Can Nitrones Functionalize Carbon Nanotubes?


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Material characterization (general consideration). MWCNTs (> 90% carbon basis) were provided by NANOCYL S.A. (Sambreville - BELGIUM) or Sigma-Aldrich®. Thermal gravimetric analysis measurements were performed on an EXSTAR Thermo Gravimetric Analyzer (TG/DTA) Seiko 6200 under N₂ atmosphere (50 mL/min) coupled with a ThermoStar™ GSD 301 T (TGA-MS) for MS gas analysis of volatiles. TEM analysis was performed using a Philips CM12 operating at 120 keV, with samples prepared by drop casting previously sonicated solutions or suspensions over graphite grating and images recorded with a CCD camera (Gatan 791). Atomic force microscopy was done using a Park System XE-100E AFM instrument, with samples prepared by spin-coating (4200 rpm, 20") previously sonicated sample solutions or suspensions on freshly cleaved mica substrates. The images were recorded with standard tips (Veeco Tips NCHV-A) in tapping mode at a scan rate of 1 Hz. The spectroscopic Raman measurements were carried out using the green line (514.5 nm) of an argon ion laser. Spectra were analyzed with a Spex Triplemate spectrometer, equipped with high resolution holographic gratings, and recorded using a liquid nitrogen cooled Horiba Jobin Yvon CCD camera. $I_D/I_G$ integrated intensity ratio for MWCNTs and f-MWCNTs have been calculated through a fitting procedure on the acquired spectra using the Peak Fitting module of Origin program. XRDP data were collected in the 10-60 region of 2theta using a Philips X’Pert PRO diffractometer with Cu Kalpha radiation (lambda= 1.5418Å). Elemental analyses were performed using a Thermo FlashEA 1112 Series CHNS-O elemental analyzer with an accepted tolerance of ± 0.4 units.
Figure S1. TGA-MS of f-MWCNTs from 60 to 800 °C. Isobutene (m/z = 56) and CO₂ (m/z = 44) evolution have been monitored throughout the range of temperatures.

Figure S2. TGA-MS of pristine MWCNTs from 60 to 800 °C. CO₂ (m/z = 44) evolution have been monitored throughout the range of temperatures.
Figure S3. TGA-DTG of f-MWCNTs from 60 to 800 °C.

Figure S4. TGA-DTG of nitronate 1 from 50 to 500 °C.
Figure S5. AFM image of f-MWCNTs

Figure S6. Raman spectra of pristine MWCNTs (black line) and f-MWCNTs (blue line) at 514.5 nm. Red line refers to a sample of MWCNTs which was treated in DMF at 160 °C for three days (standard reaction protocol) followed by the same washing/filtration/sonication/work-up procedures.
Figure S7. XRDP spectra of pristine MWCNTs (red line) and f-MWCNTs (black line).