size distribution of single walled carbon nanotubes (SWNT) obtained by asymmetric flow field fractionation (AFFF)

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The most accurate geometrical characterization of tubular nanoparticles in suspension is achieved by examining them one-at-a-time within a well defined microscopic field (transmission electron microscopy, atomic force microscopy, etc..). An alternative far easier approach involves separating particles by their size and then measuring them in real time on-line by a Multiangular Light Scattering (MALS) detector.

Asymmetric Flow Field Fractionation (AFFF) separates particles according to their sizes in a manner similar to the separation of molecules within a chromatography column. AFFF has been employed to provide separation and quantification of the size distribution of different samples of single walled carbon nanotubes (SWNT-Nanocyl) that have been previously purified and cut into short lengths by a chemical oxidation process [1].

The size distribution characterization was carried out on an Eclipse AF4 system equipped with the standard channel (25 cm), 350 μm spacer and regenerated cellulose membrane (5 kDa cutoff) (fig.1). For the determination of optimal AF4 separation conditions, different injection volumes and channel flow to crossflow ratios were tested. Immediately after the fractionation, the dimensions of the separated nanotubes were measured sequentially in a MALS (DAWN) coupled with a UV-detector and a refractive index detector. DAWN detector yields an elution curve that directly represents the size distribution of the sample (fig.2).

Figure 3 shows the cumulative weight fraction versus the root mean square (rms) radius of the purified and cut SWNT samples, respectively. The rms radius of purified SWNT ranges from **120 to 584nm** and they are distributed in three ranges. As it was expected, the size range for cut SWNT is shorter and the larger concentration (94%) ranges from **53 to 134nm**. The average size of purified and cut SWNT samples was also calculated at the same run time in both cases (8 min. and 12min). The results confirm again the shorter size of cut SWNT respect to the purified ones.

Run time (min)	Purified SWNT (nm)	Cut SWNT (nm)
8	233.3 ± 18.2 (0.5 ± 0.1)	105.1 ± 5.1 (0.3 ± 0.1)
12	141.8 ± 13.1 (0.1 ± 0)	86.4 ± 15.3 (0 ± 0)

These results show an alternative method to measure nanoparticles with tubular morphology. The AFFF/DAWN combination is a fast separation method suitable for carbon nanotubes size distribution characterization.

References

[1] Liu et al. Science, **280** (1998) 1253-1256.

Figures

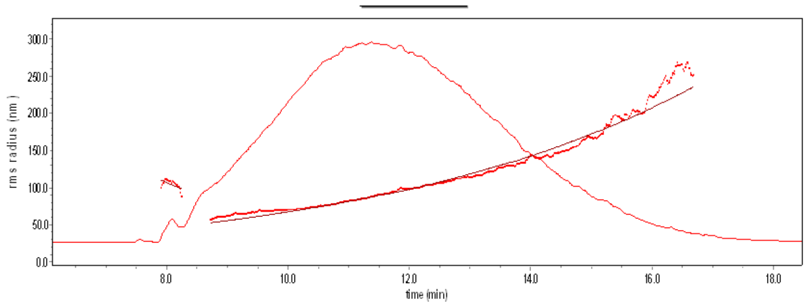

Figure 2 Particle size distribution of cut SWNT-Nanocyl samples by AFFF/DAWN analysis

Figure 1. Set-up for a complete FFF-MALS system (Eclipse, DAWN, Optilab, Agilent HPLC)

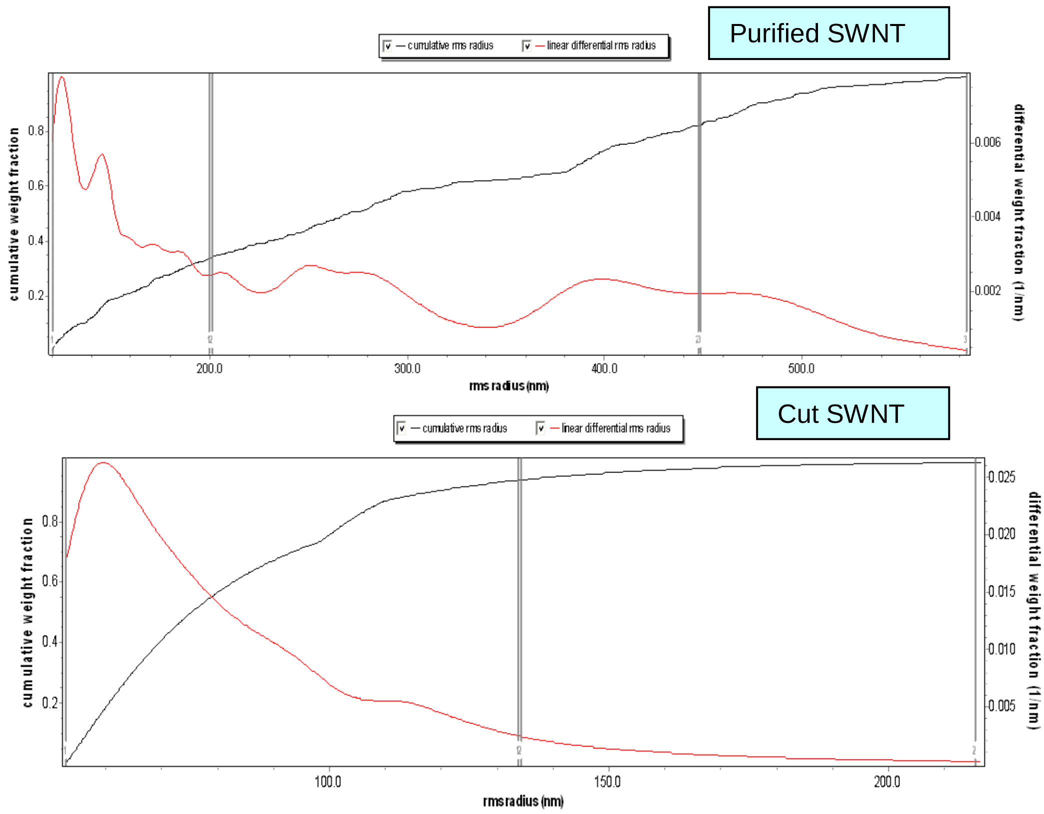

Figure 3 Cumulative weight fraction versus rms radius of the purified and cut SWNT samples

SWNT	Name	Type	Start - End	Limits (%)	Cumulative %
Purified	Range 1	rms radius	120.3 - 199.4 nm	0.6 - 33.5	32.9
	Range 2	rms radius	201.1 - 447.6 nm	34.1 - 82.0	47.9
	Range 3	rms radius	448.8 - 583.6 nm	82.2 - 99.7	17.5
Cut	Range 1	rms radius	52.9 - 133.8 nm	0.0 - 93.8	93.8
	Range 2	rms radius	134.4 - 215.5 nm	93.9 - 99.7	5.9