Electronic Supplementary Material (ESI) for Chemical Communications

Supporting information

A Facile Photo-induced Synthesis of COOH Functionalized Meso-macroporous Carbon Film and its Excellent Sensing Capability for Aromatic Amines

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1. Experimental details

Suspensions of monodispersed polystyrene microspheres (PSs) (2.5 wt % in water, surfactant-free) were obtained from polysciences Inc. Large-area (ca. 1 cm2) ordered PS colloidal monolayers with different sphere size (200 nm, 500nm, 1000nm) were synthesized by the spin-coating method on a custom-built spin-coater. 5 μl of 0.2 M glucose solution with 0.5% H_2SO_4 was taken as carbon precursor solution. Here, the H_2SO_4 was used as dehydration reagent to remove the water from glucose during conversion of glucose to carbon. As depicted in Scheme 1, a droplet of the carbon precursor was added onto the monolayer of polystyrene spheres (PSs) template with a quantitative micropipette, which infiltrates into the interstices between the substrate and the colloidal monolayer. Subsequently, the substrate, with the monolayer and solution, was dried at 100 °C and 160 °C for 6 hours each. At this time, the precursor solution becomes highly viscous which suggests the formation of some aromatic compounds or oligosaccharides and is denoted as the "polymerization" step. Meso and macroporosity in the carbon film was created by burning out the PS spheres at a high temperature in an inert atmosphere. The resulting solids were calcined at 300 °C for 2 h with the heating rate of 2 °C /min to remove the polystyrene template and carbonized at 900 °C for 2 h with the heating rate of 4 °C /min in argon atmosphere. The high temperature treatment is also necessary to connect the carbon walls via an intermolecular dehydration of linear or branchlike oligosaccharides. Finally, meso-macroporous carbon film can be obtained. Here, it should be noted that the morphology and structure of the film can be easily tuned by controlling the template, such as the layer number and the size of PS sphere.

The morphology of the films prepared at different synthesis conditions was observed on a Hitachi S-4800 filed emission scanning electron microscope (FESEM). The TEM micrographs were obtained on a JEL-2100F filed emission high-resolution transmission electron microscope (TEM) equipment. The X-ray diffraction (XRD) pattern was collected on a Rigaku diffractometer using Cu Kα (λ=0.154 nm) radiation. FT-TR spectra were recorded on a Nicolet Nexus 670 instrument. Raman spectra were measured on a Jobin-Yvon Horiba T-64000. The adsorption properties of porous carbon materials

toward various volatile substances were detected using a quartz crystal microbalance (QCM) (USI System, Japan). The QCM resonators coated by gold on both surfaces were taken as electrodes and the change of one frequency corresponds to that of about 0.95 ng in mass. For our experiment, the carbon film was detached from the substrate in water and transferred to the QCM resonators. Then, it was inserted in the QCM instrument resonator holder. At the same time, desired volatile liquids (toluene, hexane, aniline, acetic acid, 30% ammonia, ethanol and water) (10 ml) in a glass bottle were allowed in the QCM instrument. To prevent vapor leakage, the QCM instrument was fully covered during the in situ adsorption measurement. After the gas adsorption process, both the cover and the glass bottle containing the testing solution were removed exposing the film to the normal air. All experiments were carried out in an air conditioned room at 25 °C. Ozonation of the porous material was carried out using ozone cleaner (Filgen UV253S system, Japan). The oxygen, UV lamp and nitrogen were introduced in order with about 15, 30, and 5 minutes, respectively. The ozonation time was varied between 0 to 60 minutes.

2. Transferring of a meso-macroporous carbon film on the water surface

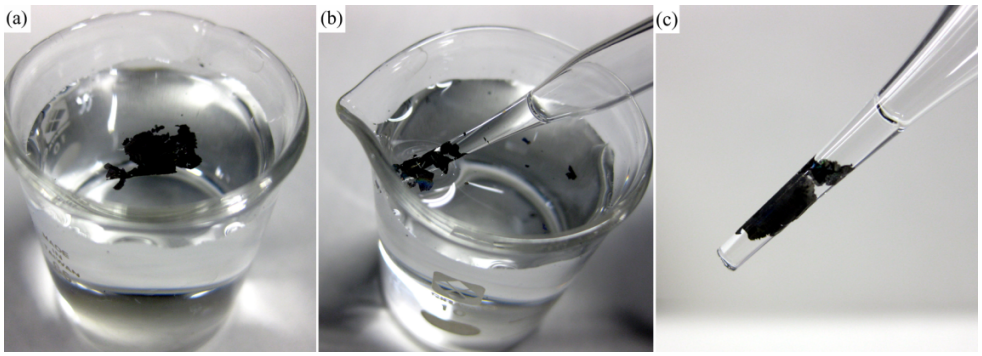

Figure S1. Images depicting the transferring of a meso-macroporous carbon film from silicon substrate onto curved glass tube. (a) Lift-off onto water, (b) Pick-up of the carbon film with curved glass tube, (c) The carbon film on a curved glass tube.

3. Images of meso-macroporous carbon films with different macropore size

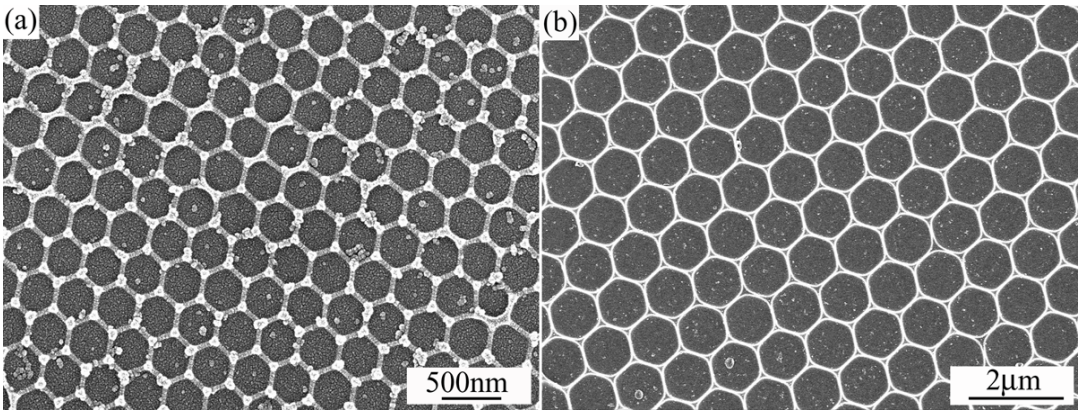

Figure S2. FESEM images from top view of monolayer meso-macroporous carbon films with different macropore size. (a) 200 nm, and (b)1000 nm. Small separated carbon particles were found in the case of 200 nm template. This might be because we used glucose solution with the same concentration for all the three kinds of templates (200, 500, 1000), and the adopted concentration is a bit higher for smaller-sized template, which could lead to extra absorption of glucose on PS template and thus the formation of separated carbon particles after the calcination treatment.

4. Small-angle X-ray scattering

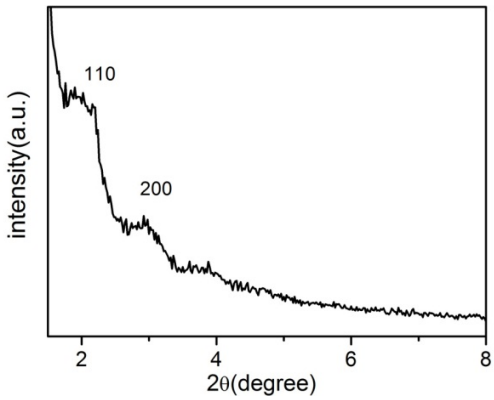

Figure S3. Small-angle X-ray scattering of monolayer meso-macroporous carbon films with 476 nm macropore size.

5. Small-angle X-ray scattering

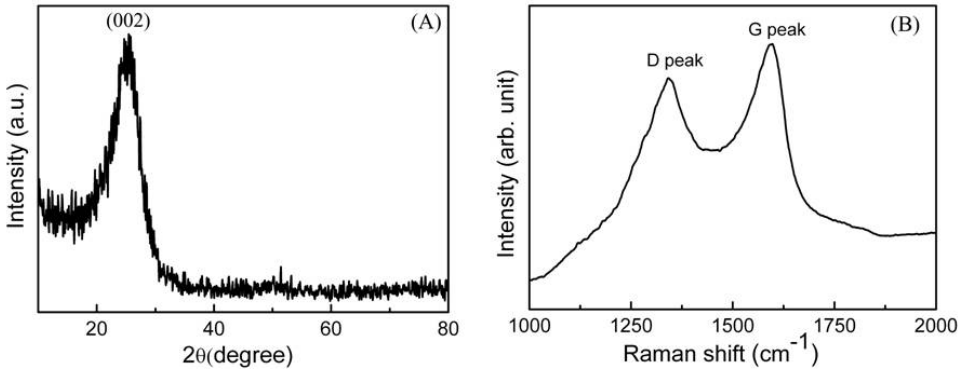

Fig. S4. Wide-angle XRD pattern (A) and Raman spectra (B) of meso-macroporous carbon film with 476 nm pore size.

6. Adsorption curve of meso-macroporous carbon without ozonation treatment toward to aniline.

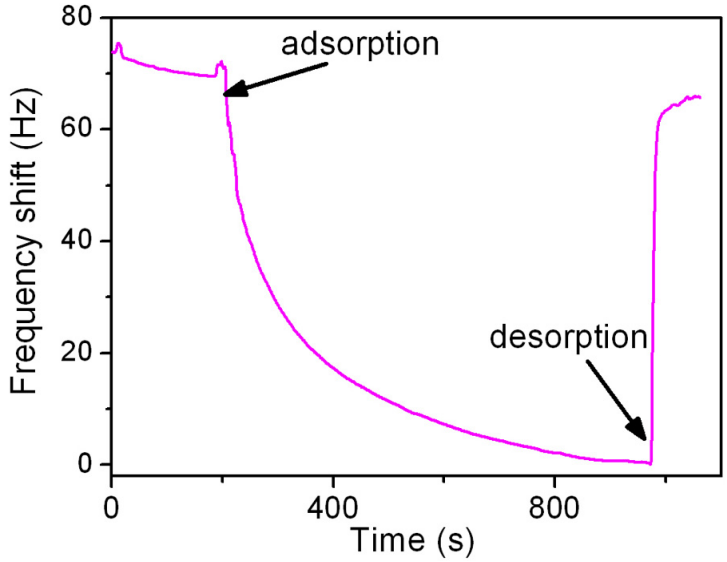

Figure S5. Adsorption curve of meso-macroporous carbon without ozonation treatment toward to aniline.

7. FT-IR spectra of meso-macroporous carbon films with and without ozonation treatment

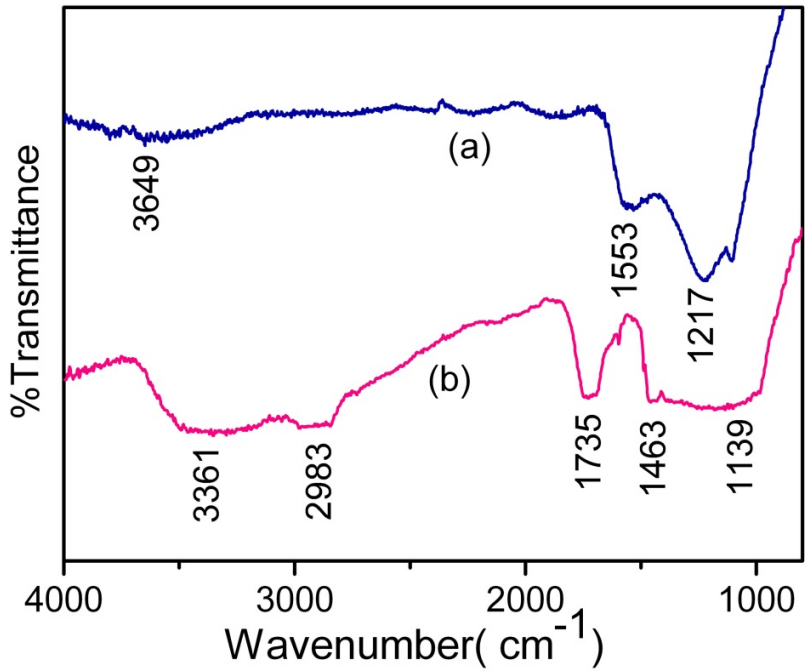

Figure S6. FT-IR spectra of meso-macroporous carbon films before (a) and after ozonation (b) for 30 min.