

Large-scale synthesis of triblock copolymer-templated ordered mesoporous carbon spheres with controlled particle size

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Characterization

The morphologies of ordered mesoporous carbon particles were observed by scanning electron microscopy (SEM) using JEOL JSM-6360LV microscope. The mean particle sizes were measured with a light scattering analyzer using Malvern Mastersizer 2000 apparatus. The XRD patterns were acquired on a Rigaku D/max 2550 diffractometer operating at 40 KV and 20 mA using Cu K α radiation ($\lambda=1.5406\text{\AA}$). The spheres were ground into powder for convenience measurement. Nitrogen adsorption/desorption isotherms were measured at 77 K with a Micromeritics ASAP2020 analyzer. Before the measurements, the samples were degassed in vacuum at 200 °C for at least 6 h. The Brunauer-Emmett-Teller (BET) method was utilized to calculate the specific surface areas using adsorption data in a relative pressure range from 0.04 to 0.2. The total pore volume was estimated from the adsorbed amount at a relative pressure P/P_0 of 0.985. The micropore volume was calculated from the $V-t$ plot method. The pore size distributions (PSD) were derived from the desorption branches of isotherms by using the Barrett-Joyner-Halenda (BJH) model. Transmission electron microscopy (TEM) experiments were conducted on a JEOL 2100 microscope operated at 200 kV. The samples were ground into powder and suspended in ethanol and then supported onto a holey carbon film on a Cu grid. The thermogravimetric analyses (TA Instrument Q600 Analyser) of the sample were carried out at a nitrogen flow rate of 100 ml/min. The samples were heated to 1000 °C with a rate of 10 °C/min.

Thermogravimetric analyses (TGA) were employed to investigate the degradation

behavior of cubic and hexagonal mesostructured particles prepared by the EISA combined with suspension polymerization method and polymer films prepared by the conventional EISA method. The triblock copolymer F127 starts to decompose at 325 °C and finishes at 410 °C with only negligible residue (0.9 wt. %). At this temperature, about 49.2 % and 33.7 % weight remain for cubic and hexagonal mesostructured particles prepared by the EISA combined with suspension polymerization method, which is much closed to the values of 48.3 % and 33.2 % for the conventional EISA method. At the final carbonization temperature of 800 °C, the same weight residues present for both synthesis methods. This result suggests the macroscopical composition of organic-organic polymer spheres is not affected by emulsion process.

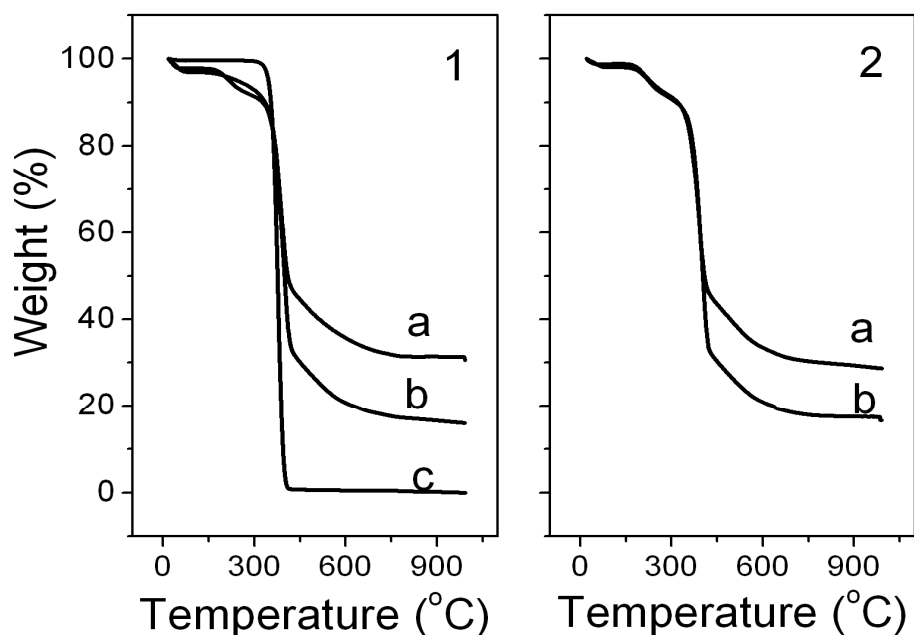


Fig. S1 TGA curves of polymer spheres (1) prepared by the EISA combined with suspension polymerization method and polymer films (2) prepared by the conventional EISA method. (a) cubic mesostructure prepared at the mole ratios of the reactants were 0.008 F127: 1 resorcinol: 1.5 furfural: 0.005 HCl: 0.1 methenamine: 20 EtOH, (b) hexagonal mesostructure prepared at the mole ratios of the reactants were 0.015 F127: 1 resorcinol: 1.5 furfural: 0.005 HCl: 0.1 methenamine: 20 EtOH, and (c) triblock copolymer 127