Spectral and Electron Microscopic Characterization of Functionalized Carbon Nanotubes

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Raw single-walled carbon nanotubes (SWNTs) produced by arc discharge were first treated with $K_2S_2O_8$ in dilute H_2SO_4 solution to generate oxygenated functional groups such as carboxyl, hydroxyl, and carbonyl on varying carbon components. Further functionalization with octadecylamine was carried out via a condensation reaction between an amine group and a carboxyl group with the assistance of dicyclohexylcarbodiimide. The attachment of long alkyl chains leads the sample to be soluble in tetrahydrofuran and other organic solvents.

The thus obtained stable dispersion was subjected to dispersion–centrifugation recycles, and then the metal catalysts and varying carbon impurities were separated with SWNTs. Figure 1 shows the absorption spectrum of the purified carbon nanotubes in the visible and near infrared ranges. Apart from the three characteristic absorption bands at approximately 1800, 1000 and 700 nm, respectively, superimposed on a broad background,[1] fine structure is also resolved in the visible range. The features at 644, 675, 699, 735, and 784 nm are ascribed to the electronic transitions between the first pair of the van Hove singularities (VHSs) in metallic SWNTs, and those at 466, 553, and 584 nm to the third pairs of VHSs in semiconducting SWNTs.[2]

The morphology of the functionalized SWNTs was observed and the degree of their purification was qualitatively estimated with a scanning electron microscope (SEM). From Figure 2 high-density SWNTs are observed without catalytic metal particles, turbostratic graphite or polyhedral carbon nanoparticles. It should be noted that the SWNTs are uniformly aggregated and buried by a substances, which should be originated from the attached octadecylamine alone with a little amorphous carbon residue. Though we could not analyze the length distribution of the SWNTs, bundles with diameters as small as 5 nm are observable in the brim of the sample. Considering the promoting effects of solvent removal on the aggregation of soluble SWNT, we believe in that the functionalized SWNTs could be dispersed in THF individually or as thin bundles.

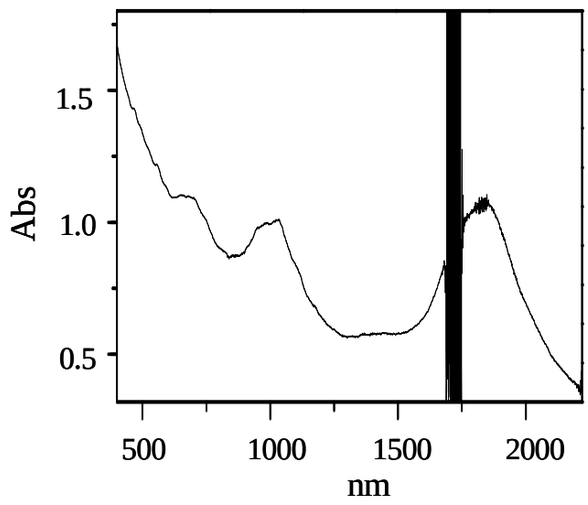

Figure 1. Absorption spectrum of the functionalized SWNTs dissolved in THF.

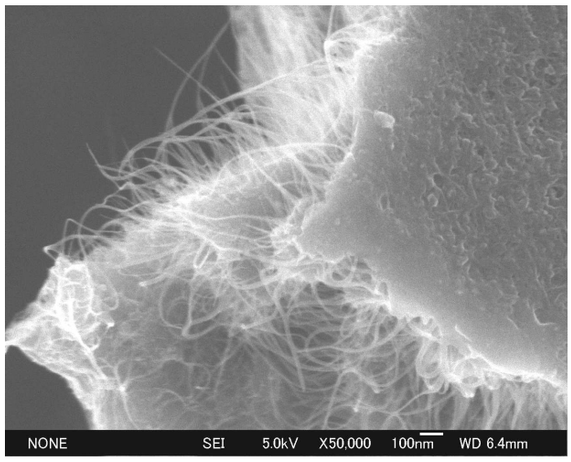

Figure 2. SEM image of the functionalized SWNTs.

References

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2. Lian, Y. F.; Maeda, Y.; Wakahara, T.; Akasaka, T.; Kazaoui, S.; Minami, N.; Choi, N.; Tokumoto, H. *J. Phys. Chem. B* **2003**, *107*, 12082.