Electronic Supplementary Information (ESI) for RSC Advances

One-pot hydrothermal synthesis and reusable oil-adsorbing properties of porous carbonaceous monoliths using multi-walled carbon nanotubes as templates

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Experimental

Materials: Multi-walled carbon nanotubes (MWNTs) with an outer diameter between 10 and 20 nm, produced by chemical vapor deposition (CVD) methods, were purchased from Chengdu Institute of Organic Chemistry, Chinese Academy of Sciences, China. Deionized (DI) water was used throughout all the experiments. All the other reagents were supplied from Sinopharm Chemical Reagent Co. Ltd. and used as received.

Synthesis of acid-treated MWNTs (t-MWNTs): The raw MWNTs were suspended in a concentrated sulfuric acid–nitric acid mixture (3:1 v/v), heated in an oil bath at 70°C for 4 h, and then diluted with DI water. The products were then filtered and washed by 5% hydrochloric acid in order to increase the content of carboxyl groups attached to the MWNT surfaces. The resulting solid was dried in a vacuum oven at 50 °C for 24 h to obtain the t-MWNTs.

Preparation of MWNT-carbon hybrid hydrogels and aerogels: 40 mg of the water-dispersible t-MWNTs were dispersed into 10 mL of DI water under sonication,

and then a desired amount of glucose was added into the upper suspension under stirring. The mixture was transferred into a Teflon-lined autoclave, and then treated hydrothermally at 180 ^{o}C for a definite period of time. After the autoclave was cooled down to room temperature, a dark-brown gel-like cylinder was formed on the bottom of the autoclave. The resultant MWNT-carbon hybrid hydrogel was carefully picked out from the autoclave and immersed in excess DI water several times to remove incompletely carbonized furan-like molecules. The gel-like samples were then kept in a laboratory freezer followed by freeze-drying to give the MWNT-carbon hybrid aerogel.

Characterization: FTIR spectra were recorded with a 4 cm^{-1} spectral resolution on a Nicolet Nexus 470 spectrometer equipped with a DTGS detector by signal-averaging 64 scans. IR samples were in the form of KBr pellets containing about 0.5 wt% predried samples. A scanning electron microscope (SEM, Tescan), performed at an acceleration voltage of 20 kV, was used to observe the morphology of fracture surface of the samples. The samples were coated with gold prior to SEM observation. Transmission electron microscopy (TEM) images were recorded on a Jeol JEM2100 TEM instrument operated under an acceleration voltage of 200 keV. The TEM samples of raw MWNTs, t-MWNTs, and MWNT-carbon hybrid aerogels were prepared by mounting the ethanol suspension on the copper grid and dried in air. Before investigating the selective adsorption behavior for lubricating oil, we labeled the lubricating oil with methyl red dye. Before and after adsorbing oil or other solvents, the as-prepared and thermally treated MWNT-carbon hybrid aerogels were weighed using a balance (Mettler Toledo XS204).

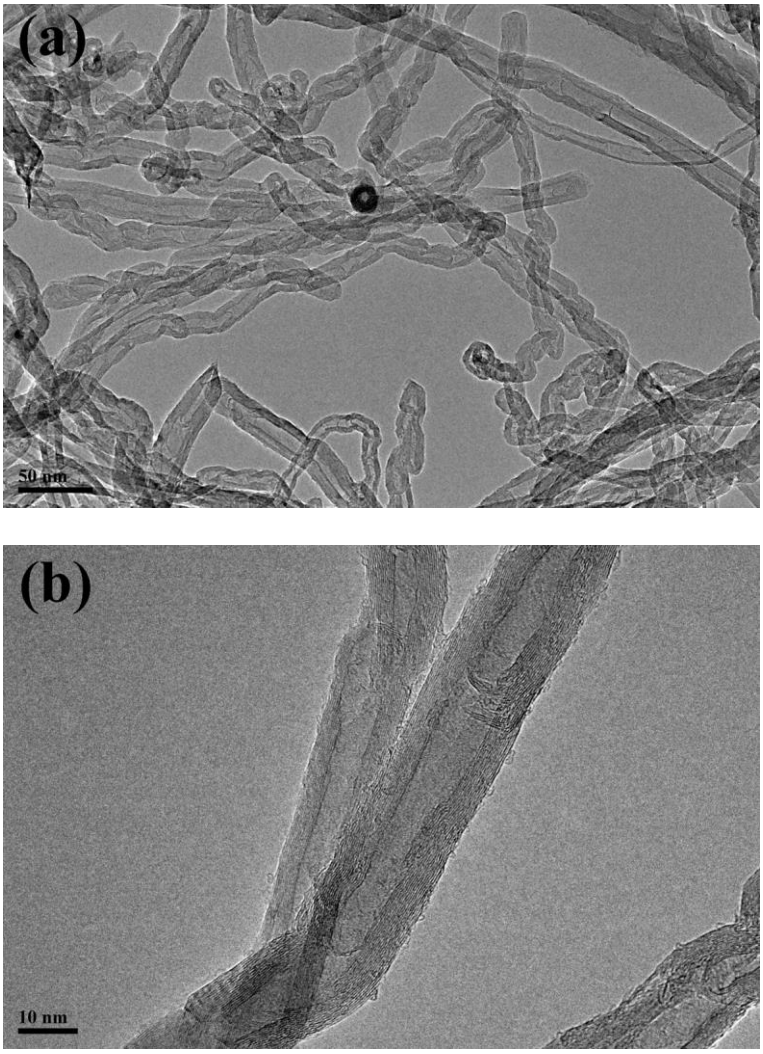

Figure S1. TEM images of the raw MWNTs at low and high magnifications.

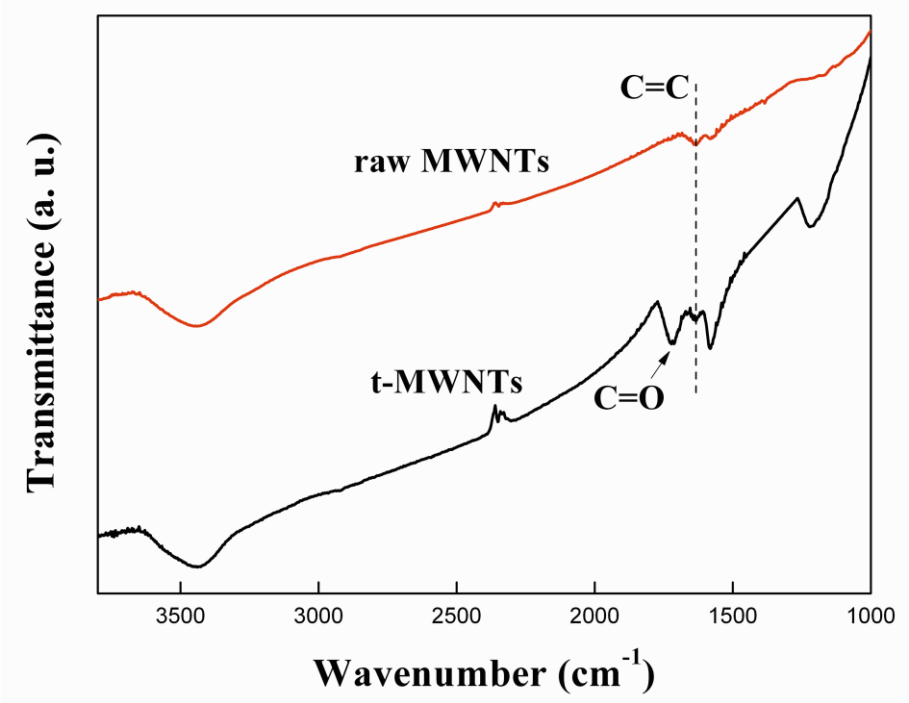

Figure S2. The FTIR spectra of the raw MWNTs and t-MWNTs, respectively. Curve of the t-MWNTs has peaks located at 1720 and 1250 cm^{-1}, corresponding to C=O and C-O stretching, respectively, indicating that a large amount of oxygen-containing groups has been introduced to the surface of MWNTs during acid treatment.

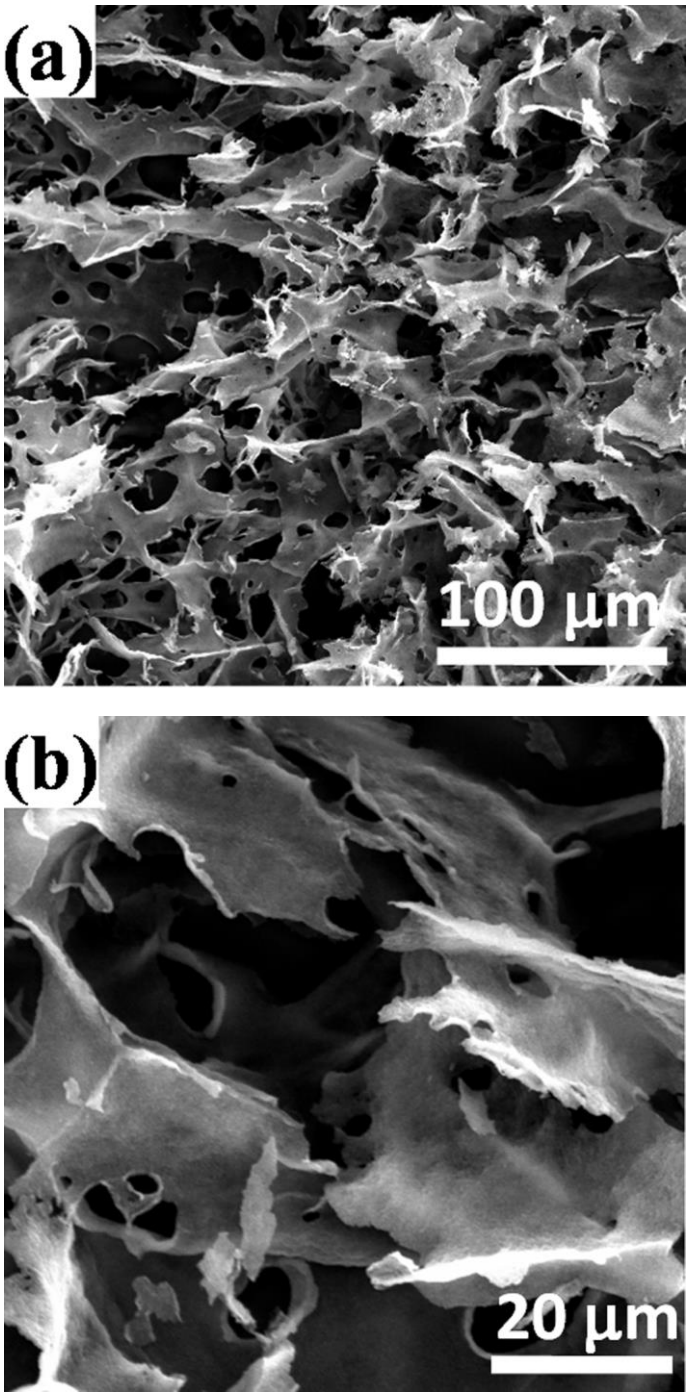

Figure S3. SEM images of the fracture surface of the thermally treated MWNT-carbon hybrid aerogel at low and high magnifications, exhibit a 3D network with pore sizes of tens of micrometers, indicating that the thermal treatments did not change the essential morphology of hybrid aerogels.

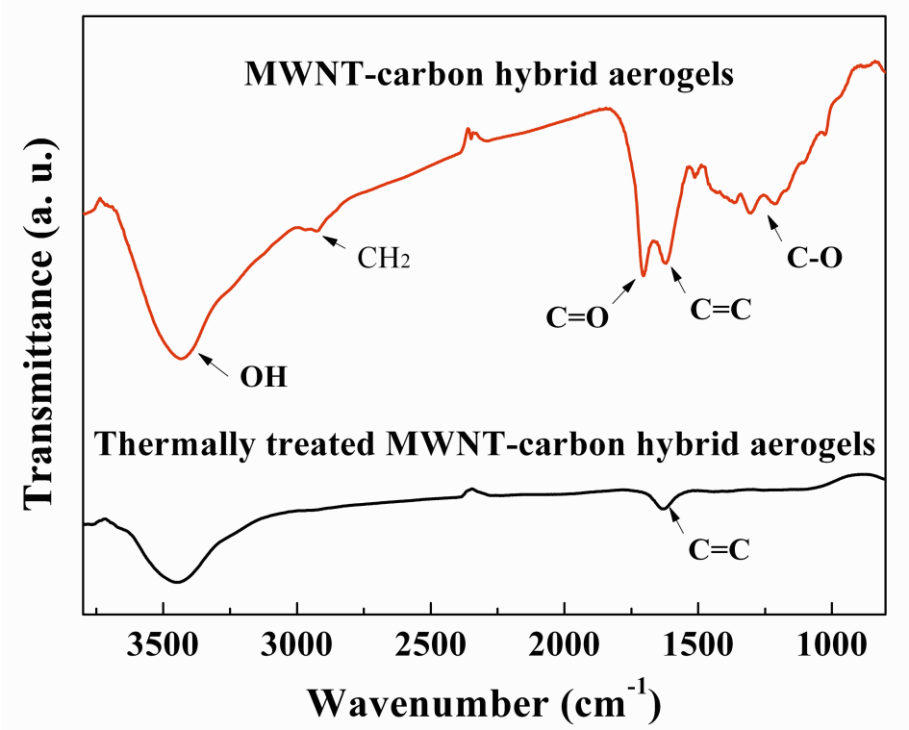

Figure S4. The FTIR spectra of the as-prepared and thermally treated MWNT-carbon hybrid aerogels, respectively. For the IR curve of the as-prepared MWNT-carbon hybrid aerogels, a broad adsorption band at 3410 cm^{-1} is assigned to the OH groups, and the peak centered at 1400 and 1730 cm^{-1} is the C-O and C=O characteristic stretching vibration, respectively, indicating the presence of the carboxyl and other oxygen-containing groups bound to the carbonaceous framework. For the IR curve of the thermally treated MWNT-carbon hybrid aerogels, the peaks ascribing to the oxygen-containing groups almost disappear.