# Deposition of carbon nanotubes in commonly used sample filter media

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**ABSTRACT:** There is no single standard technique or methodology to characterize the size, structure, number, and chemical composition of airborne carbon nanotubes. Existing analytical instruments and analytical techniques for evaluating nanoparticle concentrations cannot simultaneously provide morphology, state of agglomeration, surface area, mass, size distribution and chemical composition data critical to making occupational health assessments. This research utilized scanning electron microscopy and thermogravimetric analysis to assess the morphology and mass of carbon nanotubes collected using various commercial sample filters. It illustrated carbon nanotube agglomeration, deposition and distribution in commonly used sample filter media. It also illustrated that a sufficient mass for carbon nanotube analysis by thermogravimetric analysis is uncommon under most current research and production uses of carbon nanotubes. Individual carbon nanotubes were found to readily agglomerate with diameters ranging from 1 – 63 µm. They were collected at the face of or within the filter. They were not evenly distributed across the face of the filters.

**Keywords:** *Agglomerates, Carbon nanotubes (CNTs), Filter deposition, Particulate sample filters, Scanning electron microscopy (SEM), Thermogravimetric analysis (TGA)*

## INTRODUCTION

A paramount requirement of effective analytical techniques and methods is that they are capable of detecting desired concentrations (Seaton *et al.,* 2009). In the case of carbon nanotubes (CNTs), the technique must be capable of identifying concentrations at or below the $1\mu g/m^3$ recommended occupational (airborne concentration) exposure limit (NIOSH, 2013) required to prevent the array of adverse health effects that are reasonably attributable to exposure. The technique must also differentiate nanoparticles of interest from naturally occurring or anthropogenic nanoparticles that may originate from dust storms, fires, vehicle exhausts, indoor pollution, cigarette smoke, and consumer products (Lam *et al.,* 2006; Buzea, *et al.,* 2007; Sass *et al.,* 2008).

A nanotube is a nanometer-scale tube like structure with different material composition (Bernholc *et al.*, 1997) that includes carbon nanotubes, inorganic nanotubes that are often composed of metal oxides (Ahmadi *et al.,* 2011; Beheshtian *et al.,* 2012; Lam *et al.,* 2004) and DNA nanotubes. The application of nanotubes is in the midst of an explosive growth which is due primarily to their unique chemical and physical properties. These properties are said to promise new inventions, new products and new contributions to human knowledge (Remskar, 2004).

Carbon nanotubes (CNTs) are considered a new form of pure carbon that can be visualized as rolled hexagonal carbon networks that are capped by pentagonal carbon rings (Terrones, 2003). The simplest nanotubes are a single layer of carbon atoms arranged in a cylinder (single-walledcarbon nanotubes). They may also consist of multiple concentric tubes (multi-walled carbonnanotubes) with diameters up to 20 nm and

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lengths greater than 1 mm (Fairbrother, 2008). They have been produced with length-to-diameter rations of up to 132,000,000 to 1. CNTs are very strong (about 100 times stronger than steel), very light (one-sixth the weight of steel), and 10 times more conductive than copper (Ebbesen *et al.,* 1996; Elcock, 2007). CNTs have been reported as having a negative charge (Cheng, 2006) and can be either metallic or a semiconductor (Che *et al.,* 2000). They also tend to self-agglomerate (Grzelczak *et al.,* 2010) and easily fragment (Froeshke *et al.,* 2002). Each of these properties must be taken into consideration during analytical method and technique development processes.

NIOSH's Current Intelligence Bulletin, Occupational Exposures to Carbon Nanotubes and Nanofibers (NIOSH, 2013) recommends occupational exposures to all types of CNT may be quantified by NIOSH Method 5040, Elemental Carbon (Diesel Particulate). It also recommends using a mass-based airborne concentration measurement for monitoring workplace exposures to all types of CNT, and using additional analytical techniques to help to better characterize exposures to CNTs such as transmission electron microscopy equipped with x-ray energy dispersive spectroscopy. The NIOSH Analytical Method 5040 was developed in 1996 for use in the evaluation of occupational exposures to diesel particulates and may be prone to errors while attempting to measure airborne carbon nanotubes.

NIOSH Method 5404,served as a reference point for this research and the development of a method for the collection, measurements and analysis of CNTs because diesel particulates are typically less than 1 micron in diameter. Diesel particles are also generally expected to be equally distributed across the face of the sample (NIOSH, 1996).

Of importance in filter sampling is filter particle loading. This depends on the structure and composition of the fiber (packing density, fiber diameter, thickness), operating conditions (filtration velocity, temperature), and the density, particle size, and distribution characteristics of the filtered aerosol (Contal *et al.,* 2005).The most important mechanisms causing particle deposition in fibrous filters are diffusion, interception, and inertial impaction (Lee and Byh, 1982). Electrostatic forces and gravitational settling may also contribute to particle collection in a fibrous medium. However, particle collection is not uniformly distributed over the surface and thickness of a filter (Thomas *et al.,* 2001). The effect of particle loading is an initial increase of the filter performance. In the beginning, particles deposit in the depth of filters (Thomas *et al.,* 2001) with surface layers eventually becoming more loaded than depth layers. However, after some time, a decrease in particle collection efficiency will result with a point where particle collection will no longer be efficient. Recognizing when this transition is beginning to take place is an important part of particle collection activities.

The filters used in this research were selected because of their properties or because of their previous applications in quantifying occupational exposures to airborne contaminants: glass fiber (heat resistance, high collection efficiency), mixed cellulose esters (asbestos, metals), polycarbonate (cellulose insulation) and polyvinyl chloride (particulate matter). While each filter selected offers specific advantages with their use, their use can include disadvantages that may include potential overloading, static electricity, sampling interferences and physical damage problems that must be accounted for during use.

## MATERIALS AND METHODS
### Materials

Multi-walled carbon nanotubes were selected for this research. These CNTs were produced by chemical vapor deposition and purchased from Nanolab, Inc. of Waltham, MA. They were reported to be 15 +/- 5 nm in diameter, 1 – 5 μm in length, and > 95% purity. The sample filters selected were Millipore glass fiber (Lot #RODA25758), SKC mixed cellulose ester (Lot #8459-7D9pask-190), SKC polycarbonate (Lot # 10271-7dapask-278) and SKC polyvinyl chloride (Lot # T91021) and a SKC incorporated 37 mm, 3-piece air sampling cassettes were also used.

An Aldrich 250 ml two-neck round bottom flask (part # Z516872) was selected to serve as the test chamber. An air moving pump (Zefon product # Zefon Z-lite) was used to draw air through the test chamber. A Bjornax smoke pen was used to assess mixing within the chamber. CNT and filter analyses was accomplished with a JOEL JSM-6510 scanning electron microscope (SEM), X-ray energy-dispersive X-ray spectroscopy (EDS) and a Shimadzu TGA-50 thermogravimetric analyzer (TGA). Tygon tubing was used to connect the sampling cassette to the pump.

### Methods

To evaluate CNT mixing within the chamber, a smoke pen (part # S221, manufactured by Bjornax) was used to generate smoke particulates to be drawn into the

test chamber and through the testchamber assembly. This was accomplished without a filter in the assembly. Prior to assessing CNT deposition in sample filters at low and high volume air collection rates, and at short and long collection times, baseline data on the carbon nanotubes and sample filters had to be establish.

To establish the carbon nanotube baseline data, and verify the manufacturer's data, a sample of the CNTs were spread with a spatula across the face of mounting tape for visualization under the SEM. Additionally, 5 mg of CNT were also analyzed using TGA. To establish the baseline data for each filter, the filters were cut into squares with a sample of each visualized under SEM and approximately 5 mg of the cuttings analyzed using TGA.

The test chamber assembly, consisting of the test chamber, air sampling cassette, and air moving pump (Zefon product # Zefon Z-lite) were assembled in the order as indicated in Fig. 1, rinsed twice with acetone, and allowed to air dry.
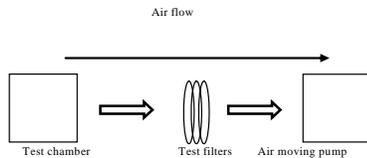


Fig. 1: Test chamber assembly set up

After allowing the test chamber to dry, the top cover and inner ring of the 37 mm filter cassette were removed from the cassette body (these were not used during the experiment) and the experimental filter was then inserted into the lower ring. The lower ring was then attached to the test chamber using duct tape. The assembly was configured so that there were no obstructions between the air drawn into the test chamber and the experimental filters. CNTs were then introduced into the chamber as indicated in Table 1. With the pump on, room air entered chamber where it would mix with the test CNTs introduced into the chamber, was drawn through the filter into the tubing, and exhaust through the pump.

At the end of each run, the filter was removed from the assembly. It was then cut into 6 mm squares for analysis by SEM and EDS as indicated in Table 2.

The entire surface of the mounted squares was viewed in a left to right, top to bottom pattern. The visual and SEM observations of each filter square and an estimate of the number of agglomerates (Froeschke *et al.*, 2003) and the sizes of each were recorded. Photographs were also taken for illustrative purposes. EDS scans were then taken of each sample filter.

## RESULTS AND DISCUSSION

The smoke pen test resulted with what appeared to be complete mixing in the text chamber. Upon assuring the test chamber would enable the complete mixing of the CNTs introduced within it, the focus then shifted to the CNTs themselves. Figs. 2, 3 and 4 depict SEM images of CNTs spread across the face of mounting tape, the accumulation of several agglomerations within a sample filter, and an indication of the large numbers of CNTs within a single agglomerate.

The TGA traces of the CNTs and sample filters are presented in Fig. 5.

The Y axis presents the sample mass (in mg) in the crucibleand the X axis presents the temperature in $^0C$ ranges of the TGA during the sample run. The colored lines (traces) indicate the mass of the sample in crucible at the start and end of the sample run for the CNTs and test filters.

The SEM images of the CNT laden glass fiber (GF) filter are presented in Figs. 6 and 7.

Fig. 6 is the GF filter upon removal from the test chamber. It is readily observable that equal particulate deposition has not taken place on the face of the filter. As particles tend to deposit only by touching and sticking to a capillary wall or other immobilized particles (Elmoe *et al.*, 2009), significant shading at the edges of the filter is visible. The SEM (13000X magnification)

Table 1: Experimental filters tests

| | CNTs introduced into system | 1st batch CNT Introduction | 2nd batch CNT Introduction | 3rd batch CNT Introduction |
|---|---|---|---|---|
| 2 lpm for 71 minutes | 200 mg | Start of test | 23 minutes into test | 46 minutes into test |
| 4 lpm for 71 minutes | 200 mg | Start of test | 23 minutes into test | 46 minutes into test |
| 2 lpm for 35 minutes | 200 mg | Start of test | 11 minutes into test | 23 minutes into test |
| 4 lpm for 35 minutes | 200 mg | Start of test | 11 minutes into test | 23 minutes into test |

Table 2: Filters analyzed by scanning electron microscopy andenergy-dispersive X-rayspectroscopy

| Filter | Manufacturer and lot number | Diameter | SEM filter squares |
|---|---|---|---|
| Glass fiber - Blank | MilliporeLot # RODA25758 | 37 mm | 4 |
| Glass fiber- Single filter | MilliporeLot # RODA25758 | 37 mm | 32 |
| Mixed cellulose ester - Blank | SKCLot # 8459-7D9PASK-190 | 25 mm | 4 |
| Mixed cellulose ester - Single filter | SKCLot # 8459-7D9PASK-190 | 25 mm | 16 |
| Polycarbonate - Blank | SKCLot #10271-7DAPASK-278 | 25 mm | 4 |
| Polycarbonate - Single filter | SKCLot #10271-7DAPASK-278 | 25 mm | 16 |
| Polyvinyl chloride - Blank | SKCLot # T91021 | 37 mm | 4 |
| Polyvinyl chloride - Single filter | SKCLot # T91021 | 37 mm | 32 |

analysis of the CNT agglomerates within the filter is presented in Fig. 7. The blue box in Fig. 6 indicates where the sample square was taken from the filter. The CNT agglomerates ranged from 1– 63 µm in diameter. The GF filter was the preferred of all the experimental filters to work with because its strength made it easy to handle and cut and it did not stick to other objects.

The SEM images of the CNT laden mixed cellulose ester (MCE) filter are presented in Figs. 8 and 9.

Fig. 8 is the MCE filter upon removal from the test chamber. An uneven particulate deposition is observable across the face of the filter. Shading at the edges of the filter is visible. The blue box in Fig. 8 indicates where the sample square was taken from the filter. The SEM (2700X magnification) analysis is of the CNT agglomerates (Fig. 9) within the bed of the filter. The CNT agglomerates ranged from 1– 63 µm in diameter. The MCE filter was moderately difficult to work with as it had a tendency to stick to most object used during its handling.

The SEM images of the CNT laden polycarbonate (PC) filter are presented in Figs. 10 and 11.

Fig. 11 is the PC filter upon removal from the test chamber. Almost no particulate deposition is visible across the face of the filter. Shading at the edges of the filter is visible. The blue box in Fig. 6 indicates where the sample square was taken from the filter. The SEM (4000X magnification) analysis of the CNT agglomerates on the filter face is presented in Fig. 11. The CNT agglomerates ranged from 1– 63 µm in diameter. The polycarbonate filter was enormously difficult to handle because it had a tendency to "roll-up" and stick to most objects used during its handling.

The SEM images of the CNT laden polyvinyl chloride (PVC) filter are presented in Figs. 12 and 13.

Fig. 12 is the PVC filter upon removal from the test chamber. An uneven particulate deposition is observable across the face of the filter; with more caking at the face than the other experimental filters. Light to moderate shading at the edges of the filter is visible. The blue box in Fig. 6 indicates where the
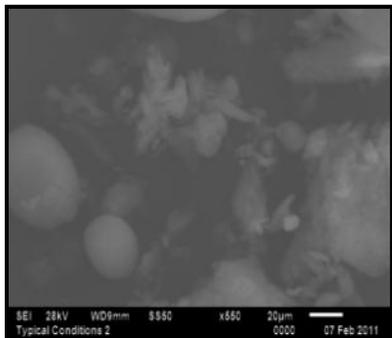


Fig. 2: CNT agglomerates on carbon adhesive tabs

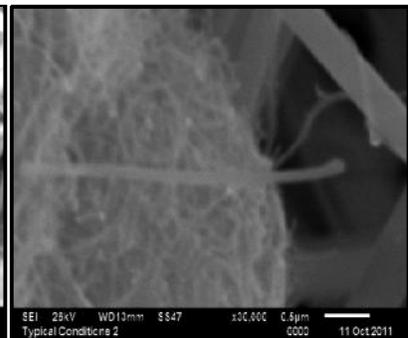Fig. 3: CNT agglomerates on the face of a glass fiber filter
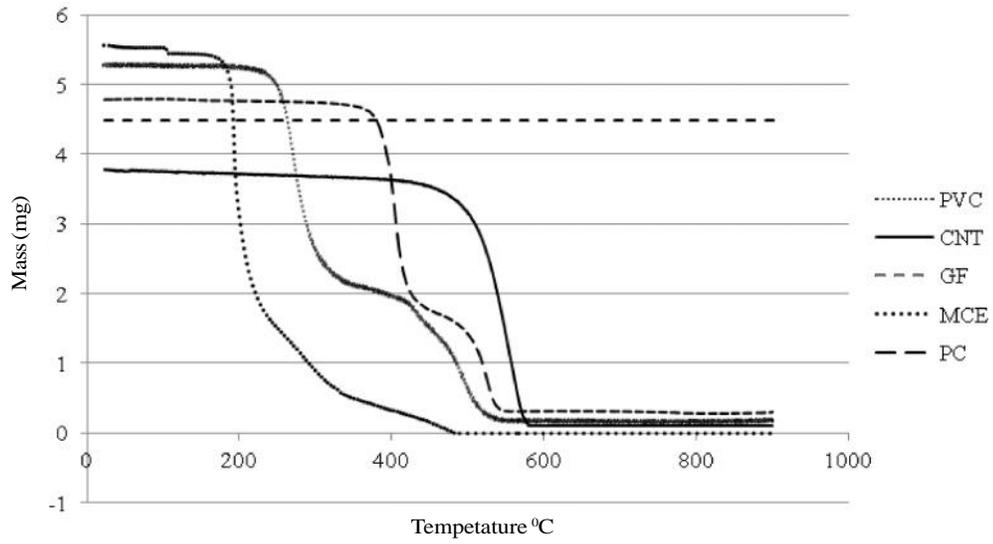
Fig. 4: Tightly agglomerated CNTs

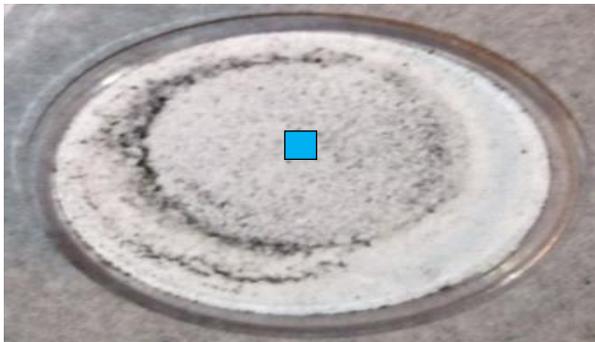Fig. 5: CNT sample and test filters TGA traces



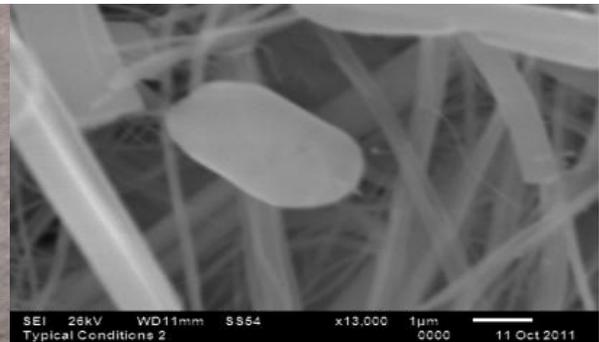Fig. 6: GF filter upon removal from test chamber



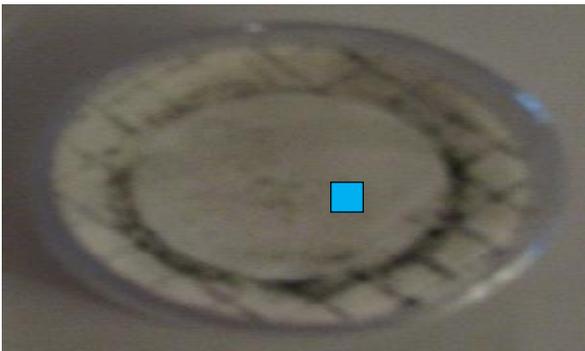Fig.7: SEM analysis of particles within depth of GF filter



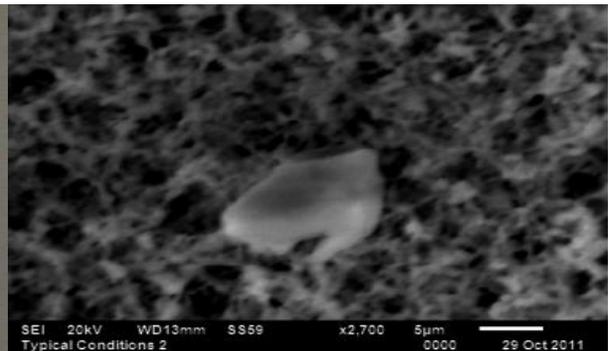Fig. 8: MCE filter upon removal from test chamber



Fig. 9: SEM analysis of particles within depth of MCE filter

sample square was taken from the filter. The SEM (1300X magnification) analysis of the CNT agglomerates within the filter is presented in Fig. 13. The CNT agglomerates ranged from 1– 63 µm in diameter. The filter tendency to stick to objects make it difficult to work with.

Figs. 14, 15, 16 and17 present the EDS scans of the CNT laden filters.

The filter spectra presented in Figs. 14, 15, 16 and 17 were collected from the highest, a medium and the lowest areas of deposits on the test filters. As the SEM and EDS operate in tandem, the spectra were obtained after the SEM analysis. EDS is capable of providing spectra data for any element with an atomic number above six (carbon). Identified elements were then classified according to their probable source and are listed accordingly. In the obtained spectra, there was no observation of elements that did not originate from the CNT synthesis, filter manufacturing, sample collection apparatus, or analytical support materials in any of the samples.

The smoke pen particles average about 1micron in diameter (Van Zant, 2000). However, the large quantity generated by the pen enabled the smoke stream to be seen with the unaided eye. Because of this, it was possible to visualize what appeared to be complete mixing of the particles in the test chamber.

The SEM, EDS and TGA of the stock CNT validated the manufacturer's listed properties in terms of the physical properties and the percent CNT purity. The observed impurities that were listed at 5% by the manufacturer were believed to be amorphous carbon and trace metals since CNT batches typically contain other carbonaceous and transition metal particles (Oswald *et al.*, 2005). The EDS scans of the CNT laden samples seemed to verify this.

The SEM visualization of CNT agglomeration in Figs. 2, 3 and 4 highlight the literature references that the normal tendency of CNTs is to agglomerate (Curtzwiler, 2008). It also answers the question as to why, unlike asbestos, determining a number of CNTs per a given air volume would prove nothing more than an impossible
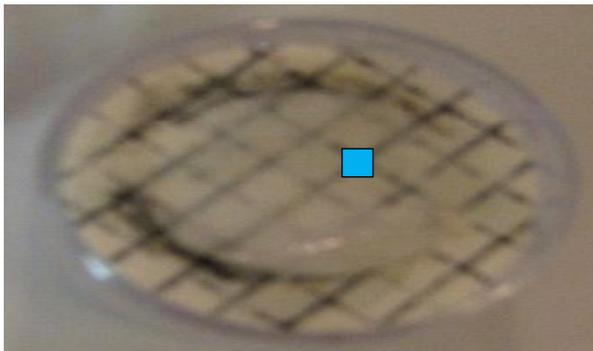


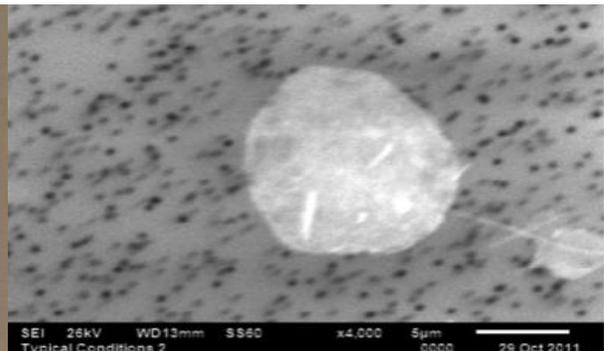Fig. 10: PC filter upon removal from test chamber



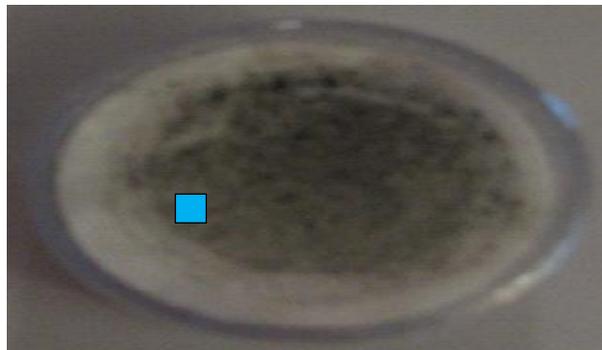Fig. 11: SEM analysis of particles on the surface of PC filter



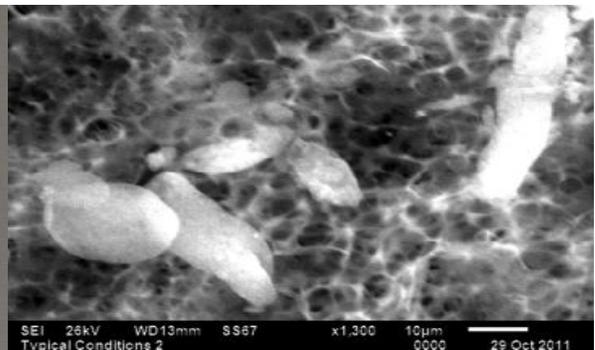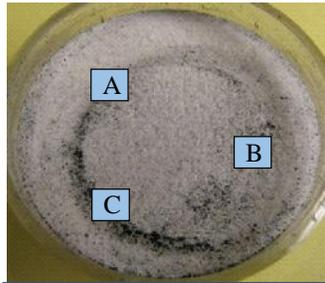Fig. 12: PVC filter upon removal from test chamber



Fig. 13: SEM analysis of particles within depth of PVC filter

Manufacturing catalysts: iron, molybdenum

Support materials: aluminum, manganese, silica

Filter: carbon, oxygen, sodium, silicon, barium, fluorine, potassium, zinc, magnesium

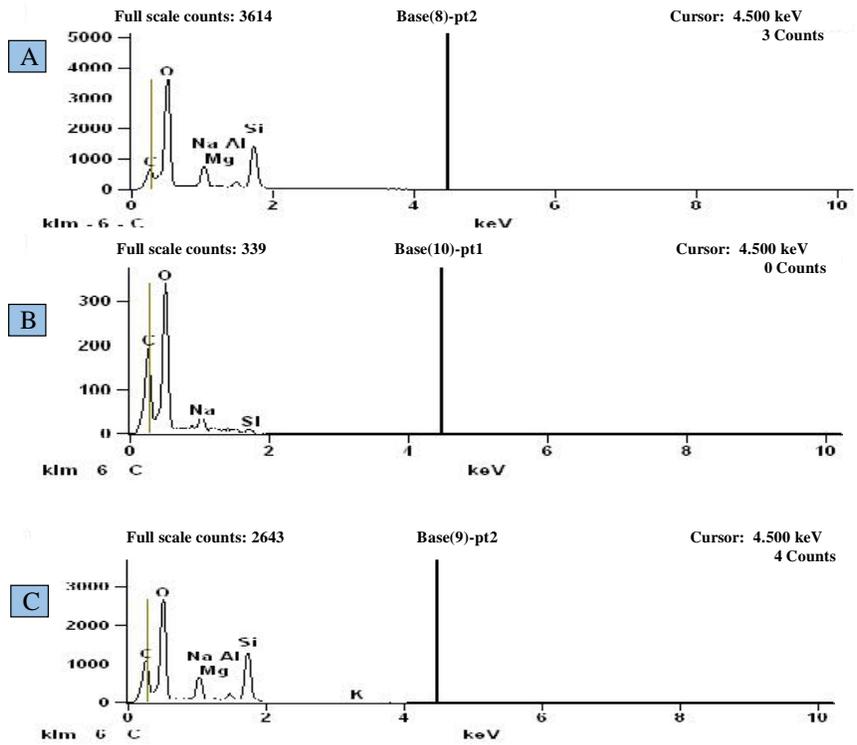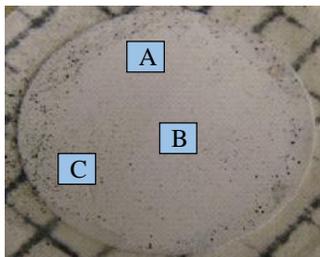SEM specimen mounts and mounting tape: aluminum, carbon

Fig. 14: Glass fiber filter spectra

Manufacturing catalysts: iron, molybdenum

Support materials: aluminum, manganese, silica

Filter: carbon, nitrogen, oxygen

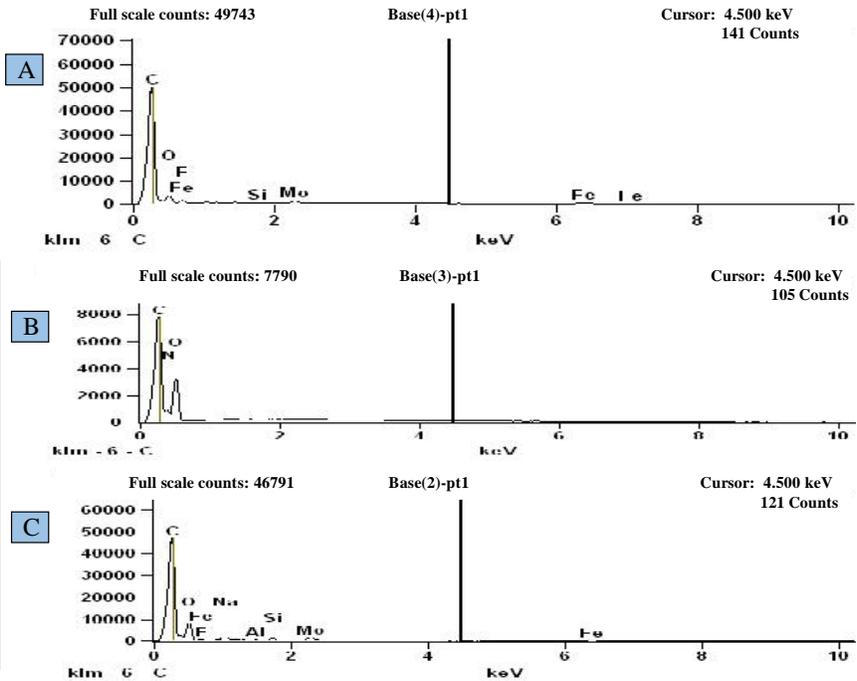SEM specimen mounts and mounting tape: aluminum, carbon

Fig. 15: Mixed cellulose ester filter spectra

Full scale counts: 2936        Base(1)-pt1        Cursor: 4.500 keV
0 Counts

Full scale counts: 3479        Base(7)-pt1        Cursor: 4.500 keV
0 Counts

Full scale counts: 2314        Base(8)-pt1        Cursor: 4.500 keV
0 Counts

Manufacturing catalysts: iron, molybdenum

Support materials: aluminum, manganese, silica

Filter: carbon

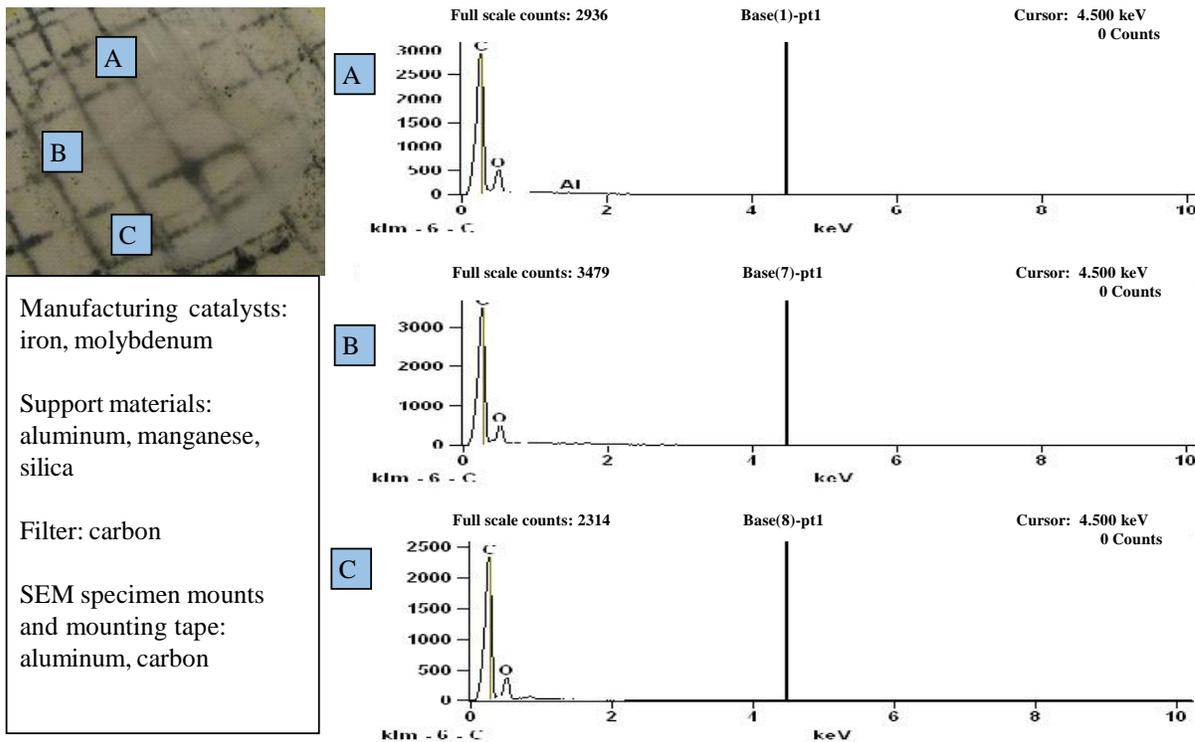SEM specimen mounts and mounting tape: aluminum, carbon

Fig. 16: Polycarbonate filter spectra

analytical data point to obtain because, as seen in Fig. 4, the CNTs are so tightly agglomerated that it is impossible to tell where one CNT stops and the other starts.

A challenge related to utilizing the TGA for the analysis of carbon nanotube exposed air sample filters is the 10 mg mass limit for samples placed into the TGA sample crucible. Because of the mass of the filters (GFF = 53.7 - 54.5 mg, MCE = 20 - 21.7 mg, PC = 3.4- 4.8 mg, and PVC = 15.6 - 16.1 mg), all (with the exception of the polycarbonate filter) must be cut prior to placement into the TGA sample crucible. These cuts could require 8 – 20 samples per filter and may cause significant carbon nanotube sample loss resulting from the handling and cutting of the filter.

With regard to the TGA shown data in Fig. 5, the traces indicates that CNT mass loss begins at approximately 420 $^0$C and concludes at approximately 580 $^0$C. The glass fiber filter, shows no mass loss through 1000 $^0$C, the upper limit of the TGA. The remaining three samples, the mixed cellulose ester filter, the polycarbonate filter, and the polyvinyl chloride filter, illustrate mass losses that either begin or conclude within the CNT loss ranges, thereby making it impossible to define what portion of sample in those temperature ranges were CNTs and what portion of the sample in those ranges were the particular filter. Because of this, those three filters were not suitable for CNT collection activities. The data is summarized in Table 3.

Increasing the flow rate or sample time had almost no bearing on the collection or deposition of CNTs using the glass fiber filters. Likewise, no difference was observed with the other three filters under the same parameters. Decreasing the flow rate or sample time decreased the volume of CNTs collected by the glass fiber filters and again made no difference whatsoever with the other three filters.

As indicated previously, EDS can be used to identify non-carbon sample constituents and may help differentiate CNTs from other possible elemental carbon containing particles such as diesel soot or carbon black or contaminants. Using the EDS point and shoot mechanism, the elements listed in Figs. 14, 15, 16 and 17 are those anticipated to be found in each of the CNT exposed filters.
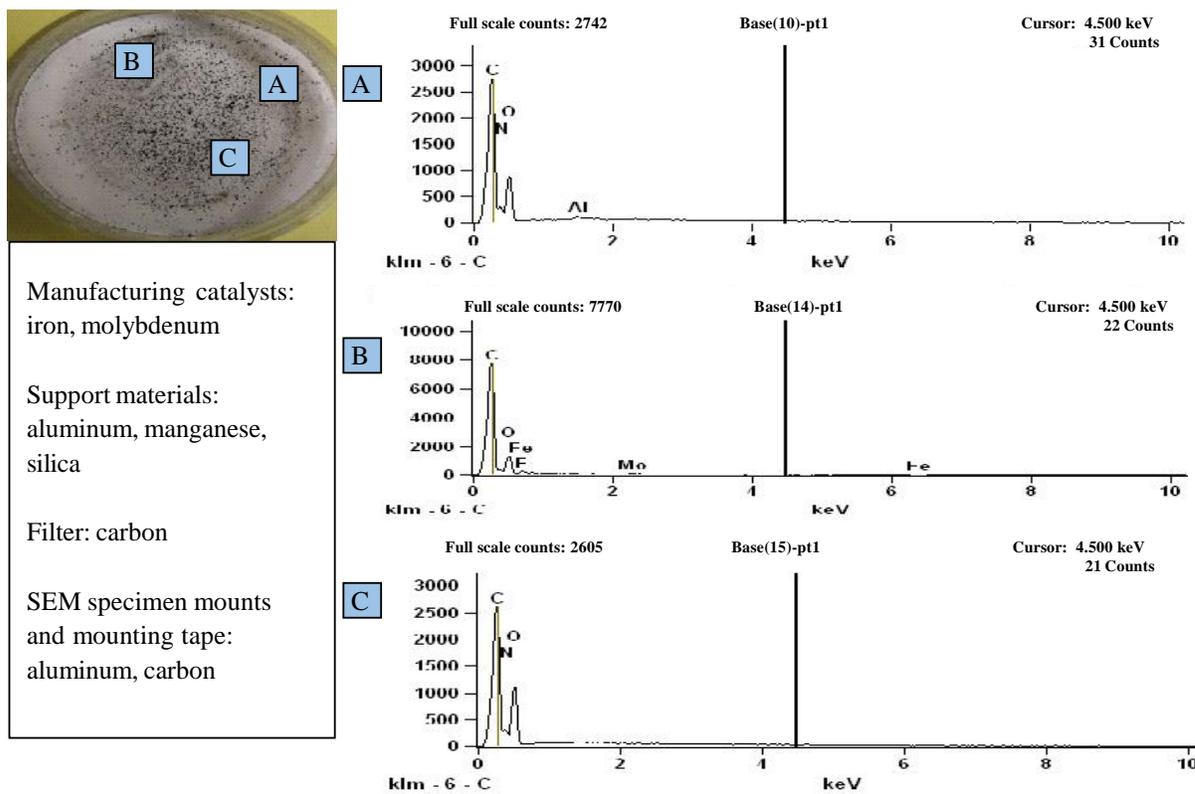
Manufacturing catalysts: iron, molybdenum

Support materials: aluminum, manganese, silica

Filter: carbon

SEM specimen mounts and mounting tape: aluminum, carbon

Fig. 17: Polyvinyl chloride filter spectra

## CONCLUSION

This research project illustrated that CNTs are not evenly distributed across the face of air sampling filters during sample collection events. It bears mentioning that, since CNT disposition across the sample filter is not uniform, the filter portion selected for analysis when following the NIOSH analytical method may significantly underestimate or overstate the true sample concentration. Consequently, analysis of the entire sample filter will generally be required to obtain an accurate CNT mass. It is for these reasons that the deposition of CNT on commonly used sample filter media is important data.

A second point that needs to be made regarding airborne carbon nanotubes is given their current use, it is unlikely that in most cases a sufficient mass of CNTs for thermogravimetric analysis. Therefore, it remains important that the analysis of airborne carbon nanotubes be accomplished by two methods in tandem, one that estimates the mass of the CNTs and one that enables the visualization of the number of particles, their shapes, sizes and states of agglomeration.

Table 3: Thermogravimetricanalysis of CNTs and sample filters

| Sample | Beginning of sample degradation ($^0$C) | End of sample degradation ($^0$C) | Maximum TGA temperature setting ($^0$C) |
|---|---|---|---|
| CNT | 420 | 580 | 1000 |
| GF | n/a | n/a | 1000 |
| MCE | 100 | 500 | 1000 |
| PC | 380 | 550 | 1000 |
| PVC | 200 | 550 | 1000 |

## CONFLICT OF INTEREST

The authors declare that there is no conflict ofinterests regarding the publication of this manuscript.

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