

Below Figures 4 and 5 are presented again including the error bars for a 95% confidence interval.

Figure 4: The samples were heated to different temperatures which were then maintained. It never took longer than three minutes to achieve thermal equilibrium at the set point temperature. The absorbance intensities have been rescaled and offset correct to represent the degree of water filling as a percentage of the initial water content. The integrated scatter intensities are offset corrected. In the legends F stands for the samples that were functionalized by heat treatment, and AP stands for the as-purchased samples. The CNTs were heated to 45 °C (a), 60 °C (b), 85 °C (c), and 95 °C (d) after having been oriented vertically so any water in the samples soaked the CNTs. The abscissa has been normalized to the time at which evaporation went to completion in the as-purchased sample. The time to which the abscissa was normalized is located next to the temperature at which the experiment was conducted. All signals depicted are the differences between the wet samples and their corresponding dry samples. The relative error for a 95% confidence interval was never greater than 4% of the signal amplitude for all curves displayed.



Figure 5: A one hour heating ramp to 320 °C and subsequent cooling period were applied to the samples. The absorbance and scatter intensities have been offset corrected. F and AP stand for functionalized and as-purchased respectively. Both the absorbance and integrated scatter signals are the differences between the wet samples and their corresponding dry samples. The relative error for a 95% confidence interval was never greater than 14% of the signal amplitude for all curves displayed.



heat treatment 1 Print Mag: 1480000x @ 8. in 11:05 04/10/12 HV=200kV Direct Mag:150000x X:Y: T: CAMR Electron Microscope Facility heat treatment 1 Print Mag: 2960000x @ 8. 11:16 04/10/12 HV=200kV Direct Mag: 300000x X: Y: T: CAMR Electron Microscope Facility

The four images presented above are TEM images taken of the as-purchased (a and b) and functionalized (c and d) CNT samples. Open and close ended CNTs can be seen in both samples. In figure a, a closed ended CNT can be seen in the aspurchased sample, and in figure b an open ended CNT is shown from the as-purchased sample. In figure c the white arrow points to an open ended CNT whose circular cross section is oriented parallel to the imaging plane. In figure d the white arrow points to an open ended CNT. It is difficult to see that this CNT is open because there is another CNT located directly beneath it with respect to the electron beam. The black arrow in figure d points to a close ended CNT. Without taking a large number of images so as to make a statistically significant number of CNT end observations in both the as-purchased and functionalized samples no statement can be made about the opening efficiency of the heat treatment. While a measure of the opening efficiency could not be obtained the closure of CNT ends with hemispherical caps and their subsequent removal by oxidative methods in order to make the inner bore accessible for capillary filling is well documented in the literature. References 12 and 14 delineate how fullerene chemistry is a consequence of the decrease in energy associated with the relief of strain inherent in this class of molecules. References 9 and 13 make note of the removal of hemispherical caps at CNT ends by heating in air in order to permit capillary filling. Reference 9 is the seminal work of Dujardin on the nanocapillarity of CNTs conducted over twenty years ago. In reference 9 Dujardin discovered an empirical surface tension limit below which liquids will capillary fill the inner bore of CNTs. Reference 11 discusses the addition of oxygen containing functional groups to the CNT surfaces upon oxidation. Given the longevity of the practice where oxidative methods are utilized to open the ends of CNTs and its commonplace occurrence in the literature it is the author's opinion that the burden of proving the effects of oxidative heating on CNTs is not theirs.