

Rich oxygen atoms decorative conjugated microporous polymers for carbon dioxide capture

## **Experimental**

### **Materials**

1,4-dioxane, mesitylene, acetic acid (HOAc), and others were purchased from J&K Scientific, Tokyo Chemical Industry Co., Ltd, and other companies.

### **Characterization**

Methods : The infrared spectra were recorded from 500 to 3500  $\text{cm}^{-1}$  on an Thermo Fisher Scientific Nicolet iS50 FT-IR by using KBr pellets. Field emission scanning electron microscopy was recorded on a FEI Apreo S HiVac microscope. Powder X-ray diffraction data were performed on a PANalytical BV Empyrean diffractometer by depositing powder on glass substrate, from  $2\theta = 2.0^\circ$  to  $35^\circ$  with  $0.02^\circ$ . Thermogravimetric analysis (TGA) was performed on a Mettler-Toledo TGA with the heating at a rate of  $10^\circ\text{C min}^{-1}$  from  $35^\circ\text{C}$  to  $800^\circ\text{C}$  under nitrogen. Nitrogen sorption and carbon dioxide isotherms were measured at 77 K with a Micromeritics ASAP2460.

### **Synthesis of O-CMP-1**

A Pyrex tube (10 mL) was added with s-indacene-1,3,5,7(2H,6H)-tetraone (21.4 g, 0.10 mmol), 4,4',4''-(1,3,5-triazine-2,4,6-triyl)tribenzaldehyde (10.8 mg, 0.067 mmol), dioxane (0.6 mL), mesitylene (0.6 mL), and acetic acid (6 M, 0.1 mL). The system was degassed via freeze-pump-thaw three times to remove the air from the mixture. The Pyrex tube was closed, stirred, and heated at 120 °C for 3 days. The O-CMP-1 was obtained via filtered, washed with acetone and tetrahydrofuran, soxhleted via tetrahydrofuran, and dried under vacuum to yield an O-CMP-1 sample (79%).

### **Synthesis of O-CMP-2**

A Pyrex tube (10 mL) was added with s-indacene-1,3,5,7(2H,6H)-tetraone (21.4 mg, 0.10 mmol), 4',4''',4''''-(1,3,5-triazine-2,4,6-triyl)tris((1,1'-biphenyl)-4-carbaldehyde) (11.9 mg, 0.067 mmol), dioxane (0.6 mL), mesitylene (0.6 mL), and acetic acid (6 M, 0.1 mL). The system was degassed via freeze-pump-thaw three times to remove the air from the mixture. The Pyrex tube was closed, stirred, and heated at 120 °C for 3 days. The O-CMP-2 was obtained via filtered, washed with acetone and tetrahydrofuran, soxhleted via tetrahydrofuran, and dried under vacuum to yield an O-CMP-2 sample (82%).

### **Synthesis of O-CMP-3**

A Pyrex tube (10 mL) was added with s-indacene-1,3,5,7(2H,6H)-tetraone (21.4 mg, 0.10 mmol), 4',4''',4''''-(1,3,5-triazine-2,4,6-triyl)tris((1,1'-biphenyl)-4-carbaldehyde) (14 mg, 0.067 mmol), dioxane (0.6 mL), mesitylene (0.6 mL), and acetic acid (6 M, 0.1 mL). The system was degassed via freeze-pump-thaw three times to remove the air from the mixture. The Pyrex tube was closed, stirred, and heated at 120 °C for 3 days. The O-CMP-3 was obtained via filtered, washed with acetone and tetrahydrofuran, soxhleted via tetrahydrofuran, and dried under vacuum to yield an O-CMP-3 sample (86%).

### **Stability experiment**

CMPs samples were dispersed in THF, DMF, water, aqueous HCl (1 M) and NaOH (1 M) solutions at room temperature for 72 h. The samples were collected by filtration, and washed with THF, water, and acetone for three times. The samples were dried under vacuum at 80 °C overnight.

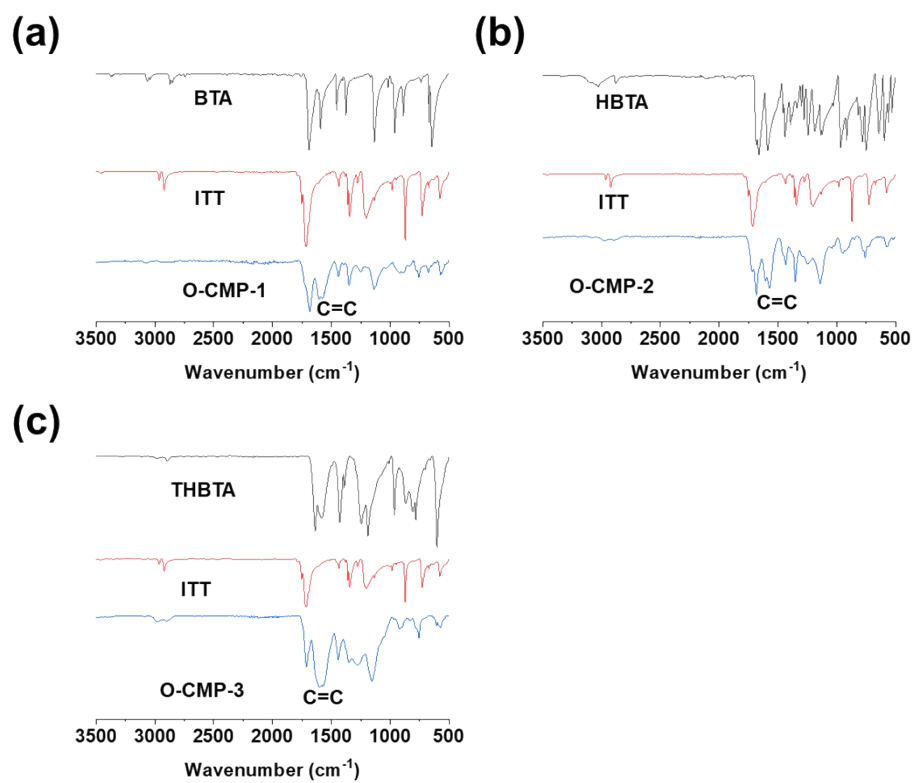


Fig. S1. FT IR spectra of O-CMPs and building units.

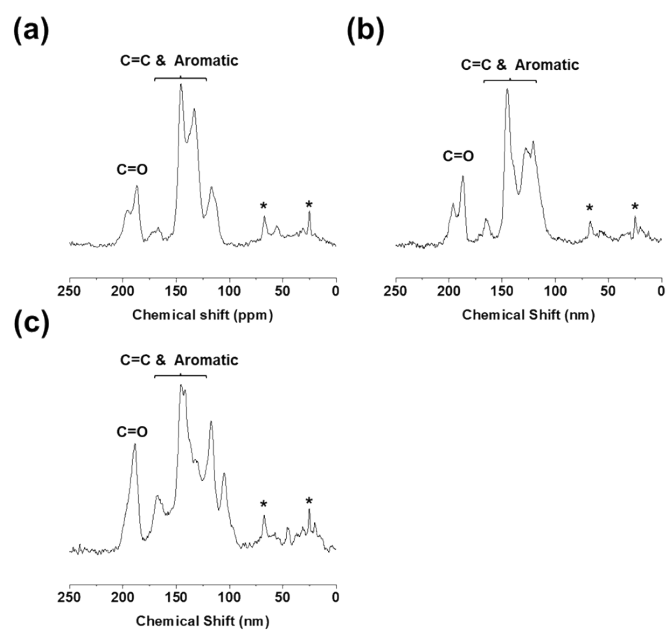


Fig. S2.  $^{13}\text{C}$  solid state NMR spectra of (a) O-CMP-1, (b) O-CMP-2, and (c) O-CMP-3.

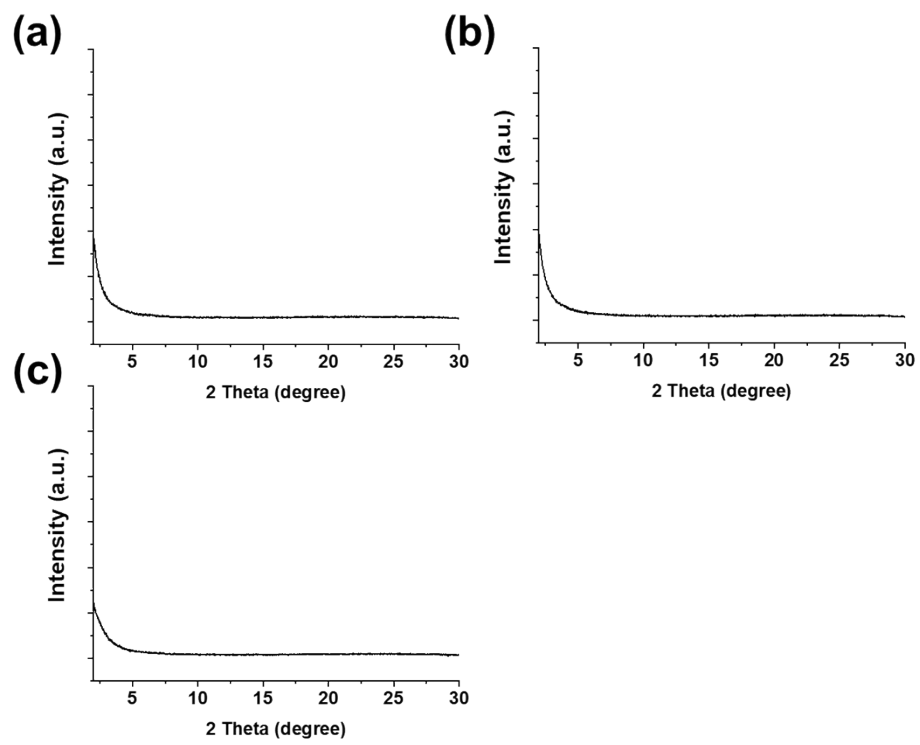


Fig. S3. PXR D patterns of (a) O-CMP-1, (b) O-CMP-2, and (c) O-CMP-3.

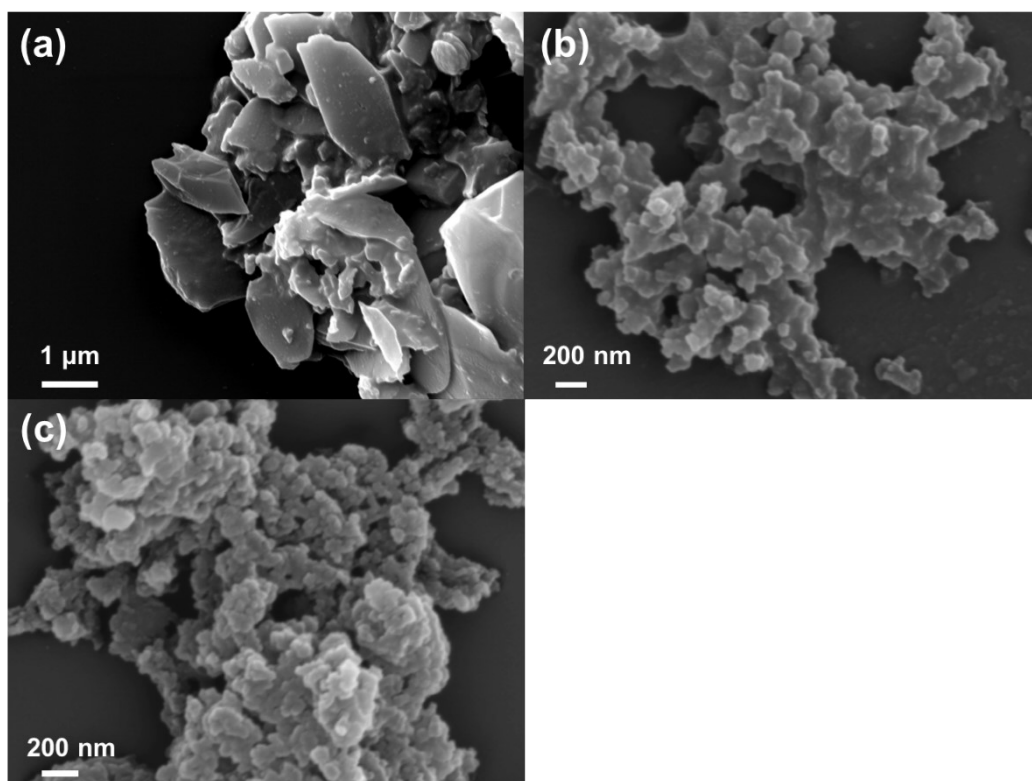


Fig. S4. FE SEM images of (a) O-CMP-1, (b) O-CMP-2, and (c) O-CMP-3.

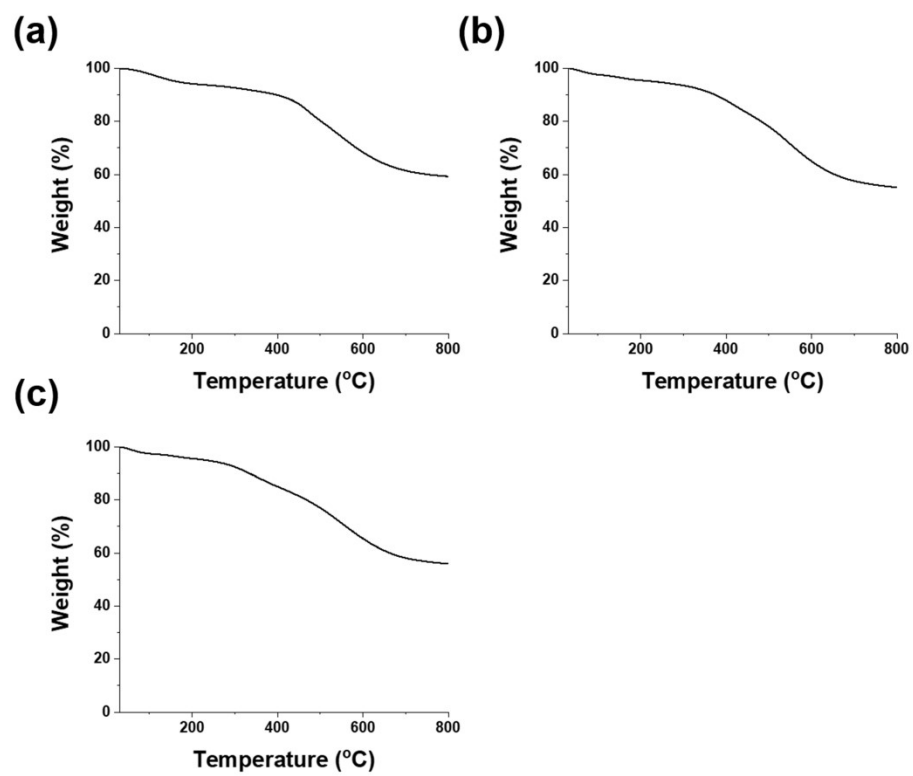


Fig. S5. TGA curves of (a) O-CMP-1, (b) O-CMP-2, and (c) O-CMP-3.



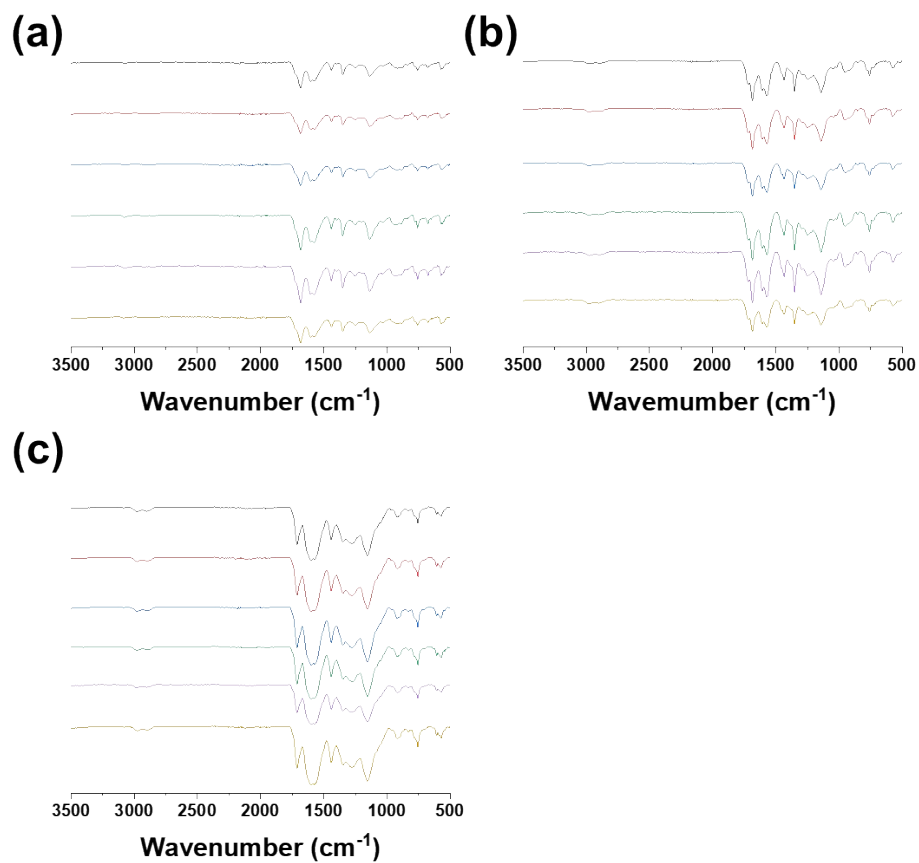


Fig. S6. FT IR spectra of (a) O-CMP-1, (b) O-CMP-2, and (c) O-CMP-3 under different conditions. (As-synthesized: black; THF: red; DMF: blue; Water: green; NaOH (1 M): purple; HCl (1 M): yellow).

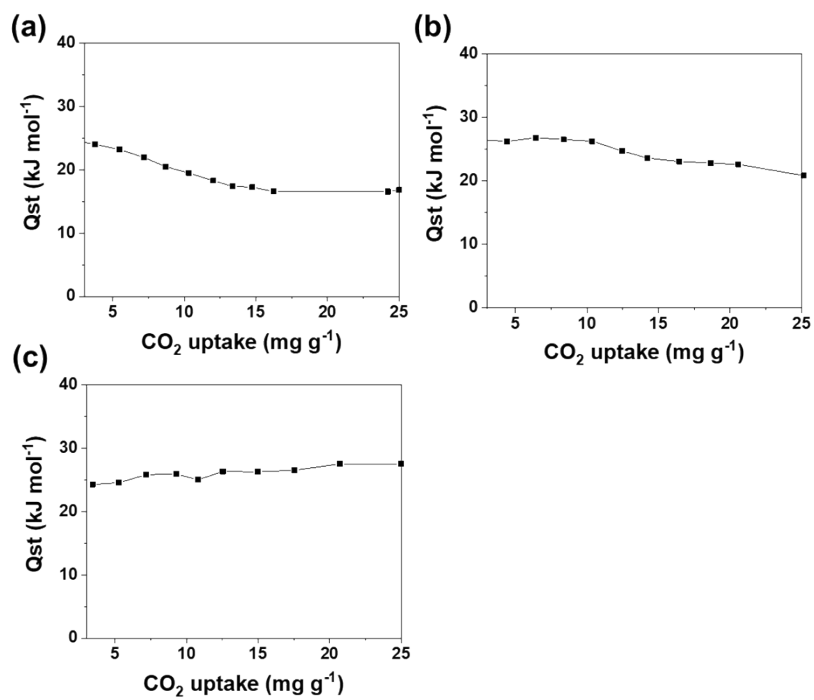


Fig. S7.  $Q_{st}$  of  $CO_2$  of (a) O-CMP-1, (b) O-CMP-2, and (c) O-CMP-3.