Preconcentration of Organic Signatures Based on Carbon Nanotube Composites

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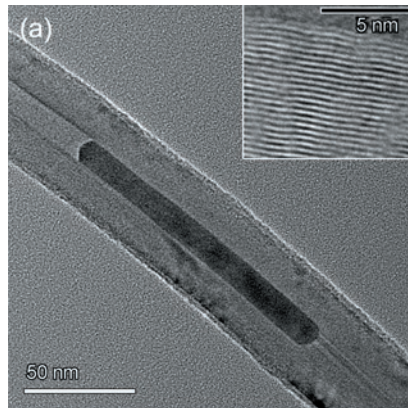
◆ This work is focused on the fundamental understanding of the interactions of carbon nanotubes with vapor species and the development of novel preconcentration platforms for trace chemical detection based on that understanding. ◆

Carbon nanotube composites represent a potentially significant advancement in the field of preconcentration and analytical separations due to their high surface area and high thermal conductivity. These two characteristics in particular suggest that carbon nanotube composites have a high probability for improving state of the art in preconcentrator-facilitated trace detection. The high surface area of carbon nanotubes allows for effective miniaturization of the preconcentrator, while high thermal conductivity allows for fast cycling of the preconcentrator. However, there is a lack of understanding of how nanotube structure affects sorption properties, which is necessary for the design of an optimized preconcentrator.

This project seeks to develop approaches for preconcentration of gas phase constituents using analyte-specific carbon nanotube composite materials. Two related research thrusts are required to reach this goal – chemical functionalization of nanotubes in composite structures and characterization of the sorption properties of the functionalized materials.

In FY 2003, we demonstrated derivatized pyrene stacking as a viable approach to functionalize carbon nanotubes through noncovalent bonding. The structures were verified by various analytical means, and it was shown that coverage of functional groups can be controlled by pyrene anchor concentration. This approach, together with other covalent approaches researched in this project (e.g., partial oxidization of carbon nanotubes), will serve as the foundation for FY 2004 efforts on the functionalization of carbon nanotubes to obtain selectivity for target analytes.

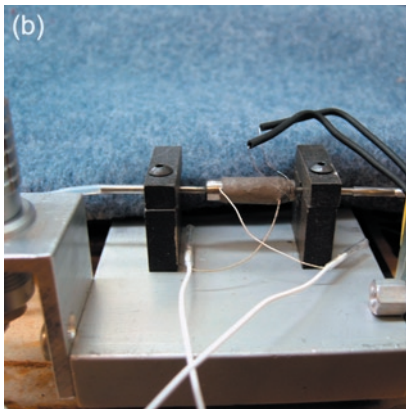
For controlled materials synthesis, a new chemical vapor deposition approach was developed based on a metallocene route. The new method allows us to increase the carbon nanotube mass fraction in preconcentrators to up to 35 wt%. Although the carbon nanotubes produced are of high quality, the size range is broad. Better control over size will be attempted in FY 2004 to quantify the effect of carbon nanotube size on adsorption characteristics.



(a) Tunneling electron microscopy micrograph of carbon nanotubes grown by metallocene route.

We made the first carbon nanotube-based preconcentrators and tested for capacity and kinetics using an existing trace gas testing system. Initial data pointed to the need for an all-metal preconcentrator construction to allow higher temperature use and avoid contamination problems associated with polymer shell materials. The redesigned metal preconcentrators eliminate interference from vapor exposure history, and we were able to demonstrate a one-part-per-billion detection limit when these preconcentrators were used with a proton transfer reaction mass spectrometer. With this added sensitivity, it is believed that significant improvements can be brought to atmospheric trace gas measurements.

In FY 2004, we will continue the testing of functionalized carbon nanotube composites in the gas detection test system, as well as evaluate these structures using a combination of analytical tools including acoustic sensors; electron microscopy; and Raman, Fourier transform infrared, and nuclear magnetic resonance spectroscopies.



(b) New all-metal preconcentrator.