Aerosolization of Carbon Nanotubes for the Development of Reference Air Samples

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ABSTRACT

Reports on the inhalation risks associated with carbon nanotubes (CNTs) express concern for exposures throughout the lifetime of CNTs and CNT-enabled products. While the aerosolization of nanomaterials has been studied previously by other groups, this study proposes and evaluates a more simplistic method for creating aerosolized CNTs with subsequent collection of airborne particulate in close proximity to the point of exposure. Our method investigates the use of an aerosolizing chamber in tandem with filtering cassettes historically used for airborne asbestos sampling and analysis. The results show that carbon nanotubes from a bulk sample are dispersed into the air and collected onto the filters. These filters can then be prepared for transmission electron microscopy and analyzed for CNTs using the test method of interest.

Keywords: nanotube, nanofiber, airborne, exposure, TEM, method

1 INTRODUCTION

Studies discussing the risk of carbon nanotube (CNT) inhalation[1,2,3] have generated concern among regulators, manufacturers and the general public. The ability to quantify airborne exposures to workers and consumers will be a key element in assessing safety. Airborne sampling is already being performed in some environments[4,5]; however, there is currently no standard test method for the sampling and analysis of airborne carbon nanotubes and proposed aerosolization techniques are complex.[6] Individuals interested in developing such a standard test method will need easily created, reliable samples with a distribution of particles capable of evaluating the method.

2 METHODS

The preparation of air samples for method development was achieved first by aerosolizing a known amount (by weight) of carbon nanotubes. The aerosolization was performed inside a small acrylic box with two ports; one for sample collection and one for the aerosolization source. Sample collection is facilitated by the use of a pump pulling air through a filter in a standard 25 millimeter cassette, historically used for asbestos testing. The cassette and attached tubing were fed into the small box through the sample collection port. The cassette housed a 0.22 micrometer pore size polycarbonate filter with a 5.0 micrometer mixed cellulose ester backing filter for support. Aerosolization of the CNT sample was performed using a 10 ounce (by volume) compressed air canister. The compressed air was released into the box, through the aerosilization port, for several seconds (aggresively aerosolizing the CNT sample material) and followed by a settling period of sixty seconds. After the settling period, the pump was turned on for a total of 10 minutes at a flow rate of 4.0 liters per minute. The entire assembly (small acrylic box, canned air, and pump) were placed into a HEPA filtered glovebox prior to and during the aerosolization and air sampling steps.

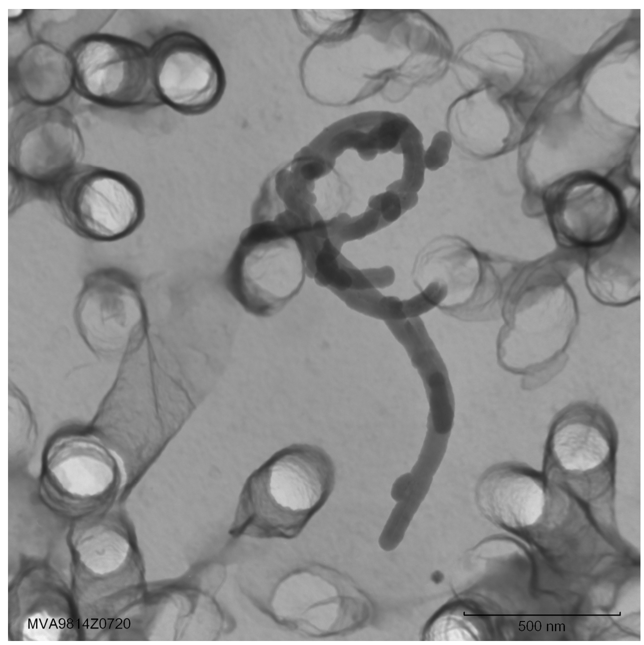

Figure 1. Airborne MW-CNT bundle collected on a polycarbonate membrane filter.

The CNT sample used in this study was a functionalized multi-walled carbon nanotube sample; however, a variety of sample types including carbon nanofibers and single-walled carbon nanotubes could potentially be used. The collected airbore sample and filter were carbon coated, placed on indexed copper grids in a Jaffe wick (a stainless steel bridge covered with lens paper in a petri dish) and treated with a mixture of 20% 1-2-diaminoethane and 80% 1-methyl-2-pyrrolidone. The mixture was added to the petri dish with the solvent meniscus coming in contact with the bridge from below and allowed to set for 25 min. The stainless steel bridge was removed from the dish with forceps, placed in another dry petri dish and rinsed with DI H_2O several times in preparation of analysis by transmission electron microscopy (TEM). The results shown here were obtained using a Philips CM 120 TEM operated at 100kV and equipped with an Oxford INCA energy dispersive spectrometer.

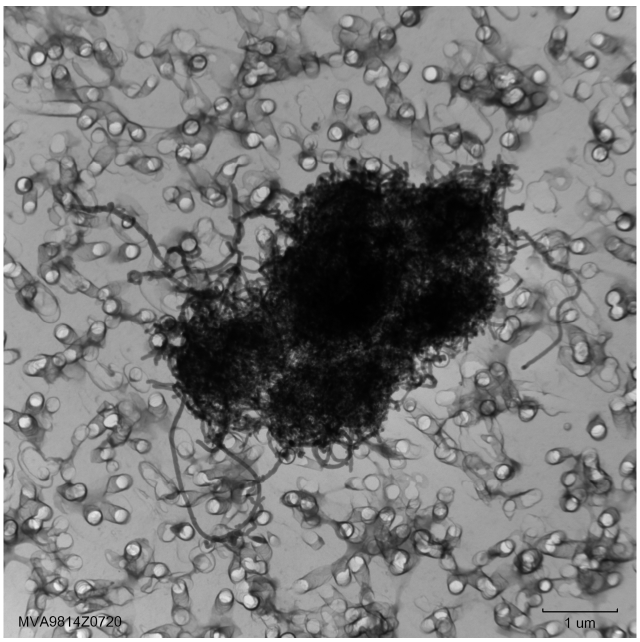

Figure 2. Airborne MW-CNT cluster collected on a polycarbonate membrane filter.

3 RESULTS AND DISCUSSION

Analysis of the prepared TEM grids show evidence of single CNT fibers, CNT bundles (Figure 1) and clusters or agglomerates of CNTs (Figure 2). Standard test methods for analyzing the airborne CNTs can be developed for exposure assessments to workers and consumers or for toxicology studies to characterize CNT release and investigate the mechanisms of disease potential. Analyses for worker exposure assessments should employ a method that facilitates a rapid assesment of air quality, focusing on either a simple fiber count or a presence/absence measurement (to some established analytical sensitivity). A more rigorous method is needed for detailed exposure or toxicology studies. This second type of analysis could provide a thorough characterization of the observed airborne CNT fibers, bundles, clusters and even matricies (CNTs overlapping or protruding from non-CNT matrix elements) by either enumeration of each observed structure or a sorting of observed CNT structures into appropriatly designated size bins and suggested by Kuempel et al.[7]

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