

Supplementary Information

**Molecular Engineering Approach to Pore-Adjustable
Nanoporous Carbons with Narrow Distribution for High-
Performance Supercapacitors**

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Experimental Section

Materials

2-Hydroxybenzyl alcohol (HA, 98%) and trifluoroacetic acid (CF₃COOH) were purchased from Aladdin. Tetrabutylammonium fluoride (TBAF, 1 M in THF) and tetraethyl orthosilicate (TEOS) were provided by Macklin Inc. Furfuryl alcohol (FA), toluene, potassium hydroxide (KOH), hydrofluoric acid (HF), dichloromethane, hexane, and diethyl ether were obtained from Sinopharm Chemical Reagent Co., Ltd., China. FA and TEOS were freshly distilled prior to use. All chemicals used are of analytical grades.

Synthesis of tetrafurfuryloxysilane (TFOS)

TEOS (9.0 mL, 40 mmol), FA (14.1 mL, 160 mmol) and 0.3 wt% KOH were well mixed under an Ar atmosphere. The reaction mixture was stirred for 3 h at 80°C. The by-products were then removed by distillation under vacuum to obtain a viscous yellow-brown liquid. Finally, the twin monomer of TFOS was achieved via crystallizing from the viscous liquid followed by recrystallization with diethyl ether.

NPC-1 derived from twin polymerization of TFOS

2.49 g TFOS (6 mmol) was slowly added into a mixture of CF₃COOH (17.8 μL) and toluene (5.0 mL) under an Ar atmosphere. The mixture was then stirred for 3 h at 80°C to obtain silica/poly(furfuryl alcohol) (SiO₂/PFFA) composites. SiO₂/PFFA was transferred into a quartz tube (GSL-1500X-OTF) and heated to 900°C at a rate of 3°C/min under a flowing Ar atmosphere. The sample was kept for 2 h at this temperature. Finally, NPC-1 was obtained by etching off SiO₂ in HF followed by washing with distilled water and drying at 80°C in vacuum for 12 h.

Synthesis of 2,2'-spirobi[4H-1,3,2-benzodioxasiline] (SBOS)

14.00 g HA (112 mmol) was dissolved in 40.0 mL toluene under an Ar atmosphere. This solution was heated to 85°C under stirring followed by adding TBAF (40 μL). Afterwards, TEOS (12.4 mL, 56 mmol) was slowly dropped into the above mixture and kept stirring for 1.5 h. The by-product of ethanol and the residual toluene were removed by distillation under vacuum to obtain a viscous light-yellow liquid. Finally, the twin monomer of SBOS was achieved via crystallizing from the viscous liquid followed by recrystallization with hexane.

NPC-2 derived from twin polymerization of SBOS

5.68 g SBOS (21 mmol) was molten at 85°C under an Ar atmosphere and then CF₃COOH (64 μL, 0.84 mmol) was added dropwise into the melt under stirring. The

resulted mixture was stirred for another 3 h at 85°C to yield a transparent monolith. The monolith was washed by dichloromethane and then dried at 60°C to obtain silica/poly(2-hydroxybenzyl alcohol) (SiO₂/PHBA) composites. SiO₂/PHBA was transferred into a quartz tube and kept at 900°C for 2 h under an Ar atmosphere. NPC-2 was finally obtained by etching off SiO₂ in HF, washing with distilled water, and vacuum-drying at 80°C for 12 h.

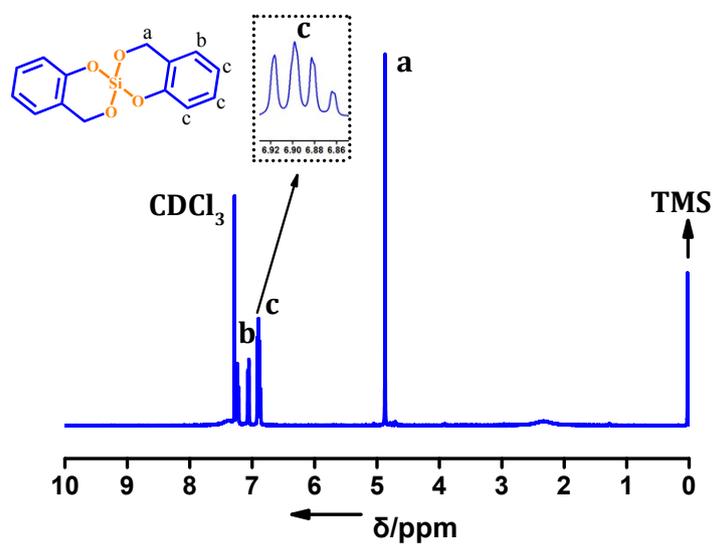
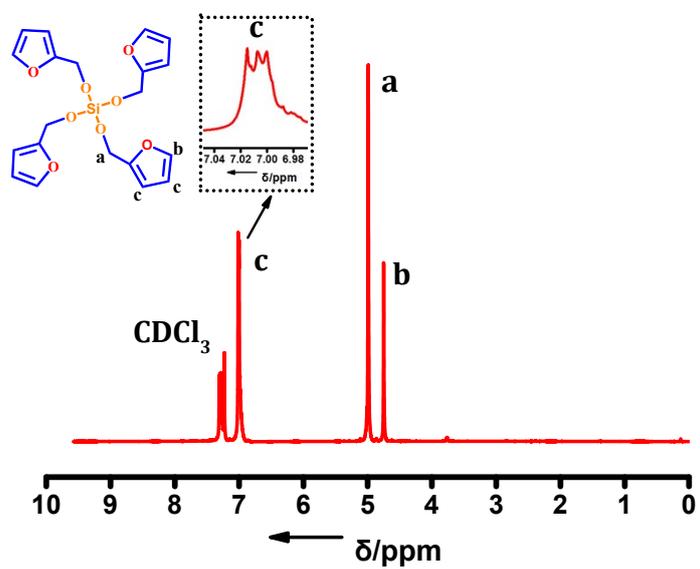
General characterization

The morphologies of porous carbon materials were characterized on a Tecnai G20 transmission electron microscope (TEM) at an acceleration voltage of 200 kV. Nitrogen adsorption-desorption measurements were performed by an American QDS-MP-30 instrument at 77 K. The specific surface area and pore size distribution were calculated based on the Brunauer-Emmett-Teller (BET) and Nonlocal Density Functional Theory (NLDFT), respectively. X-ray diffraction (XRD) measurement was carried out using a Rigaku D/Max2400 diffractometer equipped with a Cu K α radiation source. Raman spectra were collected on a Thermo Scientific DXR spectrometer with an excitation laser $\lambda=532$ nm. X-ray photoelectron spectroscopy (XPS) was recorded on a PHI MultiPak spectrometer with an Mg K α X-ray source.

Electrochemical measurements

Electrochemical measurements were carried out on a CHI 660E electrochemical workstation (CH Instruments, Inc) using 1 M H₂SO₄ as the electrolyte at room temperature. The Pt foil and saturated calomel electrode (SCE) were used as the counter electrode and reference electrode, respectively. The working electrodes were fabricated by a common slurry coating procedure. Typically, active materials (80%), acetylene black (10%), and PVDF (10%) were dispersed in *N*-methylpyrrolidinone (NMP) and then stirred to obtain a uniform slurry. The slurry was spread on the carbon cloth (1 cm²) and then infrared dried for 12 h to obtain the working electrode. Before measurements, the as-prepared working electrodes were soaked in 1 M H₂SO₄ overnight. Cyclic voltammetry (CV) measurements were conducted at the scan rate of 25 to 500 mV s⁻¹. Galvanostatic charge/discharge (GCD) experiments were performed at the current density of 0.5 to 10 A g⁻¹. Electrochemical impedance spectroscopies (EIS) were conducted in the frequency range of 10⁵-0.01 Hz.

Supplementary Figures



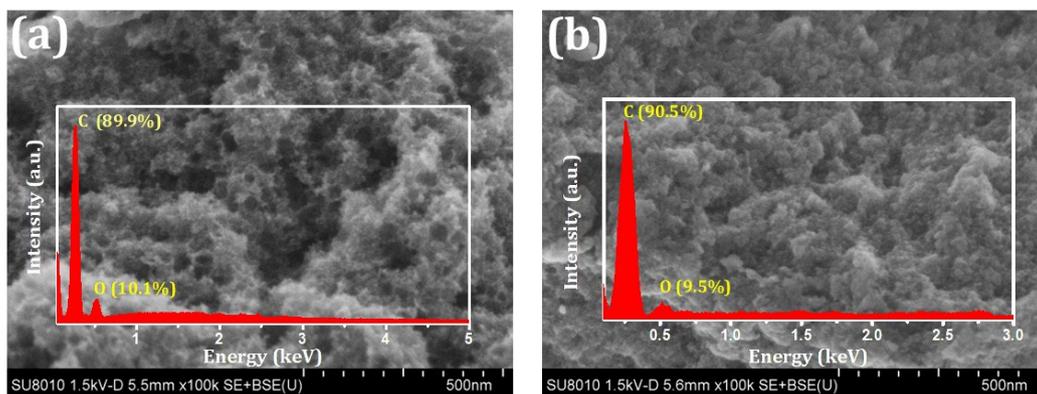


Fig. S3 SEM images of (a) NPC-1 and (b) NPC-2 and their corresponding EDS spectra.

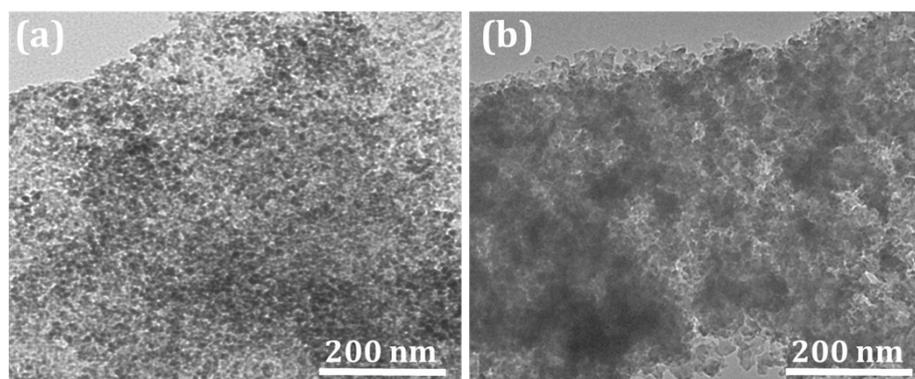


Fig. S4 Typical TEM images of SiO₂/polymer nanocomposites produced by twin-polymerization of (a) TFOS and (b) SBOS.

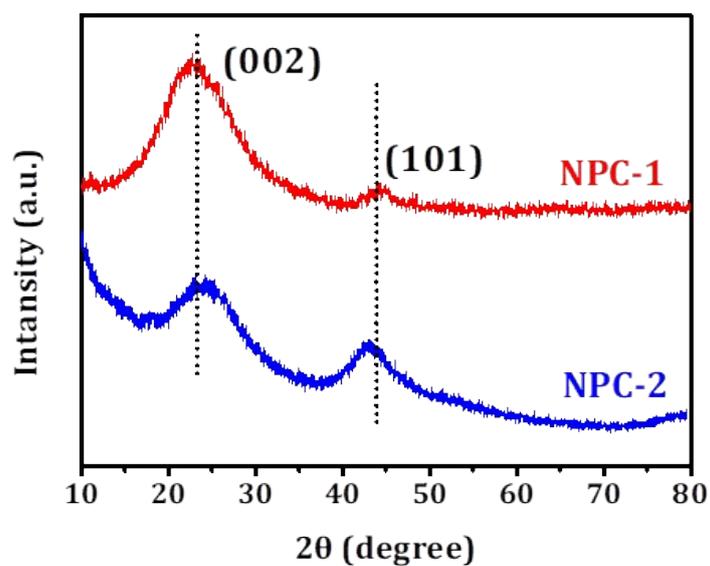


Fig. S5 XRD patterns of NPC-1 and NPC-2.

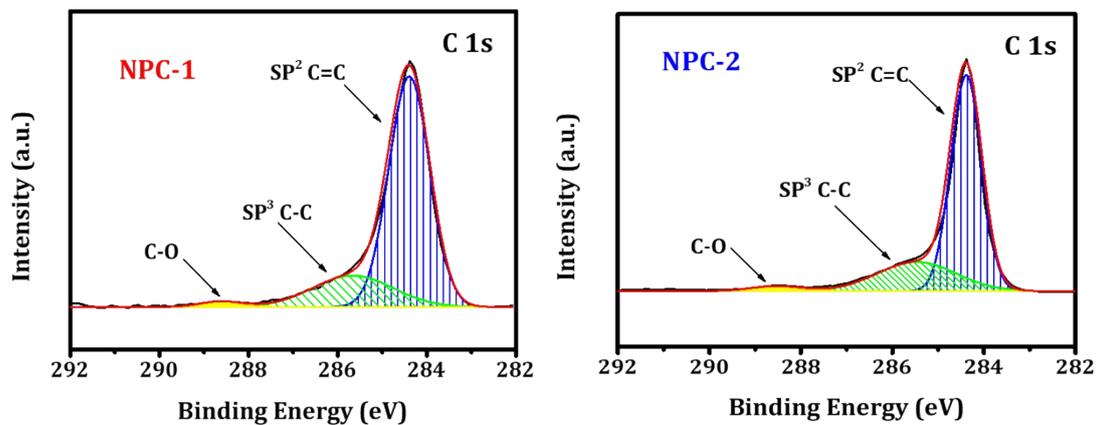


Fig. S6 C1s core-level spectra of NPC-1 and NPC-2

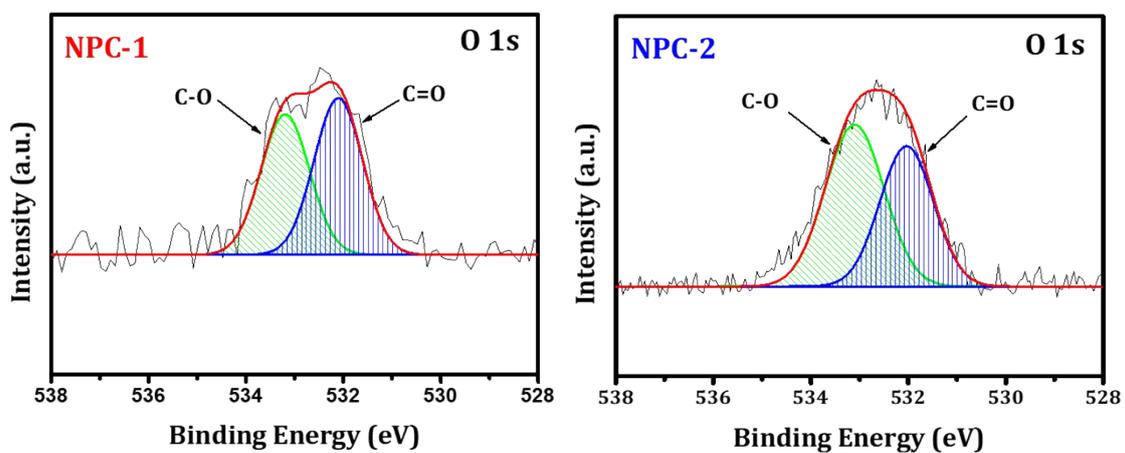


Fig. S7 O1s core-level spectra of NPC-1 and NPC-2.

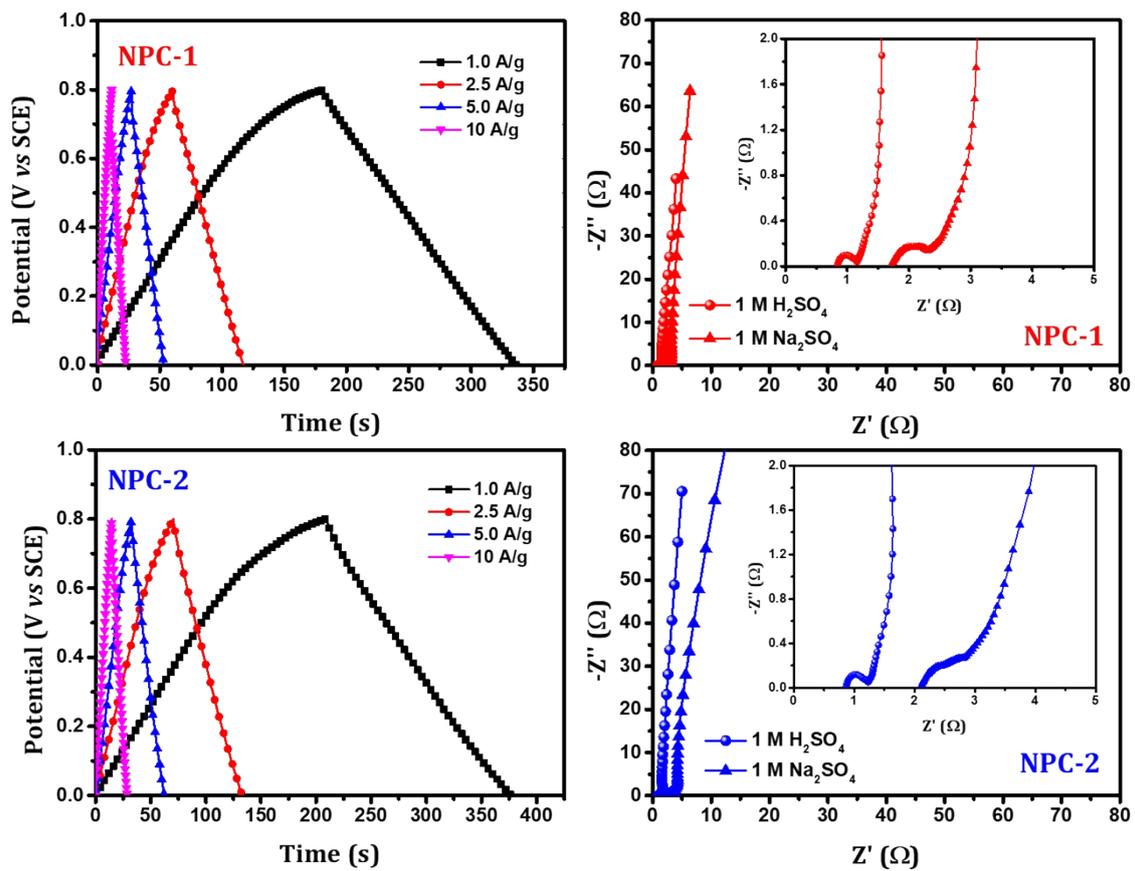


Fig. S8 GCD curves and EIS Nyquist plots of NPC-1 and NPC-2 measured in the 1.0 M Na₂SO₄ electrolyte.