

sodium deoxycholate. After the separation, 16 (1.5 mL) individual fractions were collected in each case by hand pipetting from the top in 0.75 mL increments.

[0094] Concentration of like fractions was typically performed by uniform filling of the appropriate centrifuge tube with the length separated SWCNT solution, followed by ultracentrifugation. Under these conditions, the sedimentation of the iodixanol polymer gradually causes the SWCNTs to be excluded both from the bottom (as the polymer is more dense) and the top (as the SWCNTs are more dense than the surfactant alone) of the tube. Dilution of the top one-half to two-thirds of the individual length fraction with additional stock surfactant solution, to lower the average density in the top part of the tube, dramatically speeds this process. Some of these concentrated fractions were then additionally processed by dialysis against 0.8% DOC solution in 25 k MWCO dialysis floats to remove the remaining iodixanol and to reduce the total surfactant concentration.

[0095] Ultraviolet-visible-near infrared (UV-Vis-NIR) absorbance spectroscopy was performed in transmission mode on a Perkin Elmer Lambda 950 UV-Vis-NIR spectrophotometer over the range of (2500 to 185) nm for SWCNT-surfactant solutions, and from (1450 to 325) nm for SWCNT-surfactant-iodixanol solutions. Measurements were typically performed on the extracted fractions in a 2 mm path length quartz cuvette. In all cases, the incident light was circularly polarized prior to the sample compartment, and the spectra corrected for both dark current and background. Data was recorded at 1 nm increments with an instrument integration time of at least 0.12 s per increment. The reference beam was left unobstructed, and the subtraction of the appropriate reference sample was performed during data reduction.

[0096] Dynamic light scattering (DLS) was performed in a temperature controlled cell maintained at 25° C. using a Brookhaven Instruments BI-200SM in VH (crossed polarizers) configuration with 532 nm excitation. Scattering was measured at a minimum of three different angles with a minimum of two repetitions. Dialyzed samples were typically used for these measurements. The correlation of scattering intensity in each case was fit to a double exponential, and the resultant inverse rotational relaxation time is related to the squared magnitude of the scattering vector and the rotational diffusion coefficient in accordance with formalism of Pecora. SWCNT length was obtained from D_{\perp} .

[0097] Tapping-mode atomic force microscopy (AFM) measurements were conducted in air using a Nanoscope IV system (Digital Instruments) operated under ambient conditions with 1 to 10 Ohm/cm, phosphorous (n) doped silicon tips (Veeco; RTE5P5, 125 μm length; 30 μm width, normal spring constant, 40 N/m; resonance frequency, 240 kHz to 300 kHz). Length separated, concentrated and dialyzed fractions were diluted 10x in water (18 MΩcm⁻¹) prior to being deposited (2 μL) onto plasma cleansed Si [1,1,1] wafers. After being allowed to dry, the entire sample was cleaned of surfactant with an ethyl acetate wash and wicking procedure to afford clear imaging conditions.

[0098] Raman spectra were collected in a collinear back-scattering configuration. An Ar⁺ laser (Coherent Innova Sabre with multi-line visible head) provided the excitation; approximately 20 mW of power was focused to a spot size of approximately 100 μm within the sample volume. Samples were measured in a single semi-micro spectrophotometer cell (NSG, 10 mm path length) that was held immobile for all of the measurements. The spontaneous Raman backscattered

light was collected with a triple grating spectrometer (Dilor XY800) and a liquid nitrogen cooled CCD detector. The signal was integrated for an appropriate time to obtain a signal to noise ratio greater than 50. The integration time for the CoMoCat fractions shown here was 10 s averaged over four scans. Data were collected with excitation at 514.5 nm. At the 514.5 nm excitation line Raman frequency shifts in the range (150 to 4000) cm⁻¹ were measured, with specific attention given to those between (150 and 2800) cm⁻¹. Data were corrected solely by scaling for incident laser intensity and by the subtraction of a small background, generally less than a few percent of the feature intensity.

[0099] Visible and NIR fluorescence were recorded using a Horiba Jobin Yvon nanolog-3 spectrofluorometer with a liquid N₂-cooled InGaAs detector. Emission spectra were corrected for the instrument's source spectral distribution, detector spectral response, and for the absorbance of the filter used to restrict scattered excitation light from the NIR monochromators and detector. Excitation wavelength were scanned in 5 nm increments unless otherwise noted using a 450 W xenon lamp through an 8 nm slit and emission collected at 90° in either 1, 2 or 4 nm increments through an 8 nm slit. To account for differences in concentration, fractions were diluted to a common absorbance of 0.05 per cm at 775 nm, and were measured in a 10 mm square quartz cuvette.

[0100] A schematic and photographs of aspects of preferred embodiment process are shown in FIG. 7. The band containing the SWCNTs is clearly visible. Measured lengths for the S-P95-02 CoMoCat SWCNTs shown in FIG. 7. And lengths from the collected fractions are presented in FIG. 8. Spectra, scaled for concentration at 775 nm are shown in FIG. 10A. When run to optimize the transient motion of the SWCNTs, high resolution of the separated lengths is achievable, and spectra showing well defined SWCNT peak features with increasing peak to baseline ratios are measured above the injection layer. SWCNT bundles, as determined by peak broadening, a red shift in peak location, and a decrease in peak absorption; remain in the injection layer and are typically not observed in centrifuged DOC solutions, but would be expected to fractionate downwards. High density impurities are seen to fractionate to the bottom of the tube and are not resuspended during fraction collection.

[0101] Specifically, FIG. 7 includes schematic illustrations and photographs of the length separation by centrifugation process for CoMoCat SWCNTs at 1257 Rad/s in DOC/iodixanol solution at 15° C. Longer nanotubes move further in response to the applied centrifugation, and thus separate up the tube.

[0102] As previously described, the strength of the optical transition peaks can also be used to calculate the (length weighted) average length of the separated fractions. In particular, the relationship between the peak to baseline ratio and length is known for the specific batch of S-P95-02 grade CoMoCat fractions used in this contribution from previous size exclusion chromatography separations. This relation is defined in previously noted equation (6):

$$\ell \text{ (nm)} \approx \left(\frac{\text{Absorbance}(984 \text{ nm})}{\text{Absorbance}(775 \text{ nm})} - 0.842 \right) * 160.4 \text{ nm} \quad (6)$$

The values of the length from DLS, UV-Vis-NIR, and AFM for the fractions generated by centrifugation at 1257 Rad/s, shown in FIG. 7 are shown in FIG. 8. AFM images of selected