Better Signal-to-Noise Ratios for Carbon Nanotube Spectra

EEMs with the NanoLog®

Corrected emission spectra[1] of carbon nanoparticles can provide excitation–emission matrices (EEMs) for a range of excitation wavelengths. Acquisitions take only minutes (λ_{exc} = 500–800 nm at 1-nm intervals, λ_{em} = 830–1350 nm at ~1 nm per pixel). EEMs are compiled by HORIBA Jobin Yvon's Nanosizer® software. A double-convolution algorithm (U.S. pat. pending) in the software simultaneously computes excitation and emission wavelength coordinate lineshapes for each species within minutes; contributions from all spectral bands in a region of interest are found.

EEM data (Fig. 1, solid lines) and simulations (contour maps) of two SWNT suspensions from two different manufacturing processes are distinguished by their different size and helical distributions: high-pressure carbon-monoxide method (HiPCO, Fig. 1a), and cobalt–molybdenum catalytic method (CoMoCAT, Fig. 1b).[2] HiPCO forms a broad size distribution of SWNTs (0.6–1.3 nm diam) with many helical angles and >50 species. CoMoCAT has a narrower average size (~0.8 nm diam) and smaller helical-angle distribution; just two species—(6,5) and (7,5)—comprise ~58% of the intensity. Fig. 1a identifies five main HiPCO species, while Fig. 1b identifies four main CoMoCAT species in the specified regions of interest. Fig. 1c shows a comparative helical map of species found in Figs. 1a and b, and plots the helical angle versus SWNT diameter (in nanometers) against intensity of emission (symbol size/color). HiPCO tubes have a larger average diameter than CoMoCAT.

The NanoLog®'s signal-to-noise ratio

High signal-to-noise ratio (*S/N*) is crucial to unambiguous, rapid quantitative determination of the multiple species present in SWNT samples. While the NanoLog®'s *S/N* varies with its configuration, to show the high *S/N* of the NanoLog®, we studied a sample of SWNTs prepared by the HiPCO method. The nanotubes were dispersed in SDBS. The NanoLog® consisted of a Fluorolog®-3 with a double-grating excitation monochromator and the iHR320 emission spectrograph coupled to a 512-pixel InGaAs array. Excitation = 725 nm, bandpasses = 25 nm, integration time = 1 s. Fig. 2 shows an emission spectrum (a) from 835–1359 nm with known peaks, and a second excitation spectrum (b) from 600–800 nm to record noise at 850 nm. Dark noise was subtracted from both scans. Both dark-corrected spectra were then corrected by dividing by a reference signal to remove source inhomogeneities.

We define the *S/N* in Eq. 1:

$$\frac{S}{N} = \frac{S_{peak} - S_{background}}{\sqrt{S_{background}}} \qquad \text{(Eq. 1)}$$

The peak signal, S_{peak}, is measured at the highest peak in Fig. 2(a), 1171 nm—the (8,6) species—and the noise, $S_{background}$, in a region with no signal [Fig. 2(b), 600–800 nm]. An "ideal" system

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[1] Detector dark-signal and spectral response, and lamp output.

[2] D.E. Resasco, *et al.*, *J. Nanoparticle Res.* **4**, *2002*; 131–136; S.M. Bachilo, *et al.*, *J. Am. Chem. Soc.*, **125**, *2003*; 11186–11187.
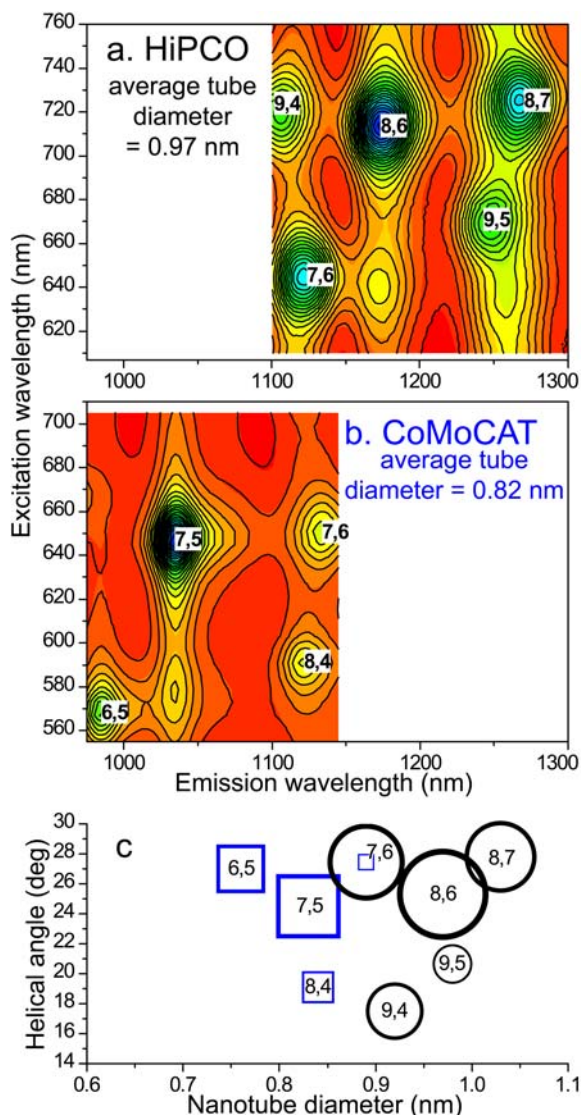
Fig. 1. EEMs (a, b) and helical maps (c) of HiPCO and CoMoCAT SWNT suspensions. Solid lines (a, b) are data; colors are simulations. Symbol sizes (c) show relative amplitudes for HiPCO (○) and CoMoCAT (□), normalized to 1. Simulation R^2 values = 0.997 (HiPCO) and 0.999 (CoMoCAT).

has $S_{background}$ = 0 at 600–800 nm. Thus Eq. 1 becomes

$$\frac{S}{N} = \frac{S_{1171nm} - S_{600-800nm}}{\sqrt{S_{600-800nm}}} \qquad \text{(Eq. 2)}$$

For Fig. 2(b), the average noise was 5.443, and the maximum signal was 7357.41 at 1171 nm, giving a S/N = 3151.
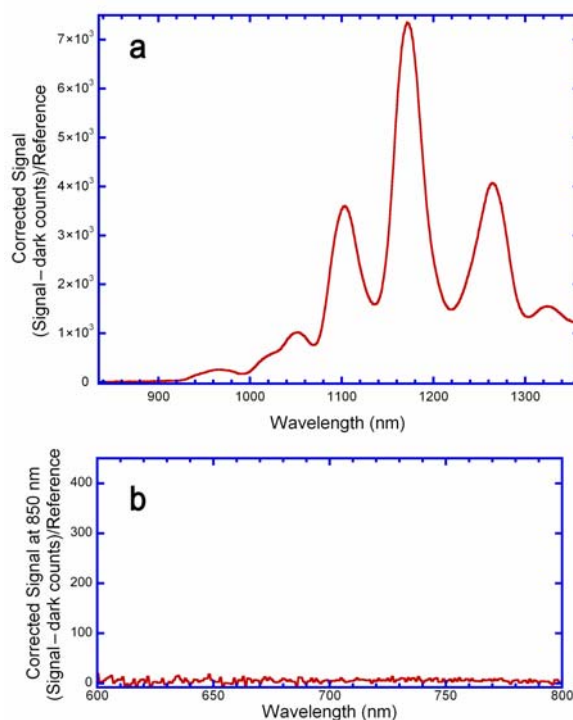
Fig. 2. Emission [Plot (a), λ_{exc} = 725 nm] and excitation spectra [Plot (b), λ_{em} = 850 nm] of HiPCO nanotubes. Spectra are corrected for excitation inhomogeneities and dark counts. Plot (b) was used to calculate noise.