Supporting Information (SI) for RSC Advances

Direct Polymer Templating Synthesis of Mesoporous Carbon via Spinodal Decomposition

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1. Experimental

Materials. Commercially available polyethylene glycol (PEG) with different average molecular weight: 1 kDa, 2 kDa, 4 kDa, and 8 kDa received from Fluka, along with 20 kDa and 14 kDa PEG and poly ethylene oxide of 100 kDa and 200 k Da received from Sigma-Aldrich, were all used as received. The carbon precursor was phloroglucinol (>99.0%, Aldrich) and formaldehyde (37 wt.%, Sigma-Aldrich). Ethanol, 190 proof, (Decon labs) and aqueous HCl (37 wt.%, Sigma-Aldrich) were used without further purification.

Synthesis. 2.3 g of phloroglucinol, 5.3 g of 1 kDa PEG was dissolved under intense stirring in 130 mL ethanol and 1 g HCl (37 wt.%) while heating to reflux. At reflux, 2.3 g of aqueous formaldehyde was added. The cloud point occurred within 3 min after addition of formaldehyde. The reaction mixture was stirred for a total of 1.5 h, which resulted in solid masses. The ivory polymer solids were washed with ethanol and dried in an oven at 80 °C overnight. Carbonization was carried out in a tube furnace under flowing Ar at a heating rate of 2 °C/min to 850 °C and held for 2 h before cooling to ambient temperature.

2. Characterization Methods

Electron Microscopy. Scanning electron microscopy (SEM) and transmission electron microscopy (TEM) images were obtained using a Hitachi HD 2000 STEM microscope at 200 kV. Samples for STEM were prepared by dispersion casting, where the sample was dispersed in ethanol with the grid and allowed to dry at ambient temperature before analysis.

 N_2 Adsorption-Desorption. Mesoporous sample measurements carried out at 77K using a Micromeritics Tristar 3000 analyzer and microporous sample measurements on Quantachrome AS-1. Prior to measurement, samples were degassed at 170 °C under N_2 for at least 6 h. The specific surface area was calculated using the Brunauer-Emmett-Teller (BET) method. The pore volume was estimated from singe point adsorption at a relative pressure of 0.995. The average pore diameter was determined from the adsorption branch, according to the Barrett-Joyner-Halanda (BJH) method using Kruk-Jaroniec-Sayari (KJS) correction.

Small Angle X-Ray Scattering (SAXS). SAXS data were collected with a Panalytical Empyrean diffractometer with Cu K α radiation.

2.1 SEM and TEM images



Figure S1. The SEM image (a) and TEM image (b) of mesoporous carbon derived from PF-PEG 2 k M_w after carbonization at 850 °C for 2h.



Figure S2. The SEM image (a) and TEM image (b) of mesoporous carbon derived from PF-PEG 4 k M_w after carbonization at 850 °C for 2h.



Figure S3. The SEM image (a) and TEM image (b) of mesoporous carbon derived from PF-PEG 8 k M_w after carbonization at 850 °C for 2h.



Figure S4. The SEM image (a) and TEM image (b) of mesoporous carbon derived from PF-PEG 20 k M_w after carbonization at 850 °C for 2h.



Figure S5. The SEM image (a) and TEM image (b) of mesoporous carbon derived from PF-PEG 100 k M_w after carbonization at 850 °C for 2h.



Figure S6. The SEM image (a) and TEM image (b) of mesoporous carbon derived from PF-PEG 200 k M_w after carbonization at 850 °C for 2h.



Figure S7. The SEM image (a) and TEM image (b) of mesoporous carbon derived from PF-PEG 14 k M_w after carbonization at 850 °C for 2h, referenced as PEG concentration of 0.63 mM.



Figure S8. The SEM image (a) and TEM image (b) of mesoporous carbon derived from PF-PEG 14 k M_w after carbonization at 850 °C for 2h, referenced as PEG concentration of 1.4 mM.

2.2 Additional N₂ adsorption isotherms



Figure S10. The N_2 adsorption isotherms corresponding to (a) carbon produced using the typical synthesis without the addition of HCl (b) as synthesized polymer (c) carbon produced using the typical synthesis without the addition of PEG (d) carbon produced using PF-PEG 1k polymer.





Figure S11. Small-angle scatterings of mesoporous carbons using respective MW PEG.