

## Supplementary Information

### **Compressible and monolithic microporous polymer sponges prepared via one-pot synthesis**

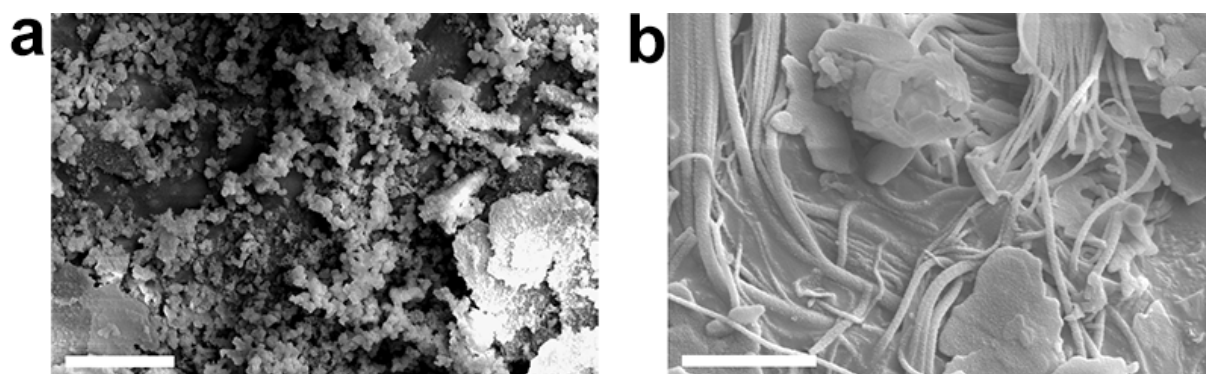
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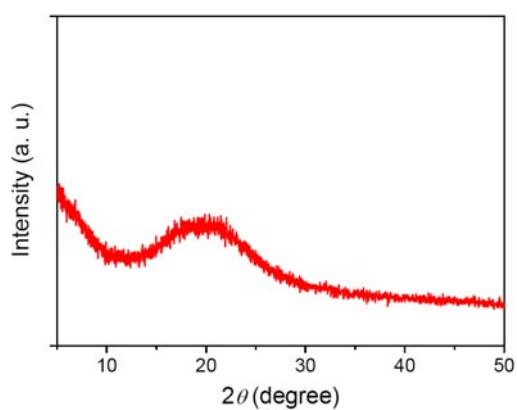
College of Engineering, Seoul National University

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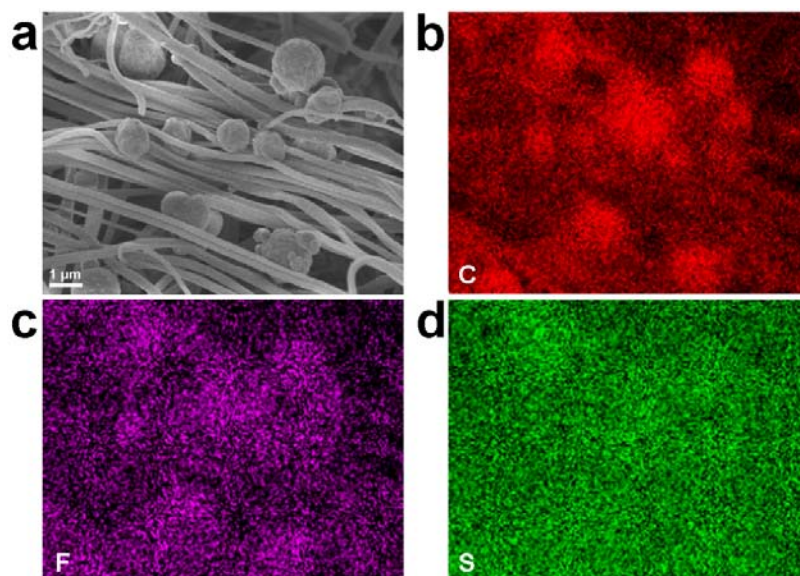
E-mail: [jichang@snu.ac.kr](mailto:jichang@snu.ac.kr)



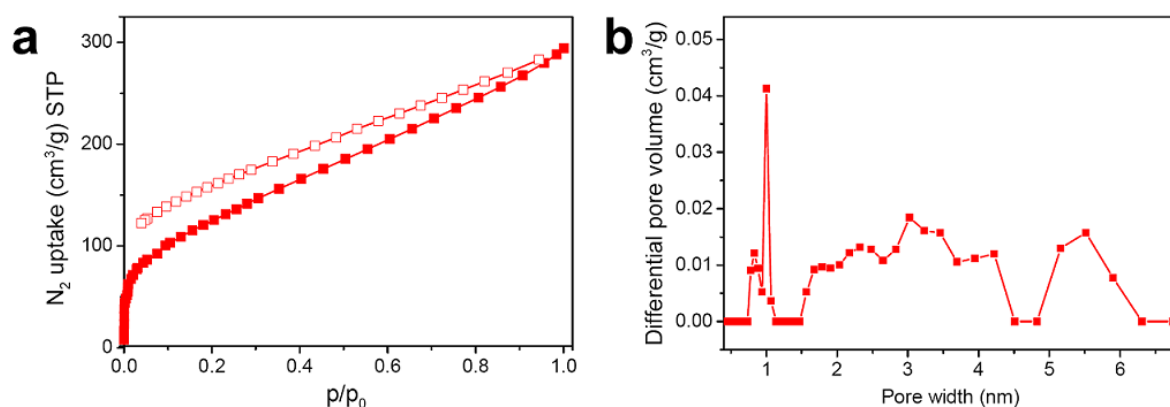
**Supplementary Figure S1. SEM images of the polymers.** SEM images of the polymers obtained by the reaction of 2,2'-(1,4-phenylene)bis(5-bromothiophene) and TEB (the molar ratio = 0.8:1) in toluene/DMF (1:1 v/v) (a) and in toluene (b) at 100 °C for 24 h (scale bar = 5  $\mu\text{m}$ ).



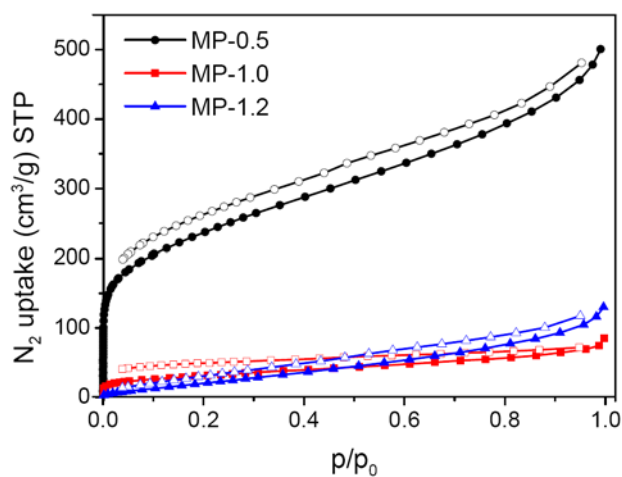
**Supplementary Figure S2. PXRD measurement.** PXRD pattern of MP-0.8.



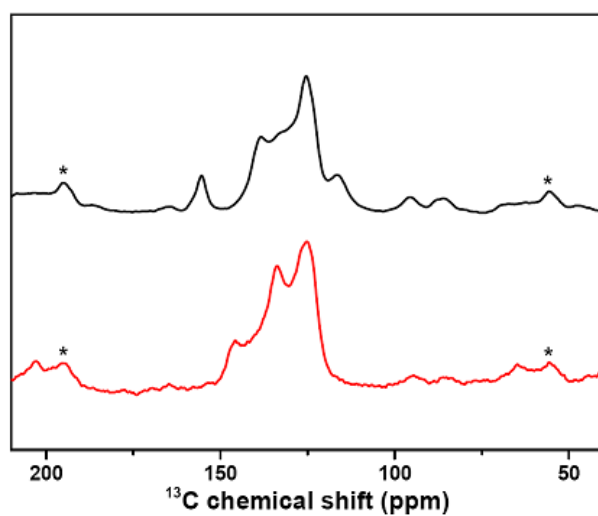
**Supplementary Figure S3. EDS measurement.** (a) SEM image and EDS elemental maps of (b) carbon, (c) fluorine, and (d) sulfur for **MP-0.8**.



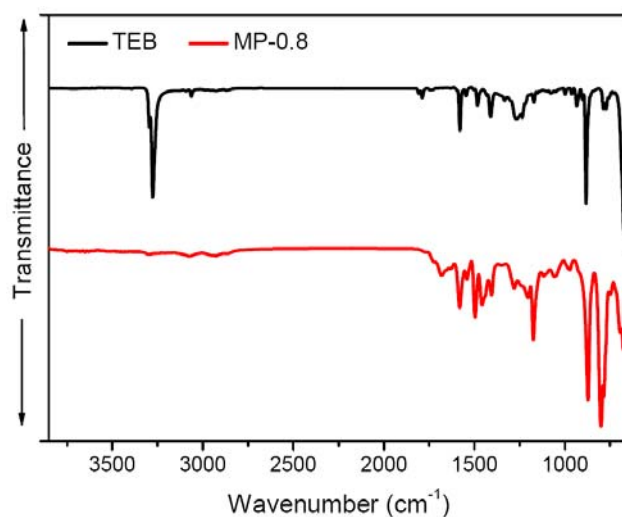
**Supplementary Figure S4. Microporosity of MP-0.8.** (a)  $N_2$  adsorption–desorption isotherms of **MP-0.8** measured at 77 K. (b) NL-DFT pore size distribution of **MP-0.8**. The total pore volume and micropore volume of **MP-0.8** were  $0.454 \text{ cm}^3 \text{ g}^{-1}$  at  $p/p_0 = 0.99$  and  $0.157 \text{ cm}^3 \text{ g}^{-1}$  at  $p/p_0 = 0.10$ , respectively.



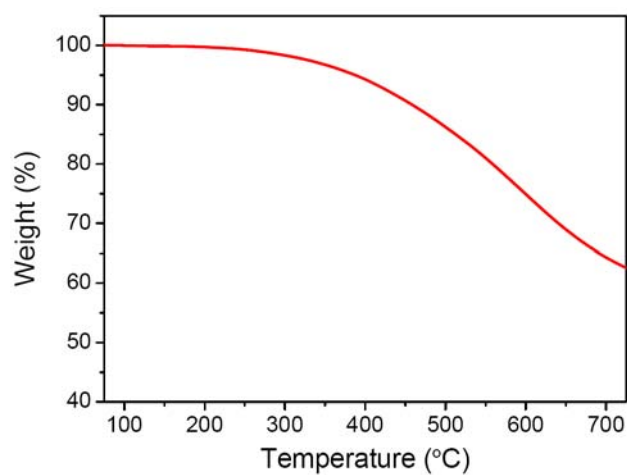
**Supplementary Figure S5. Microporosities of MPs.** N<sub>2</sub> adsorption-desorption isotherms of **MP-0.5**, **MP-1.0**, and **MP-1.2**.



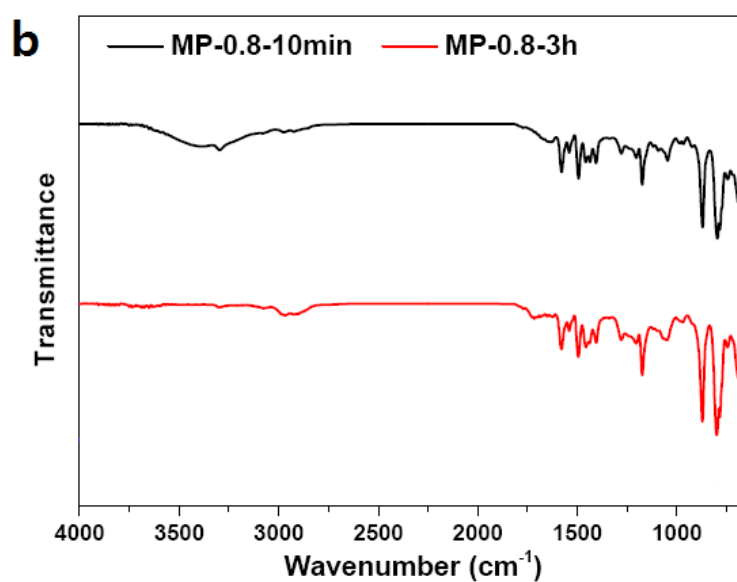
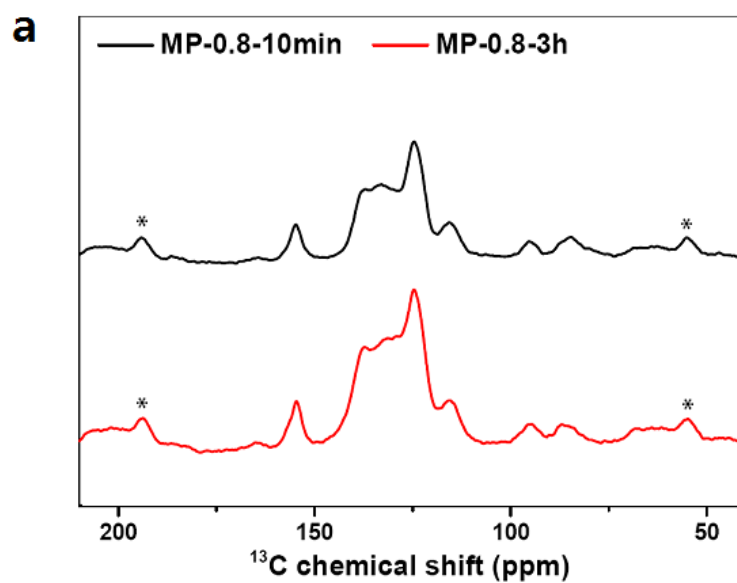
**Supplementary Figure S6. NMR measurement.** Solid state <sup>13</sup>C CP/MAS NMR spectrum of **MP-0.8** (black line) and the polymer obtained by the reaction of 2,2'-(1,4-phenylene)bis(5-bromothiophene) and TEB (the molar ratio = 0.8:1) in toluene/DMF (1:1 v/v) at 100 °C for 24 h (red line).



**Supplementary Figure S7. FT-IR measurement.** FT-IR spectra of **TEB** and **MP-0.8**. For the monomer, the FT-IR spectrum was recorded on a PERKIN ELMER Spectrum GX I using a KBr pellet. For the polymer, the FT-IR spectrum was recorded on a Thermo Scientific Nicolet 6700 FT-IR spectrometer using attenuated total reflectance (window ZnSe/diamond).



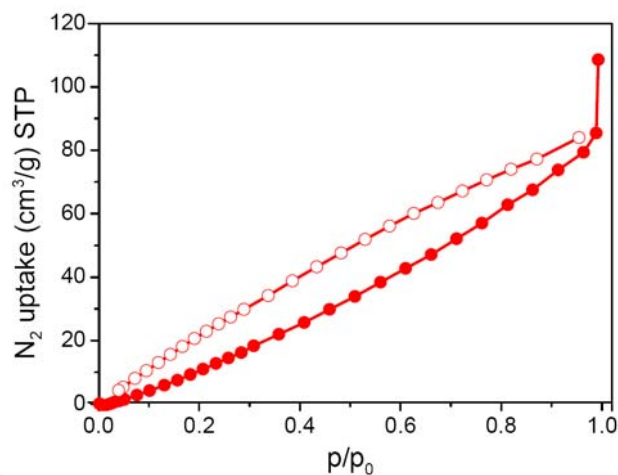
**Supplementary Figure S8. TGA measurement.** TGA curve of **MP-0.8**.



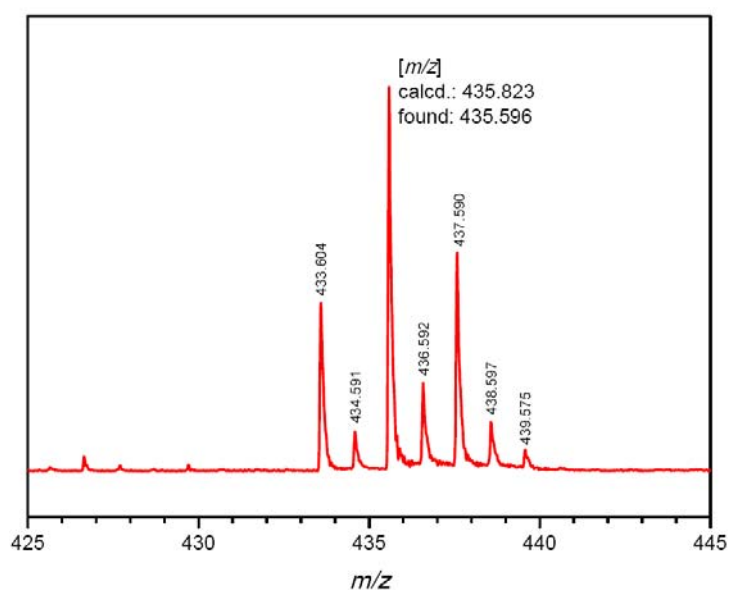
**c**

	carbon	hydrogen	sulfur
MP-0.8-10min	69.39	2.65	12.23
MP-0.8-3h	69.33	2.70	11.84

**Supplementary Figure S9. Characterization of the polymers.** (a) Solid state  $^{13}\text{C}$  CP/MAS NMR spectra, (b) FT-IR spectra and (c) elemental analysis results for **MP-0.8-10min** and **MP-0.8-3h**.



**Supplementary Figure S10. Macroporosity of urethane sponge.**  $N_2$  adsorption-desorption isotherms of the urethane sponge used in this study.  $V_{tot} = 0.168 \text{ cm}^3 \text{ g}^{-1}$  at  $p/p_0 = 0.99$  and  $V_{micro} = 6.34 \times 10^{-3} \text{ cm}^3 \text{ g}^{-1}$  at  $p/p_0 = 0.10$ .



**Supplementary Figure S11. MALDI-TOF MS spectrum of PBT-Br.** Calculated  $[m/z]$ : 435.823 (100%), 433.825 (51%), 437.820 (49%), 436.826 (10%), 438.824 (7%), 434.828 (4%), 439.816 (4%); Found: 435.596 (100%), 433.604 (44%), 437.590 (57%), 436.592 (23%), 438.597 (13%), 434.591 (11%), 439.816 (5%).

**2,2'-(1,4-Phenylene)bisthiophene.** This compound was prepared following the procedure for the preparation of **PBT** in 72% yield.  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ , ppm):  $\delta$  7.62 (s, 4H), 7.34 (d,  $J = 3.6$  Hz, 2H), 7.29 (d,  $J = 5.1$  Hz, 2H), 7.10 (t, 2H).  $^{13}\text{C}$  NMR (400 MHz,  $\text{THF-d}^8$ , ppm):  $\delta$  144.73, 134.65, 129.05, 127.04, 125.89, 124.20. Analysis (calcd, found for  $\text{C}_{14}\text{H}_{10}\text{S}_2$ ): C (69.38, 69.35), H (4.16, 4.19), S (26.46, 26.43).

**Synthesis of 2,2'-(1,4-phenylene)bis(5-bromothiophene).** This compound was prepared following the procedure for the preparation of **PBT-Br** in 93% yield.  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ , ppm):  $\delta$  7.51 (s, 4H), 7.08 (d,  $J = 3.9$  Hz, 2H), 7.05 (d,  $J = 3.9$  Hz, 2H). The collection of  $^{13}\text{C}$  NMR data was precluded due to sparing solubility. Analysis (calcd, found for  $\text{C}_{14}\text{H}_8\text{Br}_2\text{S}_2$ ): C (42.02, 42.19), H (2.02, 2.04), S (16.03, 16.15).