Aerosol and Air Quality Research, 14: 1352–1359, 2014

ISSN: 1680-8584 print / 2071-1409 online
doi: 10.4209/aaqr.2014.01.0025

Filtration and Length Determination of Airborne Carbon Nanotubes in the Submicrometer Range Using Nanofiber Filters

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ABSTRACT

The growing usage of carbon nanotubes (CNTs) in different applications increases concern of negative influence of CNTs on human health. Therefore the control of CNTs is significant and filtration is an effective method to control airborne CNTs. We investigated penetration of airborne CNTs through nanofiber filters composed of micro and nanometer fibers. The theoretical model for nanofiber filter media was also investigated and the experimental results agreed with the model. The penetrations of CNTs through the nanofiber filters were compared with the penetration through a screen type filter made of stainless steel. Moreover, the geometrical lengths of CNTs were calculated by the filtration method using the nanofiber filter. The calculated lengths showed a good agreement with the measured lengths by scanning electron microscopy analysis. The developed method for the length measurement of CNTs provides the possibility of online measurement for elongated particles such as CNTs.

Keywords: Carbon nanotubes; Filtration; Nanofiber filter; Length measurement.

INTRODUCTION

Recently many studies regarding toxicity of CNTs showed possible negative influence of airborne CNTs on human health under specific conditions. For instance, dependent on types and amount of metals on CNTs, also shape, length and agglomeration status, CNTs can induce dose-dependent lesions such as inflammation, granulomas and necrosis (Sato *et al.*, 2005; Lam *et al.*, 2006; Wick *et al.*, 2007; Poland *et al.*, 2008). For this reason, filtration of airborne CNTs is important in order to avoid the health risks using CNTs in various applications in mechanical, electrical and material fields.

Wang *et al.* (2011a) evaluated stainless screens, known as a diffusion battery, in filtration tests against airborne CNTs. They performed numerical calculations of penetration of CNTs through the filter screen to understand the capture mechanisms of elongated particles by the screen. The results showed the importance of geometrical length in the filtration study of elongated particles. The dependence of interception increases due to longer effective interception lengths of elongated particles than those of spherical particles. A numerical model for nanofiber filters was developed by Wang *et al.* (2008a, b) and compared with experimental results. The model showed good agreement with experiments for 20–780 nm particles. Filtration experiments have been performed using silver particles, NaCl particles and polystyrene latex (PSL) particles for 3–20 nm, 20–300 nm and 780 nm particles, respectively.

Nanofiber filter media possess higher efficiencies for submicron particles due to their larger specific surface area to collect and lower resistance for air flow than conventional filter media. The most common method to produce the nanofibers is using electrospinning method, which can generate fibers with diameters from several nanometers to a few micrometers depending on fabrication conditions (Graham *et al.*, 2002; Gradón *et al.*, 2006; Podgorski *et al.*, 2006; Wang *et al.*, 2008a). In this study nanofiber samples with diameters in the range 150–300 nm were used to filter CNTs.

We investigated the filtration method to calculate the geometrical length of CNTs using the screen filters (TSI 3040) in order to develop a fast measurement of geometrical parameter of elongated particles including CNTs (Bahk *et al.*, 2013). The filtration method is based on the single fiber filtration theory, which includes the capture mechanisms such as diffusion, interception and impaction (Hinds, 1999). We investigated the filtration method using nanofiber filter samples with different solidities in the present study. Compared to our previous study of using mesh screens (Bahk *et al.*, 2013), the present study demonstrates that the method of CNT length determination can be implemented

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with different types of filters, and the accuracy depends on the used filter. In addition, the data here support that nanofiber filters can provide high filtration efficiency for CNTs at relatively low pressure drop. We compared the obtained results with measured geometrical lengths of CNTs by scanning electron microscopy (SEM) analysis.

EXPERIMENTAL SETUP

Fig. 1 shows a schematic diagram of CNT filtration system. A Collison type atomizer was used to generate airborne CNTs. Generated multi walled carbon nanotubes (MWCNTs) went through a diffusion dryer to have liquid vapors from the generator removed and were classified by a differential mobility analyzer (DMA, TSI 3081). In order to avoid electrostatic effect in the filter, a neutralizer (Kr-85) was used. The penetrations through the filters were obtained by measuring concentrations of CNTs upstream and downstream of the filter using condensation particle counters (CPC, TSI 3775). The controlled face velocity on the filter was 5 cm/s. Pressure drop values caused by the filters were measured by a pressure gauge.

15–20 nm diameter MWCNTs (Baytubes, BMS, Germany) were functionalized by the nitric acid refluxing method and dispersed in deionized water. Functionalized MWCNTs, which possess a functional group COOH, with diameters about 20–30 nm (Cheaptubes, USA) were also tested with the deionized water suspension. Fig. 2 shows mobility size distributions of airborne MWCNTs obtained by using a scanning mobility particle sizer (SMPS). Curves possessing peaks around 20 nm sizes were residual particle distributions in the water suspension without CNTs. Distributions of CNTs were well differentiated from the residual particles in the suspensions. 65–160 nm and 75–170 nm mobility size ranges were chosen for Baytubes and Cheaptubes, respectively, to avoid including residual particles and agglomerated CNTs (Bahk *et al.*, 2013). In order to compare the results of CNT filtration with those of spherical particles, PSL particles with 51.4, 95.6 and 193.3 nm diameters, were used. The obtained penetrations of CNTs were compared with the model calculation and also those of PSL particles.

Nanofiber samples with four different solidities, which were obtained by Wang *et al.* (2008a) in the previous study as samples A, B, C and D with solidities 0.134, 0.104, 0.059 and 0.034, respectively, were tested against CNTs with a controlled flow rate. The pressure drops for the sample A to D were 12, 7, 4 and 2 Pa, respectively, with 5 cm/s face velocity on the filters. Fig. 3 show SEM images of nanofiber samples. As shown in the figure, airborne PSL particles and CNTs of different sizes were captured by nanofibers. Substrate micrometer fibers possess about 20 µm diameters and nanofibers on the substrate fibers possess 150–300 nm diameters. In the experiments three layers of the same type nanofiber filters were used in order to show clear differences among the different nanofiber samples. Results were compared with penetrations through screens made of 635-mesh type 304 stainless steel, which have well-defined structures (TSI, Model 3040) and was used by Wang *et al.* (2011a) to investigate the filtration model for airborne CNTs. The solidity α for the 20 layers of screens is 0.345 and pressure drop was 67 Pa with 5 cm/s face velocity.

We developed the filtration method for calculation of geometrical lengths of CNTs from the penetration of particles through filter media in order to develop a fast measurement method of structural characteristic of elongated particles (Bahk *et al.*, 2013). We evaluated the method with CNT penetrations through nanofiber filters and the obtained geometrical lengths by the filtration method were compared with measured lengths by SEM analysis. In order to measure the geometrical length of CNTs, a large number of generated airborne CNTs were collected on silicon substrates with the nanometer aerosol sampler (TSI 3089) and the obtained SEM images were analyzed by an image-processing software (Image J).

FILTRATION MODEL

The single fiber theory including capture mechanisms such as diffusion, interception and inertial impaction, was applied to the model calculation for the filtration of CNTs using nanofiber filters. Penetration of the airborne CNTs

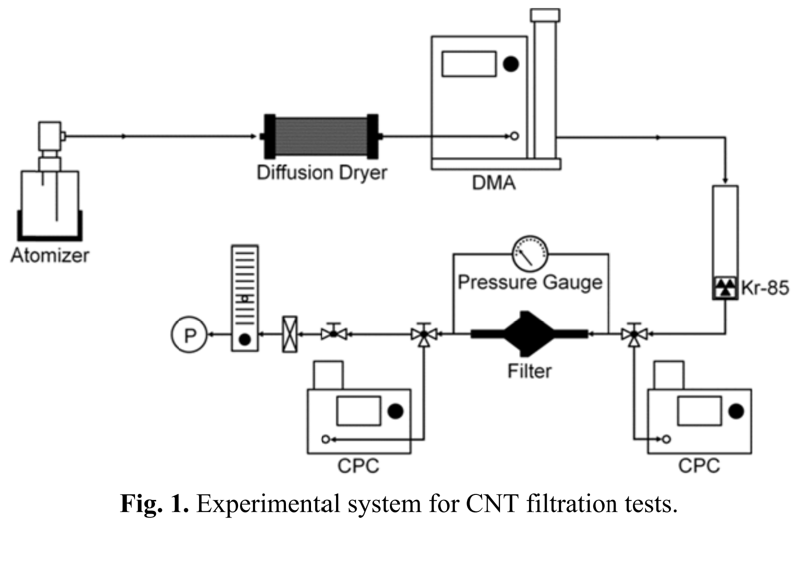

Fig. 1. Experimental system for CNT filtration tests.

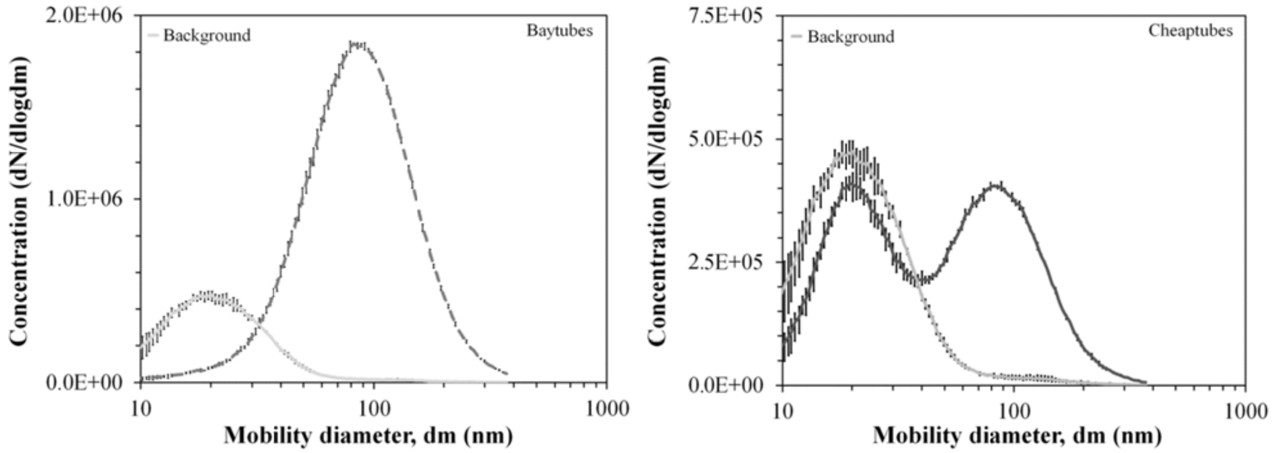

Fig. 2. Size distributions of airborne MWCNTs generated by the atomizer.

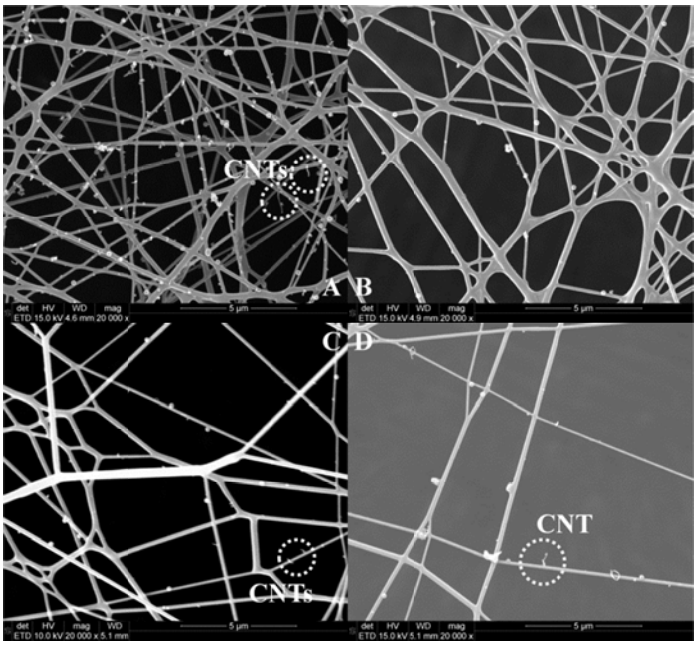

Fig. 3. SEM images of nanofiber filter samples A, B, C and D with solidities 0.134, 0.104, 0.059 and 0.034, respectively. PSL particles and unclassified CNTs were captured by nanofibers.

can be expressed as:

$$P = \exp\left(-\frac{4\alpha E_T t}{\pi d_f (1-\alpha)}\right), \tag{1}$$

where α is the solidity of the filter, E_T is the total efficiency, which is summation of efficiency due to the all capture mechanism considered in the single fiber theory, t is the thickness of filter layer and d_f is the fiber diameter in the filter. 250 nm diameter and 20 µm diameter were used for nanofibers and substrate fibers, respectively. 3 layers of filters were considered in the model and total thicknesses were 750 nm and 450 µm for nanofibers and substrate fibers, respectively.

The single fiber efficiency due to diffusion can be written as (Pich, 1965):

$$E_D = 2.27Ku^{-1/3}Pe^{-2/3}(1 + 0.62KnPe^{1/3}Ku^{-1/3}), \tag{2}$$

where Ku is Kuwabara hydrodynamic parameter, Pe is Peclet number and Kn is Knudsen number and expressed as $Kn = 2\lambda/d_f$.

$$Ku = -\ln\alpha/2 - 3/4 + \alpha - \alpha^2/4, \tag{3}$$

$$Pe = U_0 d_f/D, \tag{4}$$

where U_0 is the face velocity and D is the particle diffusion coefficient. The diffusion coefficient for CNTs can be calculated with mobility diameters of CNTs, since the diffusion coefficient is directly related to the electrical mobility (Wang *et al.*, 2011a).

The efficiency due to interception is given by (Pich, 1966):

$$E_R = \frac{(1+R)^{-1} - (1+R) + 2(1+1.996Kn)(1+R)\ln(1+R)}{2(-0.75-0.5\ln\alpha)+1.996Kn(-0.5-\ln\alpha)} \quad (5)$$

where $R = d_p/d_f$; d_p represents the particle size and is the diameter for spherical particles. In case of elongated particles such as CNTs, different options including the geometrical length and aerodynamic diameter have been considered (Wang *et al.*, 2011b). In the current study, we found the aerodynamic diameter gave results in much better agreement with the experiments. In this case, the interception parameter R for CNTs is

$$R = 2R_{ae}/d_f, \quad (6)$$

where R_{ae} is the aerodynamic radius of a fibrous particle and expressed as:

$$R_{ae} = R_{ae1}\sin^2\psi + R_{ae2}\cos^2\psi, \quad (7)$$

where $R_{ae1} = d_{CNT}\beta/3[\ln(2\beta) - 0.5]$ and $R_{ae2} = 2d_{CNT}\beta/3[\ln(2\beta) + 0.5]$. R_{ae1} is for a parallel orientation, R_{ae2} is for a perpendicular orientation, d_{CNT} is a tube diameter of CNT and $\beta = L_{CNT}/d_{CNT}$ is the aspect ratio of CNT where L_{CNT} is the geometrical length of CNT. Angle ψ for the random orientation takes the value 54.74° (Fu *et al.*, 1990).

The single fiber efficiency due to inertial impaction is written as (Landahl and Herrmann, 1949):

$$E_I = \frac{St^3}{St^3 + 0.77St^2 + 0.22}, \quad (8)$$

where $St = d_{CNT}^3\rho_{CNT}U_0/18\mu d_f R_{ae}$, ρ_{CNT} is the CNT density (= 1.74 g/cm^3, kim *et al.*, 2009), and μ is the air viscosity.

The interaction term E_{DR} to consider interception of particles undergoing diffusion can be written as (Hinds, 1999):

$$E_{DR} = \frac{1.24 \cdot R^{2/3}}{(Ku \cdot Pe)^{1/2}}. \quad (9)$$

According to the theory, when the diameter and density of CNT and fiber diameter are known and penetration is determined experimentally, the geometrical length of CNT can be calculated from the equations.

RESULTS AND DISCUSSION

Penetration of Airborne MWCNTs through Nanofiber Filters

Nanofiber filter samples with four different solidities were tested against airborne MWCNTs. Penetrations of classified MWCNTs and PSL particles were obtained by measuring particle concentrations upstream and downstream of the filter. The size range, we chose for the experiments, was already in the interaction regime of diffusion and interception, and the smaller size end could be more dependent on diffusion, the other end could be more dependent on interception. In the interaction regime, the higher solidity samples showed higher collection efficiency for airborne CNTs as shown in Fig. 4. Penetrations of both types of CNTs showed lower values than those of PSL particles due to the longer effective lengths of CNTs for interception than diameters of PSL particles, which were used as the effective interception length for spheres. It is also explainable by the single fiber theory. The particles possessing longer effective lengths can have more chances to be collected by the nanofibers. The lower solidity sample possessed lower fraction of nanofibers, thus discrepancy between penetrations of CNTs and PSL particles was decreased. It can be seen that discrepancies were decreased with decreasing solidities of filter samples in the figures. Same penetrations were obtained with sample D for all particles used in the experiments, since it possessed lower solidity and the expected effect of nanofibers was insignificant.

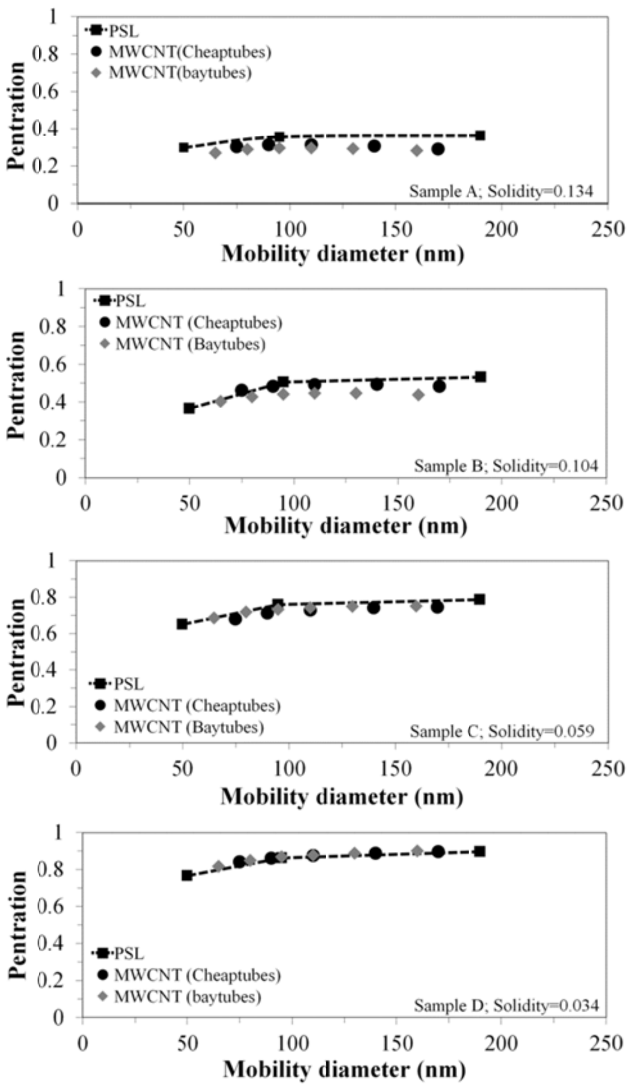

Fig. 4. Penetrations of MWCNTs and PSL particles through nanofiber filter sample A, B, C and D with solidities 0.134, 0.104, 0.059 and 0.034, respectively.

We compared results of filtration using nanofiber filter sample A with those of using diffusion screens (Fig. 5). In order to compare two different kinds of filters, we measured the pressure drop for each filter. Nanofiber filters showed a relatively lower pressure drop with 5 cm/s face velocity than that screens showed and obtained values were 36 Pa and 67 Pa for three layers of nanofibers and screen filters, respectively. Although nanofiber sample A possesses lower solidity than the diffusion screen, higher filtration efficiency was obtained with CNTs. In the equations of single fiber efficiency, the efficiency due to the interception increases when the fiber size decreases. Thus the nanofiber filters showed higher efficiency than screens in the interaction regime, because of their smaller fiber size. The results showed a good agreement with the theory, which represents the relations between the fiber size and the minimum efficiency and most penetrating particle size (MPPS). The minimum efficiency increases and the MPPS decreases when the fiber size decreases (Lee and Liu, 1980; Hinds, 1999). The calculated MPPSs with the experiment conditions were 487 nm and 96.1 nm for the screens and nanofiber filter sample A, respectively. The equation for MPPS was derived by Lee and Liu (1980) and expressed as:

$$\hat{d}_{\mathrm{p}} = 0.885\left(\left(\frac{Ku}{1-\alpha}\right)\left(\frac{\sqrt{\lambda}kT}{\mu}\right)\left(\frac{d_f^2}{U_0}\right)\right)^{2/9}, \quad (10)$$

where k is Boltzmann constant and T is the temperature.

CNT Length Determination by the Filtration Method Using Nanofiber Filters

We investigated the length determination method using filtration model in order to develop the fast geometrical characterization method for elongated particles such as CNTs. We used PSL particles to show the agreement between penetrations calculated by the model and measured in experiments. The comparison showed reasonable agreement as shown in Fig. 6. The figure shows also comparison results between the model and penetration of CNTs passing through the nanofiber filters. The comparison results between the model and experiments showed good agreements for CNTs as well with inserting the measured geometrical length of CNTs into the model as the effective length for the filtration efficiency due to interception.

In order to evaluate the fast length determination of CNTs using filtration method, we calculated the lengths of CNTs by inputting penetration of CNTs through the nanofiber filters as a parameter to the model. The results were shown in Fig. 7 and Fig. 8, for CNTs from Cheaptubes and Baytubes, respectively. The obtained geometrical lengths of CNTs were compared with measured lengths by SEM analysis. The uncertainties due to the measurement variation among several trials (± 1%) and fluctuation of the filtration velocity in the system (± 3%) were considered in the model. The standard deviation was used to insert the error bars for obtained lengths by SEM analysis. The comparisons showed reasonable agreement in the range that we chose in the study, however, results for the smaller

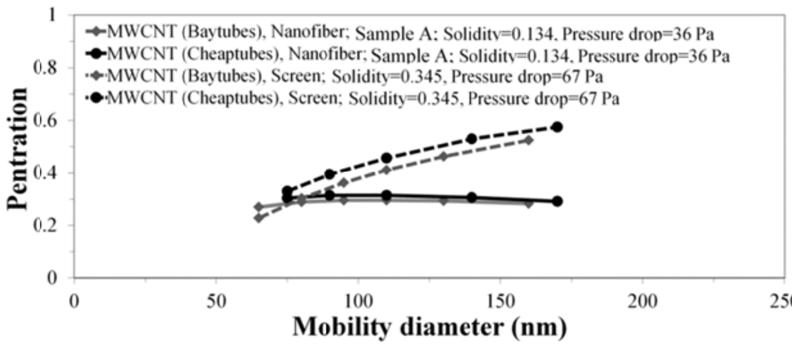

Fig. 5. Comparison of the penetration results between using 3 layers of nanofiber filter sample A and 20 layers of diffusion screens.

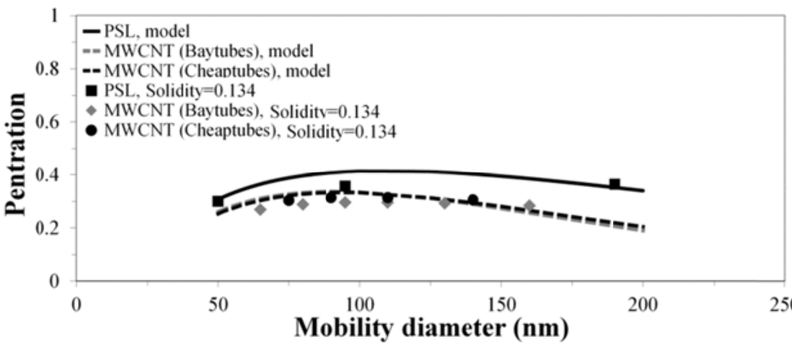

Fig. 6. Comparison of penetration results of experiments of CNTs and PSL particles through the nanofiber sample A with obtained penetration from filtration model.

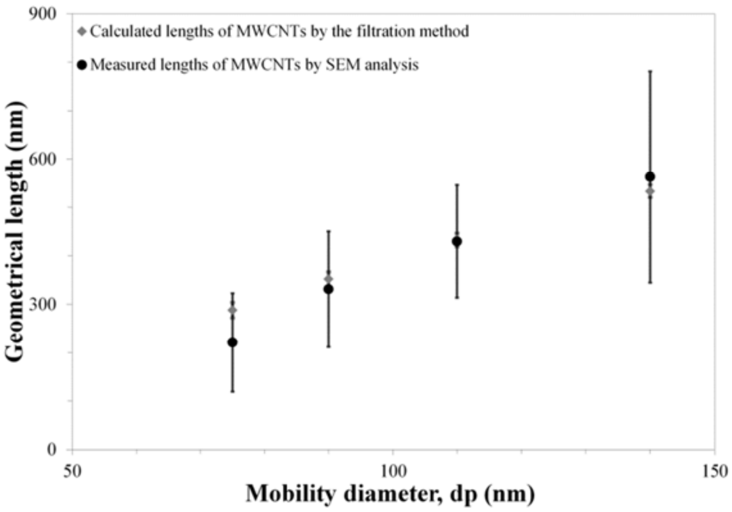

Fig. 7. Comparison between calculated lengths of CNTs using filtration method and measured geometrical lengths of CNTs by SEM analysis (Cheaptubes).

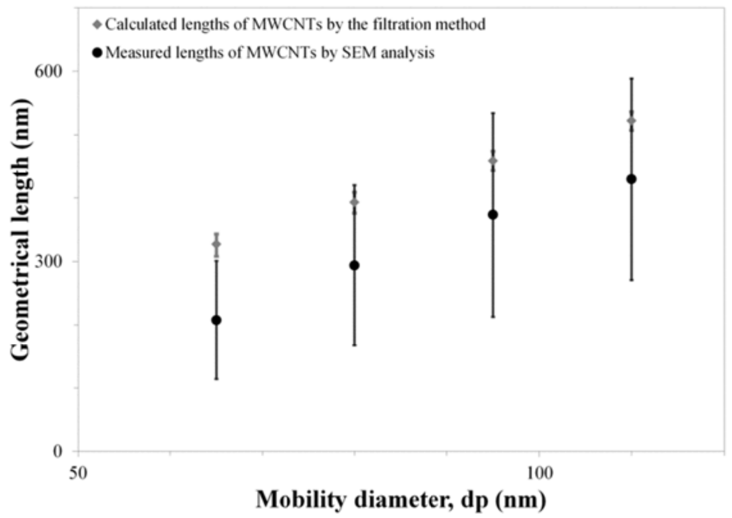

Fig. 8. Comparison between calculated lengths of CNTs using filtration method and measured geometrical lengths of CNTs by SEM analysis (Baytubes).

size end in the range showed bigger discrepancy than larger size end in the range. The reason might be that smaller size particles are mainly captured by diffusion rather than interception, thus the model, which relies on the efficiency due to interception to compute the geometrical length, showed a discrepancy in the smaller size range.

Using the aerodynamic diameters of CNTs for the interception parameter gave rise to better agreement between the model and experiments than using the measured geometrical lengths of CNTs. The reason is that the consideration of aerodynamic diameter includes effects of bending and curling of CNTs fortuitously, because when bending and curling happen on CNTs the interception lengths of CNTs are shortened (Wang *et al.*, 2011b). However, the aerodynamic diameters of CNTs used in the model for interception might be shorter than the actual interception lengths of CNTs and led to a model overestimation of the penetration, which can be seen in Fig. 6 especially for Baytubes in the range of 65–110 nm. This also provides an explanation for the discrepancy between the length calculation and measurement for Baytubes in Fig. 8. The better agreement for Cheaptubes shown in Fig. 6 and Fig. 7 indicates that the aerodynamic diameter is a better approximation to the interception length in this case.

In the model, condition of filters during the experiments as particle loading effect on the filters is not considered. That might be another reason for the discrepancy between the model and experimental results. The measured and calculated lengths, penetration through the nanofiber filters and uncertainties for each case are listed in Table 1.

CONCLUSIONS

In the study the nanofiber filter was successfully investigated for the filtration of airborne CNTs. We tested nanofiber filters with different solidities against CNTs and

Table 1. Measured and calculated geometrical lengths of CNTs.

Cheaptubes						
d_m	Measured length from SEM (nm)	σ_g	Penetration	Calculated length (nm)	Minimum length considering uncertainty (nm)	Maximum length considering uncertainty (nm)
75	221	1.46	0.303	288	272	304
90	331	1.36	0.314	352	337	367
110	430	1.27	0.315	434	419	447
140	563	1.34	0.306	533	521	547
Baytubes						
d_m	Measured length from SEM (nm)	σ_g	Penetration	Calculated length (nm)	Minimum length considering uncertainty (nm)	Maximum length considering uncertainty (nm)
65	207	1.45	0.269	327	308	343
80	294	1.43	0.289	393	376	409
95	373	1.43	0.296	459	444	474
110	429	1.37	0.296	522	507	536

compared with the filtration model, which includes the particle capture mechanisms. The comparison showed good agreements between the model and experiments for both PSL particles and CNTs. CNTs showed lower penetration through the higher solidity samples than PSL particles with comparable mobility diameters as expected in the model. That is caused by the longer effective interception lengths of CNTs than PSL particle diameters. CNT penetrations through the nanofiber filters were also compared with those through the diffusion screen filters. The nanofiber filter with 0.134 solidity, which led to lower pressure drop than screens, showed higher collection efficiency for the CNTs than screens, especially with larger CNT sizes in the range.

We evaluated the fast length determination method for CNTs using the filtration model with the nanofiber filter sample with 0.134 solidity. We calculated the lengths of CNTs using the filtration method by inputting the penetration, which was obtained experimentally, as a parameter to the model, and compared with measured lengths by the SEM analysis. Both CNTs from different providers showed reasonable agreements with measured lengths from SEM measurements. Since the chosen ranges were in the interaction regime between diffusion dominant regime and interception dominant regime, comparisons showed much discrepancy in the smaller end closed to the diffusion dominant regime. The filtration method is able to be applied for the fast geometrical measurement of elongated particles.

ACKNOWLEDGMENTS

This study was partially supported by the Swiss National Science Foundation (NFP 64), "Evaluation platform for safety and environment risks of carbon nanotube reinforced nanocomposites", 406440 131286. The authors thank the support of members of the Center for Filtration Research: 3M, Boeing Commercial Airplanes, Cummins Filtration Co. Inc., Entergris Inc., Ford Co., Hollingworth & Vose Co., MANN+HUMMEL, GMBH, MSP Corp., Samsung Electronics Co. Ltd, Shigematsu Works Co. Ltd, TSI Inc., and W. L. Gore & Associates Inc. and affiliate member NIOSH.

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Received for review, January 8, 2014
Accepted, April 1, 2014