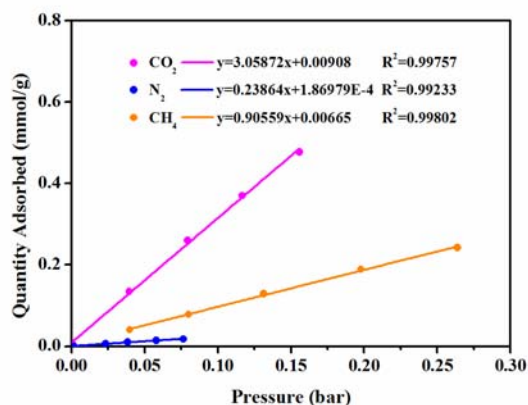
**Figure S15.** Adsorption selectivity of CO<sub>2</sub>/N<sub>2</sub> and CO<sub>2</sub>/CH<sub>4</sub> for polymer **1** calculated using Henry's law initial slope method according to the adsorption isotherms of CO<sub>2</sub> (pink), N<sub>2</sub> (blue), and CH<sub>4</sub> (orange) at 298.15 K and a low pressure coverage of less than 0.3 bar.



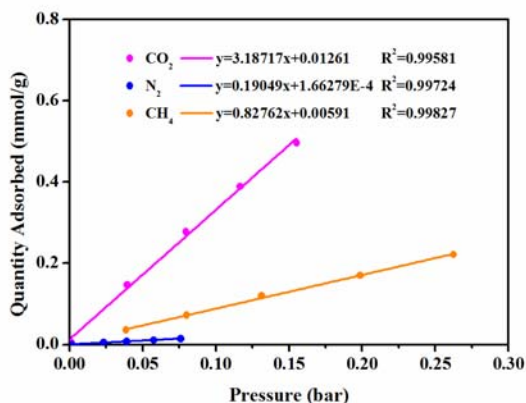
**Figure S16.** Adsorption selectivity of CO<sub>2</sub>/N<sub>2</sub> and CO<sub>2</sub>/CH<sub>4</sub> for polymer **2** calculated using Henry's law initial slope method according to the adsorption isotherms of CO<sub>2</sub> (pink), N<sub>2</sub> (blue), and CH<sub>4</sub> (orange) at 298.15 K and a low pressure coverage of less than 0.3 bar.



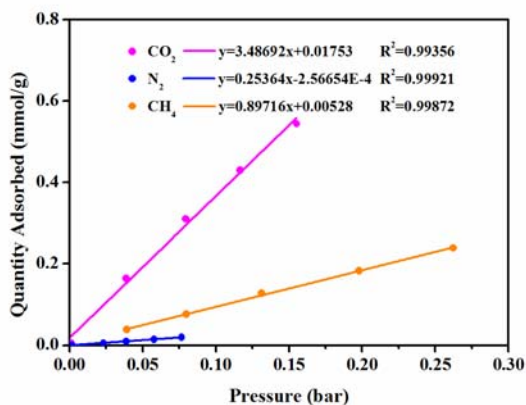
**Figure S17.** Adsorption selectivity of CO<sub>2</sub>/N<sub>2</sub> and CO<sub>2</sub>/CH<sub>4</sub> for polymer **3** calculated using Henry's law initial slope method according to the adsorption isotherms of CO<sub>2</sub> (pink), N<sub>2</sub> (blue), and CH<sub>4</sub> (orange) at 298.15 K and a low pressure coverage of less than 0.3 bar.



**Figure S18.** Adsorption selectivity of CO<sub>2</sub>/N<sub>2</sub> and CO<sub>2</sub>/CH<sub>4</sub> for polymer **4** calculated using Henry's law initial slope method according to the adsorption isotherms of CO<sub>2</sub> (pink), N<sub>2</sub> (blue), and CH<sub>4</sub> (orange) at 298.15 K and a low pressure coverage of less than 0.3 bar.



**Figure S19.** Adsorption selectivity of CO<sub>2</sub>/N<sub>2</sub> and CO<sub>2</sub>/CH<sub>4</sub> for polymer **5** calculated using Henry's law initial slope method according to the adsorption isotherms of CO<sub>2</sub> (pink), N<sub>2</sub> (blue), and CH<sub>4</sub> (orange) at 298.15 K and a low pressure coverage of less than 0.3 bar.



**Figure S20.** Adsorption selectivity of CO<sub>2</sub>/N<sub>2</sub> and CO<sub>2</sub>/CH<sub>4</sub> for polymer **6** calculated using Henry's law initial slope method according to the adsorption isotherms of CO<sub>2</sub> (pink), N<sub>2</sub> (blue), and CH<sub>4</sub> (orange) at 298.15 K and a low pressure coverage of less than 0.3 bar.

$$\text{Yield \%} = \frac{m_1 \text{ (g)}}{m_2 \text{ (g)}} \times 100\%$$

Where  $m_1$  is the weight of polymers **1**, **3**, and **5** measured after drying in a vacuum oven at 70 °C for 24 h,  $m_2$  is the weight of monomer 1, 4-diphenylbenzene; and  $m_1$  is the weight of polymers **2**, **4**, and **6** measured after drying in a vacuum oven at 70 °C for 24 h,  $m_2$  is the weight of monomer 1, 3-diphenylbenzene.

**Equation S1** The yield estimation of polymer materials from polymer **1** to polymer **6**.