Evaluation of Filter Media for Particle Number, Surface Area and Mass Penetrations LIN LI¹*, ZHILI ZUO¹, DANIEL A. JAPUNTICH² and DAVID Y.H. PUI¹

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The National Institute for Occupational Safety and Health (NIOSH) developed a standard for respirator certification under 42 CFR Part 84, using a TSI 8130 automated filter tester with photometers. A recent study showed that photometric detection methods may not be sensitive for measuring engineered nanoparticles. Present NIOSH standards for penetration measurement are mass-based; however, the threshold limit value/permissible exposure limit for an engineered nanoparticle worker exposure is not yet clear. There is lack of standardized filter test development for engineered nanoparticles, and development of a simple nanoparticle filter test is indicated. To better understand the filter performance against engineered nanoparticles and correlations among different tests, initial penetration levels of one fiberglass and two electret filter media were measured using a series of polydisperse and monodisperse aerosol test methods at two different laboratories (University of Minnesota Particle Technology Laboratory and 3M Company). Monodisperse aerosol penetrations were measured by a TSI 8160 using NaCl particles from 20 to 300 nm. Particle penetration curves and overall penetrations were measured by scanning mobility particle sizer (SMPS), condensation particle counter (CPC), nanoparticle surface area monitor (NSAM), and TSI 8130 at two face velocities and three layer thicknesses. Results showed that reproducible, comparable filtration data were achieved between two laboratories, with proper control of test conditions and calibration procedures. For particle penetration curves, the experimental results of monodisperse testing agreed well with polydisperse SMPS measurements. The most penetrating particle sizes (MPPSs) of electret and fiberglass filter media were ~50 and 160 nm, respectively. For overall penetrations, the CPC and NSAM results of polydisperse aerosols were close to the penetration at the corresponding median particle sizes. For each filter type, power-law correlations between the penetrations measured by different instruments show that the NIOSH TSI 8130 test may be used to predict penetrations at the MPPS as well as the CPC and NSAM results with polydisperse aerosols. It is recommended to use dry air (<20% RH) as makeup air in the test system to prevent sodium chloride particle deliquescing and minimizing the challenge particle dielectric constant and to use an adequate neutralizer to fully neutralize the polydisperse challenge aerosol. For a simple nanoparticle penetration test, it is recommended to use a polydisperse aerosol challenge with a geometric mean of ~ 50 nm with the CPC or the NSAM as detectors.

Keywords: correlation; filter tests; nanoparticle; overall penetration; penetration curve

INTRODUCTION

*Author to whom correspondence should be addressed. Tel: +3145182090; fax: 6126256069 email: llin@umn.edu Engineered nanoparticles are encountered in industrial workplaces including electronic, magnetic and optoelectronic, biomedical, pharmaceutical, cosmetic, energy, and catalytic applications (Stamatakis and Natalie, 1991). As more studies reveal which materials in nanoparticle (<100 nm) size ranges may be associated with deleterious health effects (Oberdörster et al., 2005, 2007; Gwinn and Vallyathan, 2006; Kreyling et al., 2006), it will be necessary to monitor and control them where workers or the public are potentially exposed. To reduce the inhalation of airborne inert and biological particles, respiratory protection is often required. Many researchers have evaluated the filter penetration (the ratio of downstream to upstream concentration) performance of air filter media and personal protective equipment against engineered nanoparticles (Shaffer and Rengasamy, 2009; Mostofi et al., 2010; Lore et al., 2011; Rengasamy et al., 2011). However, there is lack of standardized filter test methods for engineered nanoparticles.

The National Institute for Occupational Safety and Health (NIOSH), in cooperation with the Mine Safety and Health Administration (MSHA), developed a standard for the certification of air-purifying respirators under 42 CFR Part 84 (1996). The NIOSH certification filter penetration test uses a TSI Inc. 8130 automated filter tester with light scattering photometers to measure the upstream and downstream particle mass concentrations. A recent study showed that the photometric method could not effectively detect engineered nanoparticles (<100 nm in size) due to its insensitivity to the light scatter of nanoparticles, which brought into question the capability of the current NIOSH standard in predicting the penetration of ultrafine particles through respirators (Eninger et al., 2008a).

The particle size that gives the highest penetration, also known as the most penetrating particle size (MPPS), has been studied both theoretically and experimentally. The shift in MPPS for respirator filters from 100-300 nm (uncharged filters) to about <100 nm (electret filters) has been discussed in the literature (Lee and Liu, 1980; Brown, 1981; Kanaoka et al., 1987; Hinds, 1999; Balazy et al., 2006a; Huang et al., 2007; Eninger et al., 2008a). For over 25 years, the MPPS for electret filters has been shown to be predictable from filter structure, fiber charge and challenge aerosol characteristics (Emi et al., 1984). A polymeric electret in a fiber may be defined as a bipolar domain of dielectric material exhibiting a quasi-permanent electrical charge. Electret filter medium filtration performance is unaffected by high humidity storage (Ackely, 1982), and such filters may be created by many different methods (Meyers et al., 2003).

Using particle spectrometers capable of sizing and counting particles down to the 2.5 nm level, several

researchers have measured nanoparticle penetration through filter media and respirators. Filter penetration as a function of particle size was obtained by measuring particle concentrations upstream and downstream with condensation particle counters (CPCs) using a monodisperse aerosol or with a scanning mobility particle sizer (SMPS) using polydisperse aerosol challenges. Japuntich et al. (2007) and Lore et al. (2011) found nearly identical penetration results using these two test systems, when sources of error and proper calibration procedures were addressed. For a limited number of N95 electret respirators (NIOSH certified by photometric penetration filter testing), higher than 5% penetrations as measured by particle count, not mass, were found at the MPPS from 40 to 60 nm (Martin and Moyer, 2000; Moyer and Bergman, 2000; Bałazy et al., 2006a,b; Rengasamy et al., 2007, 2008, 2011; Eninger et al., 2008b).

Penetration measurements by current NIOSH standards are mass-based, e.g. related to a mass concentration threshold limit value/permissible exposure limit (TLV/PEL) (e.g. 5% penetration is by mass, not by count). Although traditional massbased permissible exposure limits (PEL) exist for many of the substances that engineered nanoparticles are made from, the PEL for an engineered nanoparticle challenge worker exposure is not yet clear and under extensive research. Furthermore, there is still a debate on the exposure metric that is most appropriate: mass, surface area, or number (count). There have been attempts to establish a multiple-metric approach or integrate the single matrices. It may be hypothesized that filter testing for engineered nanoparticles will need to be by count or by surface area and not by mass-calibrated photometers, due to their insensitivity to nanoparticles (<100 nm diameter).

The use of one test to predict the performance of another test is common in science and in filtration testing. Johnson and Smith (1998) showed that different aerosols and filtration measurement techniques, such as photometric and count measurements, resulted in different penetration results, but that strong predictions may be made with correlative comparisons to monodisperse challenges. Pierce (2006) presented a power-curve correlation between the MPPS penetration as measured by the TSI CertiTest 8160 monodisperse aerosol test and the penetration values obtained by the TSI model 3140 (a polydisperse aerosol variation of the NIOSH certification test using CPCs). In addition, Pierce (2006) presented a correlation of MPPS penetration with filter media face velocity that may be useful for extrapolation purposes.

Particle surface area as a measure of the ability of engineered nanoparticles to coat the interior surface of the lungs has recently been reported to play an important role in toxicity studies of engineered nanoparticles (Tran et al., 2000; Oberdörster, 2001; Dick et al., 2003; Stoeger et al., 2006; Duffin et al., 2007; Monteiller et al., 2007; Sager and Castranova, 2009). Although the health effect prediction of the concentration of engineered nanoparticles might be small in terms of mass, it might be quite large if based on surface area. The nanoparticle surface area monitor (NSAM) device has been developed to monitor the surface area of inhaled particles deposited in the lung (Shin et al., 2007). Compared with photometric measurements, an NSAM detects particles <20 nm, and it needs less maintenance than CPC. Thus, it is surmised that the NSAM may be used to measure 'surface area' upstream and downstream concentrations as a possible filter penetration test (Stanley et al., 2010).

Aerosol filtration testing involves many complicated parameters, e.g. particle charge, environmental humidity and, most importantly, the detection methodology, which may give different test results between laboratories without careful control (even when the same filter media are tested under similar test conditions). To test if the NIOSH standards can correlate to and be able to predict the same information (e.g. how good a filter is) as obtained by other test methods, the performances of three filter media for particle number, lung deposited surface area, and mass penetrations were evaluated at two different laboratories: 3M Company in St Paul, MN and University of Minnesota (UMN) in Minneapolis, MN. Monodisperse particle penetrations through the media were measured at 3M Company using a TSI 8160 automated filter tester. Particle penetration versus particle diameter curves and penetrations from

overall number, lung deposited surface area, and mass were measured using an SMPS, CPC, NSAM, and TSI 8130 automated filter tester with polydisperse sodium chloride (NaCl) particles. For each filter at one test condition, we repeated the experiment three to five times and used mean values as the presented results. We further compared and correlated the results among the five tests (monodisperse test, SMPS, CPC, NSAM, and TSI 8130). Procedures for calibration and test methodology were developed with the goal of minimizing variability between laboratories.

MATERIALS AND METHODS

Flat filter media

Three different types of filter media (one fiberglass filter and two electret filters) were used for comparisons and correlations between the two laboratories. For each filter medium, Table 1 shows an assortment of filtration parameters. Tested at a constant face velocity, these media were chosen to have similar single layer filter penetrations within a halflog decade at each of their respective most penetrating particle size ranges. The uncharged fiberglass filter was HD-2583, an HVAC medium made by Hollingsworth and Vose of East Walpole, MA. The polypropylene electret filters were manufactured by 3M Company, St Paul, MN. Fiber charge density was estimated using the ionizing X-ray bipolar fiber charge method as in Brown (1979) with cavity correction as in Waker and Brown (1988). Correlations of microscopic median fiber diameter versus effective fiber diameter (Davies, 1952) yielded a microscopic median fiber diameter as one-half the effective fiber diameter for the electret filters for the charge calculation. Electret 1 is more highly charged

Table 1. Characteristics of the fiberglass and electret filter media.

Air filtration media parameters	Fiberglass HD-2583	Electret 1	Electret 2	
Thickness (cm)	0.060	0.069	0.077	
Basis weight (g m ⁻²)	79.8	57.7	56.3	
Fiber density (g cm ⁻²)	2.4	0.92	0.92	
Solidity (–)	0.056	0.089	0.078	
Effective fiber diameter (µm) (Rubow, 1981)	1.9	7.1	6.9	
Effective fiber diameter (µm) (Davies, 1952)	2.1	7.6	7.5	
Effective pore diameter (µm) (Benarie, 1969; Chen, 1955)	8.2	22.7	24.1	
Pressure drop (mmH ₂ O) at 5.3 cm s^{-1}	11.3	2.1	2.1	
Penetration (–) TSI 8130 AFT: NaCl at 5.3 cm s^{-1}	3.94	0.082	3.23	
Figure of merit (mmH ₂ O ^{-1}) [= $-\ln(\% \text{pen.}/100)/\text{press. drop})$] (Chen, 1955)	0.29	3.38	1.63	
Manufacturer of filter	H&V	3M Co.	3M Co.	

(118 μ C m⁻²) for high-efficiency filter laminates, whereas Electret 2 has a lower charge (25 μ C m⁻²) and is used for lower efficiency filter laminates. The three filter media were tested at two face velocities and three thicknesses to give a wide range of performances. It should be noted that these are flat filter medium initial filter penetration tests, and when these media are incorporated into various filtration device designs, the filtration penetration and flow resistance test results may be different.

In this study, the flat filter media was mounted and clamped in the filter test chucks and tested at the face velocity of 5.3 and 10.6 cm s⁻¹. The 10.6 cm s⁻¹ is roughly the face velocity of a typical N95 respirator having a surface area of 135 cm² at 851 min⁻¹, the flow rate for N95 certification (NIOSH 42 CFR Part 84). High-efficiency non-woven P100 filters may have a face area (two filters/respirator) of 270 cm² at 851 min⁻¹, which gives a face velocity of 5.3 cm s⁻¹. Typical face velocities of pleated high-efficiency filter papers have filter medium face velocities <5.3 cm s⁻¹, making this a conservative choice.

Monodisperse NaCl aerosol test (3M)

The TSI 8160 automatic filter tester, first introduced in 1987, was designed for conducting particle size-dependent penetration measurements on high-efficiency air filter media (an updated design from TSI is the TSI 3160 automated filter tester). The TSI 8160 is a fully self-contained testing apparatus for conducting initial filter penetration tests of single particle size within a range between 15 and 400 nm diameter using challenge aerosols of dioctyl phthalate (DOP) and NaCl. For this study, as shown in Fig. 1, three TSI constant output atomizers individually supplied NaCl aerosol at 3.75 l \min^{-1} from feedstock solution concentrations of 1, 0.1, and 0.01% NaCl by weight in distilled water, respectively. Only polydisperse NaCl aerosol was generated and flowed to a TSI model 3071 electrostatic classifier to select a very narrow size distribution as a challenge aerosol for filter testing. Exiting the electrostatic classifier, the aerosol was brought to the Boltzmann equilibrium charge distribution by passing through a 10 mCi Kr-85 neutralizer. Prior to the filter testing chucks, the aerosol flowed through a dilution bridge into an aerosol manifold where compressed air of <20% RH was served as dilution air. We also tested with summertime outside room air of 70% RH as the other option for makeup air, which was used in the original TSI 8160 setup. After dilution, the challenge aerosol was at concentration levels within the single particle counting mode capabilities of the CPCs. The volumetric flow rate and the pressure drop across the media were monitored in the TSI 8160 system. The percent penetration was calculated as the ratio of downstream to upstream concentration measured by two TSI 3760A CPCs, sampling at 1.4 l min⁻¹. In this device, to increase the filter penetration range, the upstream concentration is diluted using a primary split-flow diluter. To compensate for the possible error from two CPCs due to particle size sensitivities and different sampling line losses, the manufacturer has set up a means of calculating correction factors for each requested challenge particle

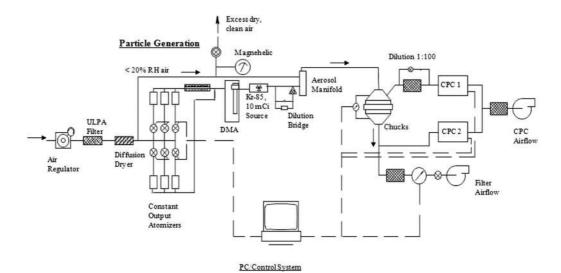


Fig. 1. Experimental schematic of a TSI 8160 for monodisperse aerosol tests at 3M Company.

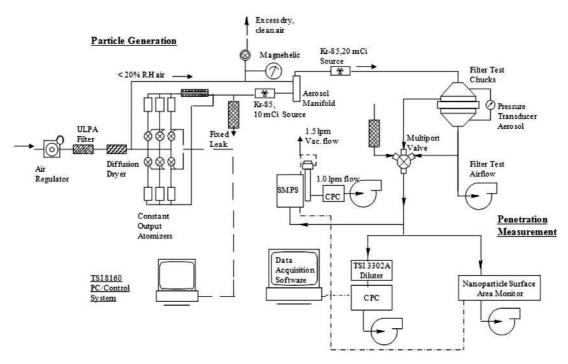


Fig. 2. Experimental schematic for polydisperse aerosol filter testing using an SMPS, CPC, and NSAM at 3M Company.

diameter. The TSI 8160 system was calibrated as described in Japuntich *et al.* (2007).

Polydisperse NaCl aerosol test (3M)

The polydisperse aerosol test system at 3M Company consisted of an aerosol generation subsystem, part of the TSI 8160, and particle measurement subsystem, as shown in Fig. 2. In the aerosol generation system, polydisperse NaCl aerosol was generated from the constant output atomizer with a portion of the flow allowed to exhaust through the fixed leak to reduce the aerosol concentration to a suitable level for measurements. The aerosol then flowed through a 10 mCi Kr-85 neutralizer into the aerosol manifold, where dry compressed air or outside 70% RH room air was filtered and used as the makeup air. Prior to the filter testing chucks, the aerosol flowed through another 20 mCi Kr-85 neutralizer for full neutralization. We also tested the case when no neutralizer was used to investigate the neutralization effect. The filtration penetration testing system consists of pneumatic chucks to hold the filter medium with a crosssectional area of 100 cm², a pressure transducer and a sampling system for the sequential measurement of the particle concentration upstream and downstream of the filter sample in the chucks, by using an SMPS, CPC, and NSAM (TSI Model 3550). The TSI 3936 SMPS system used a TSI 3786 ultrafine condensation particle counter and the TSI 3081 differential mobility analyzer (DMA) column. Previous research found that for higher upstream SMPS particle concentrations (> $8 \times 10^6 \ \text{\# cc}^{-1}$), the time it took to purge the Kr-85 neutralizer in the TSI 3936 SMPS system between upstream and downstream samples was greater than 3 min to obtain stable penetration measurements for a 1 l min⁻¹ CPC flow, and even then was erratic (Japuntich et al., 2007). The addition to the SMPS system of a vacuum flow of 1.5 l min⁻¹ between the neutralizer and CPC increased the flow through the neutralizer to 2.5 l min⁻¹, which brought purge times to <1 min and gave stable upstream and downstream concentrations for the penetration calculations. A check on particle size measurements with and without the additional vacuum flow gave identical size distributions, showing the neutralizer was functional at the higher flow.

Polydisperse NaCl aerosol test (UMN)

Figure 3 shows the experimental setup of polydisperse aerosol filter tests at UMN. A constant-output atomizer (TSI Model 3076) was used to produce polydisperse particles with 0.01, 0.1 and 1% NaCl solutions (Liu and Pui, 1974). The flow rate output from the atomizer was 4.0 1 min⁻¹ when the compressed

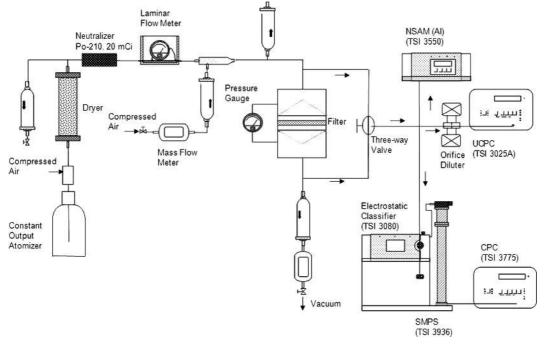


Fig. 3. Experimental schematic of polydisperse aerosol filter testing using an SMPS, CPC, and NSAM at UMN.

air pressure was at 30 psig. Droplets produced by the atomizer were passed through two diffusion drvers to dry the particles and also through a 20 mCi Po-210 radioactive neutralizer to remove electrical charges on the particles. The aerosol stream was bled off at 1 l min⁻¹ through the laminar flow element and then mixed with compressed air to lower the concentration of the test aerosol within the measurement range of the NSAM and SMPS. The test aerosol went into the pneumatic chucks holding the filter media with a cross-sectional area of 100 cm². Two plates with smaller holes of 50 cm² were used to hold the filter media and double the test velocity without any change of the air flow rate. A pressure gauge was used to monitor the pressure drop across the filter media. By matching the pressure drops with those of monodisperse tests by adjusting the flow, we could get the same face velocities as those of monodisperse tests. A three-way valve was used to switch between upstream and downstream for SMPS (TSI Model 3936 with TSI 3775 CPC), CPC (TSI Model 3025A), and NSAM (TSI Model 3550) measurements with the sampling flow rate $\sim 4.5 \ lmin^{-1}$. The sampling place was at the side of the filter chuck, similar to that used in TSI 8130. The overall number and lung deposited surface area penetrations were calculated as the ratio of downstream to upstream concentrations measured by CPC and NSAM, respectively. Particle penetration

curve was obtained as the ratio of downstream to upstream size distributions measured by SMPS. Similar to the study by Japuntich *et al.* (2007), to compensate for the particle loss in the filter chucks and transportation, particle penetrations through the chucks without filter were measured by SMPS, CPC, and NSAM, which were used as calculating correction factors at each test condition.

TSI 8130-photometric method

For the tested filter media, overall mass-based penetration were measured by a TSI 8130 used for NIOSH certification. Penetration obtained from the TSI 8130 is based on the measurement of the flux of light scattering from particles upstream and downstream of the media. Initial penetration levels for polydisperse NaCl particles were measured to avoid a loading effect for better comparison and correlation with other tests. The test face velocities were set at 5.3 and $10.6 \,\mathrm{cm \, s^{-1}}$, the same as the other tests.

RESULTS AND DISCUSSION

In total at the two laboratories, five filtration test protocols were performed including: polydisperse SMPS, CPC, NSAM, TSI 8130, and monodisperse penetration tests. The characteristics of monodisperse

Table 2. Particle sizes of monodisperse aerosol test at 3M Company.

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Particle size (nm) (room air)	23	30	47	85.9	165.4	256.1	307
Particle size (nm) (compressed air)	25.1	30.9	48.5	91.4	161.8	280	

Table 3. Characteristics of polydisperse challenge aerosol between two laboratories.

Polydisperse challenge aerosol	3M			UMN		
	0.01% NaCl	0.1% NaCl	1% NaCl	0.01% NaCl	0.1% NaCl	1% NaCl
Count median diameter (nm)	36.4	51.9	68.1	37.4	45.8	67.0
Geometric standard deviation	1.50	1.68	1.89	1.80	1.82	1.93
Lung deposited surface area median diameter (nm)	43.0	76.5	90.0	54.1	64.0	92.1

and polydisperse challenge particles between the two laboratories are listed in Tables 2 and 3, respectively. The count median diameters and standard deviations of polydisperse aerosol were calculated by Sigma-Plot from the log-normal curve fitting of SMPS data. The count median diameters between the two laboratories were very close to each other, and were varied from 36 to 68 nm with NaCl solution from 0.01 to 1% by weight, respectively. The standard deviations of the challenge particles at UMN were generally larger than those at 3M Company, especially for the 0.01% NaCl solution. To calculate the lung deposited surface area median diameter, the number-based size distribution of challenge aerosol was combined with the lung deposition curve of the alveolar region to get the lung deposited particle size distribution (Shin et al., 2007). Then the Hatch-Choate conversion equation was used to calculate the lung deposited surface area median diameter from the count median diameter and standard deviation of the lung deposited particle distribution (Hinds, 1999). The lung deposited surface area median diameter was varied from 43 to 92 nm for NaCl solution from 0.01 to 1% by weight, respectively.

Penetration curves

Monodisperse particle penetrations through the filter media were measured at classified particle sizes ranging from 20 to 300 nm using the TSI 8160 in 3M Company. Figure 4 shows the penetration results of monodisperse tests using very dry (<20% RH) compressed air or summertime room air (70% RH) for dilution. Generally for electret filter media, the monodisperse penetration dropped obviously when using the room air with high humidity as dilution makeup air, while there was not too much difference for the fiberglass filter media. It is well known that NaCl deliquesces into water droplets >50% RH (Dai *et al.*, 1997; Luna *et al.*, 1999), and the 3M Company summer humidity was >70% RH. The humidity effect on NaCl for this testing was then not only a particle size change from solid to liquid (deliquescence), but also an unknown dielectric constant change from ~6 (dry NaCl) to a higher dielectric constant at a higher humidity. The net result of a higher dielectric constant is a lower penetration for the electret filters due to better induced dipole capture (Brown 1993; Lore *et al.*, 2011). For this reason, the Electret 1 filter with the highest electret charge was most sensitive to the humidity, which resulted in more obvious penetration drops.

Polydisperse particle penetrations as a function of particle size in the size range from 10 to 400 nm were measured by an SMPS at each laboratory and the results were also shown in Fig. 4. The SMPS penetration curves of each type of filter media were the average results of three polydisperse challenge aerosols. The penetration curves were visually very similar between the two laboratories. The average penetration difference was <1.5%. The UMN SMPS penetration curves were generally more variable than the 3M results especially at three layer thickness conditions, probably due to the lower challenge aerosol concentrations, which were ~30% less at UMN than those at 3M Company. The penetrations of polydisperse and monodisperse tests using very dry compressed air visually agreed well with each other. The maximum penetration difference between the monodisperse and polydisperse aerosol tests was generally <3%. The penetration curves of Electret 2 media were generally higher and flatter than those of Electret 1 media. Slightly higher penetration levels were obtained at higher face velocity for the different size particles. The MPPSs of the electret and fiberglass filters were ~50 and 160 nm, respectively, which were consistent with previously reported MPPS values (Japuntich et al., 2007; Shaffer and Rengasamy, 2009; Lore et al., 2011).

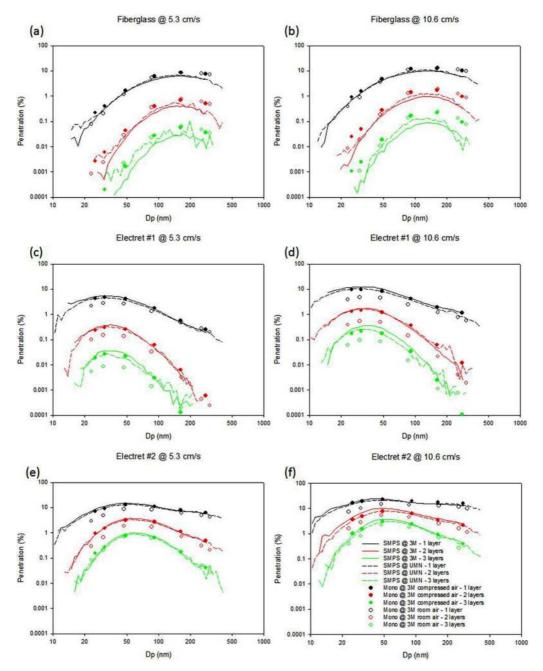
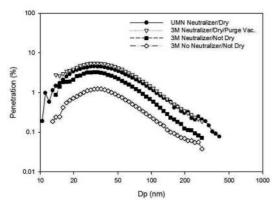


Fig. 4. Comparison of penetration curves for one fiberglass and two electret filter media between monodisperse and polydisperse SMPS measurements at two laboratories.

Figure 5 shows the SMPS penetrations of the polydisperse aerosol test through Electret 1 filter media with a face velocity of 5.3 cm s^{-1} at different levels of humidity and neutralization. The 3M penetration data for Electret 1 shows the progressive improvements of neutralization, dry aerosol, and proper purge flow for data similarity to UMN results (which was already at 10% RH and highly neutralized). Particle penetrations were lower when there was no neutralizer after the aerosol manifold or when using room air with high humidity as the makeup air. Aerosol neutralization is the other important factor in filter

589



Electret #1 @ 5.3 cms-1 layer

Fig. 5. Penetration curves of Electret 1 filter at different humidity and neutralization conditions.

penetration measurement. The Electret 1 filter had the highest charge and was most sensitive to the degree of aerosol neutralization by the Kr-85 neutralizers. The original TSI 8160 system had a Kr-85 neutralizer (TSI 3077A) of ~7 mCi activity in-line from the aerosol diffusion dryer, which was adequate for the low aerosol concentration from the DMA system for monodisperse aerosols. However, when the full polydisperse concentration flowed from the nebulizer for SMPS testing, it was inadequate for neutralization. In other words, particles still carried a large number of charges, which caused more capture due to coulombic attraction and resulted in lower penetration through the electret filter media (Kanaoka et al., 1987; Balazy et al., 2006a). A large neutralizer (TSI 3054A) with about 15 mCi activity was added to the aerosol flow after the dilution with compressed air. This raised the 3M penetration curve to become similar to the results from UMN.

SMPS and overall penetrations

The overall number and lung deposited surface area penetrations were measured by CPC and NSAM at each laboratory and are shown in Fig. 6. Three filter media were challenged with three polydisperse aerosols at two face velocities and three thicknesses. The plotted data points are the averages of replicate tests with the count median diameters and lung deposited surface area median diameter as the corresponding particle sizes of CPC and NSAM measurements. At some test conditions, the downstream aerosol concentrations were too low to have valid NSAM readings. Therefore, there is no result of NSAM penetration at these test conditions. Generally, the overall penetrations were close to the penetration curves at the corresponding particle sizes. For fiberglass filter media, the overall penetrations were lower than the maximum value at the MPPS because the corresponding particle sizes were far away from the MPPS. However, for electret filter media, the overall penetrations were closed to the maximum value since the corresponding particle sizes were within the range of the MPPS, which is not really a single particle size but a wide range of almost a third of a particle diameter decade.

Generally, the overall penetrations were visually very close to each other between the two laboratories. There was a small discrepancy for fiberglass filter media when using smaller polydisperse challenge aerosols. One reason is the difference in the geometric standard deviation of polydisperse aerosols generated in the two laboratories, e.g. 1.80 at UMN and 1.50 at 3M for 0.01% NaCl solution. Larger geometric standard deviations of challenge aerosol mean a wider size distribution, which takes account of higher penetration of larger particles from the penetration curve of fiberglass filter media. The difference of particle size sensitivities and different sampling line losses for CPCs and diluters used in the two laboratories may cause the discrepancy in penetration results.

For a simple nanoparticle penetration test, it is recommended to use a polydisperse aerosol ~50 nm with the CPC or the NSAM as detectors. For polydisperse aerosols with geometric means <50 nm, a large portion of particles may be below the detection limit of a CPC or the NSAM. For polydisperse aerosol challenges of larger sizes, deposition within the experimental system may cause clogging in the transportation tube or connection.

Correlations among monodisperse and polydisperse aerosol tests

To better understand the relationships between the polydisperse photometric (TSI 8130) and the CPC and NSAM tests, the CPC penetrations were plotted as a function of the TSI 8130 data for the same filter media at the same face velocities and thicknesses, shown in Fig. 7. Power-law curves were used to fit the data points from each laboratory. All the data and fitting curves were visually very close between laboratories. For the fiberglass filter media, the CPC penetrations are slightly lower than TSI 8130 results. However, for electrets filter media, the CPC penetrations are about two orders of magnitude higher than TSI 8130 results. If we plot the NSAM penetrations as a function of TSI 8130 data at the same test conditions, power-law curves can also be obtained and similar trends can be found to the CPC and TSI 8130 data sets.

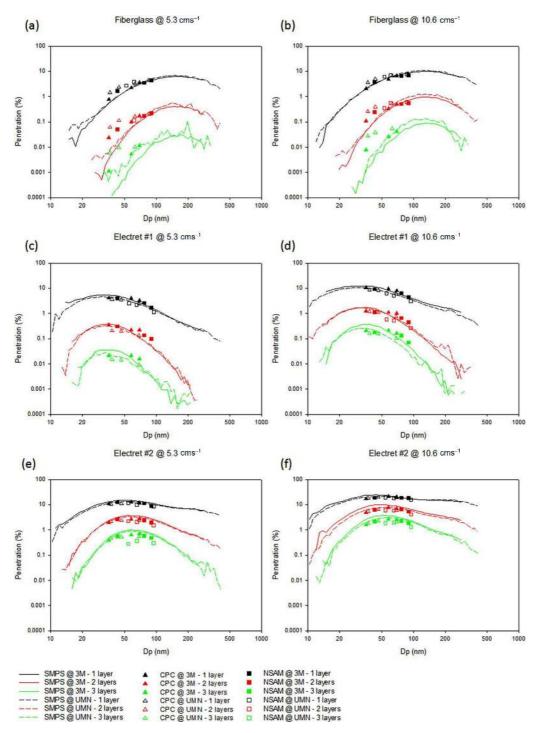


Fig. 6. Overall CPC and NSAM penetrations of one fiberglass and two electret filter media at two laboratories.

Figure 8 shows an example of the correlations between monodisperse and polydisperse tests. Based on the monodisperse aerosol test, the MPPSs for fiberglass and electret filter media were ~ 161.8 nm and 48.5 nm, respectively. We then plotted the overall penetrations from NSAM as a function of the



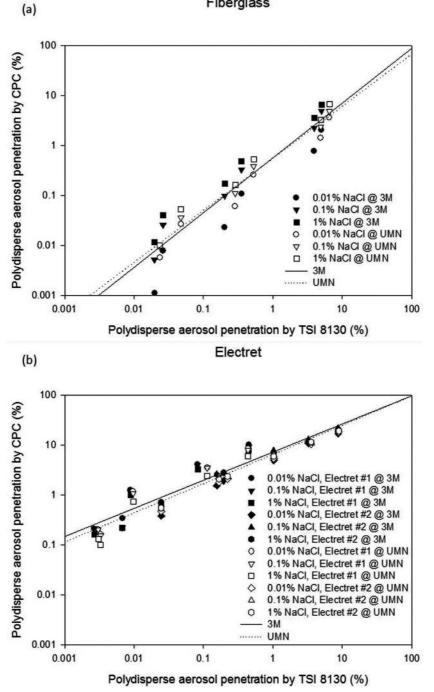


Fig. 7. Correlations between penetration of polydisperse aerosol measured by TSI 8130 and CPC.

penetrations around the MPPS at the same test conditions for both fiberglass and electret filter media. Power-law curve fitting was also used for all the data points for each laboratory. Generally, the data and curves between laboratories were visually very similar. If we plot the overall penetrations from TSI 8130



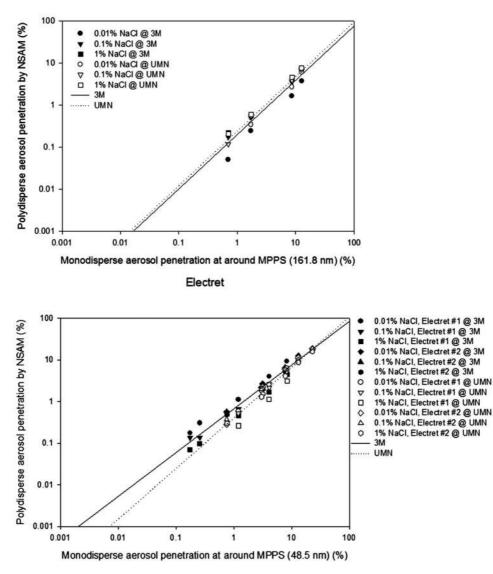


Fig. 8. Correlations between monodisperse aerosol penetrations near MPPS and polydisperse aerosol penetration measured by NSAM.

and CPC as a function of the penetrations near the MPPS, power–law curves were also found for each type of test filter media.

Theoretically, the power–law correlation between the monodisperse and polydisperse aerosol tests depends on the challenge aerosol distribution, the penetration curve of the test filter, and the test conditions. According to the single fiber filter theory, for each filter and challenge aerosol, there is a specific power–law correlation between two filter tests at a certain test condition, and the power curve exponent relates to the ratio of the two single fiber efficiencies, shown in Japuntich (1984) for NaCl and DOP aerosols in photometric filter penetration testing.

In Figs 7 and 8, where the results of three challenge aerosol and two face velocity for two electret and one fiberglass filter media are included, good power–law correlations with $r^2 > 0.9$ were found for all the data points. The monodisperse particle penetration levels measured at the MPPS rank uniformly with the

number-based, lung deposited surface area-based, and mass-based penetration levels, as measured by the CPC, NSAM, and NIOSH standards, respectively. Thus, it is possible to use the penetration results measured by one test method to predict the filtration performance for nanoparticles evaluated by other tests methods based on the established power-law correlations. This method is very useful in respirator penetration testing and has potential application for development of more challenging NIOSH respirator certification protocols. Since most respirators have similar MPPS and penetration curves (Shaffer and Rengasamy, 2009), the mass-based filtration testing equipment used in the NIOSH certification test, which is much easier to maintain and less expensive to purchase, can be used to measure filtration performance for engineered nanoparticles at the MPPS, once the power-law correlation is achieved with extensive tests of respirators at different conditions.

One limitation of the study was that only two electret and one fiberglass filter media were tested using monodisperse and polydisperse aerosol measurements by SMPS, CPC, NSAM and TSI 8130. Further research with other filter media is needed to confirm the results obtained in this study. In addition, the penetration results were obtained using NaCl particles as challenge aerosols. Particles of other materials, e.g. DOP, ambient aerosols or metal oxide particles in the manufacture workplace may be needed to measures the penetration levels of filter media.

CONCLUSIONS

In this study, two laboratories evaluated the performance of different nanoparticle filtration penetration tests for two electret and one fiberglass filter media at two face velocities and three layer thicknesses using NaCl particles. The tests include monodisperse aerosol penetration measurement using a TSI 8160 monodisperse particle test system and polydisperse aerosol measurement using CPC, NSAM, SMPS, and TSI 8130. With proper control of test conditions and calibration procedures, reproducible, comparable filtration data was achieved between two laboratories. Dry air (<20% RH) is needed as makeup air in the test system to prevent deliquescing of the aerosol and possible increases in the NaCl challenge particle dielectric constant. Polydisperse challenge aerosols need to be fully neutralized to obtain accurate penetration measurements.

For a simple nanoparticle penetration test, it is recommended to use a polydisperse aerosol challenge of \sim 50 nm geometric mean diameter, using a CPC or an NSAM as detectors for count or surface area penetration calculations, respectively.

For particle penetration curves, the experimental results of the monodisperse test agreed well with polydisperse SMPS measurements. The MPPSs of electret and fiberglass filter media were ~50 and 160 nm, respectively. For overall penetrations, the CPC and NSAM results of polydisperse aerosols were close to the penetration curves at the corresponding particle sizes. For each filter type, power–law correlations show that the present NIOSH photometric certification test (TSI 8130) may be used to predict penetrations at the MPPS and the CPC and NSAM results with polydisperse aerosols.

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REFERENCES

- Ackley, MW. (1982) Degradation of electrostatic filters at elevated temperature and humidity. 3rd World Filtration Congress; 169–76.
- Bałazy A, Toivola M, Reponen T *et al.* (2006a) Manikin-based performance evaluation of N95 filtering-facepiece respirators challenged with nanoparticles. Ann Occup Hyg; 50: 259–69.
- Bałazy A, Toivola M, Adhikari A *et al.* (2006b) Do N95 respirators provide 95% protection level against airborne viruses, and how adequate are surgical masks? Am J Infect Control; 34: 51–7.
- Benarie M. (1969) Influence of pore structure upon separation efficiency in fiber filters. Staub R Luft; 29: 37–42.
- Brown, RC. (1979) Electrical effects in dust filters. Proceedings of the 2nd World Filtration Congress; 291–301.
- Brown, RC. (1981) Capture of dust particles in filters by linedipole charged fibers. J Aerosol Sci; 12: 349–56.
- Brown RC. (1993) Air filtration: an integrated approach to the theory and applications of fibrous filters. Oxford, UK: Pergamon Press. ISBN: 978-0-08-041274-0.
- Chen CY. (1955) Filtration of aerosols by fibrous media. Chem Re.; 55: 595–623.
- Dai Q, Hu J, Salmeron M. (1997) Adsorption of water on NaCl (100) surfaces: role of atomic steps. J Phys Chem B; 101: 1994–98.
- Davies CN. (1952) The separation of airborne dust and particles. Proc Inst Mech Engrs (London); 1B: 185–213.
- Dick CA, Brown DM, Donaldson K et al. (2003) The role of free radicals in the toxic and inflammatory effects of four different ultrafine particle types. Inhal toxicol; 15: 39–52.

- Duffin R, Tran L, Brown D *et al.* (2007) Proinflammogenic effects of low-toxicity and metal nanoparticles in vivo and in vitro: highlighting the role of particle surface area and surface reactivity. Inhal Toxicol; 19: 849–56.
- Emi H, Kanaoka C, Otani Y, Iiyama T. (1984) Most penetrating particle size in electret fiber filtration. In Liu BYH, Pui DYH, Fissan HJ, editors. Aerosols, science technology and industrial applications of airborne particles. Elsevier Science Publishing Co., Inc. pp. 567–72, ISBN: 0-444-00947-7.
- Eninger RM, Honda T, Reponen T *et al.* (2008a) What does respirator certification tell us about filtration of ultrafine particles? J Occup Environ Hyg; 5: 286–95.
- Eninger RM, Honda T, Adhikari A *et al.* (2008b) Filter performance of N99 and N95 facepiece respirators against viruses and ultrafine particles. Ann Occup Hyg; 52: 385–96.
- Gwinn MR, Vallyathan V. (2006) Nanoparticles: Health effects—pros and cons. Environ Health Perspect; 114: 1818–25.
- Hinds WC. (1999) Aerosol technology: properties, behavior, and measurement of airborne particles, 2nd edition. New York, USA: John Wiley. ISBN 978-0-471-19410-1.
- Huang S-H, Chen C-W, Chang C-P *et al.* (2007) Penetration of 4.5 nm to 10 μm aerosol particles through fibrous filters. J Aerosol Sci; 38: 719–27.
- Japuntich DA. (1984) A particulate respirator certification test apparatus. Journal of the International Society for Respiratory Protection; 2: 249–60.
- Japuntich DA, Franklin LM, Pui DYH *et al.* (2007) A comparison of two nano-sized particle air filtration tests in the diameter range of 10 to 400 nanometers. J Nanopart Res; 9: 93–107.
- Johnson T, Smith S. (1998) Correlation of penetration results between filter testers that use different particle generators and detection methods. TAPPI Nonwovens Conference, 9 March 1998, St. Petersburg, FL.
- Kanaoka C, Emi H, Otani Y *et al.* (1987) Effect of charging state of particles on electret filtration. Aerosol Sci Technol; 7: 1–13.
- Kreyling WG, Semmler M, Möller W. (2006) Health implications of nanoparticles. J Nanopart Res; 8: 543–62.
- Lee KW, Liu BYH. (1980) On the minimum efficiency and most penetrating particle size for fibrous filters. J Poll Control Assoc; 30: 377–81.
- Lore MB, Sambol AR, Japuntich DA *et al.* (2011) Inter-laboratory performance between two nanoparticle air filtration systems using scanning mobility particle analyzers. J Nanopart Res; 13: 1581–91.
- Luna M, Rieutord F, Melman NA *et al.* (1998) Adsorption of water on Alkali Halide surfaces studied by Scanning Polarization Force Microscopy. J Phys Chem A; 102: 6793–800.
- Martin SB Jr, Moyer ES. (2000) Electrostatic respirator filter media: filter efficiency and most penetrating particle size effects. Appl Occup Environ Hyg; 15: 609–17.
- Monteiller C, Tran L, MacNee W *et al.* (2007) The pro-inflammatory effects of low-toxicity low-solubility particles, nanoparticles and fine particles, on epithelial cells in vitro: the role of surface area. Occup Environ Med; 64: 609–15.
- Mostofi R, Wang B, Haghighat F et al. (2010) Performance of mechanical filters and respirators for capturing nanoparticles—limitations and future direction. Industrial Health; 48: 296–304.
- Moyer ES, Bergman MS. (2000) Electrostatic N-95 respirator filter media efficiency degradation resulting from intermittent

sodium chloride aerosol exposure. Appl Occup Environ Hyg; 15: 600-8.

- Myers DL, Arnold BD. (2003) Electret media for HVAC filtration applications. Int Nonwov J; 12: 43–54.
- NIOSH (1995) Approval of Respiratory Protective Devices, Code of Federal Regulations Title 42, Part 84. Available from: http://www.cdcgov/niosh/npptl/part84pdf (accessed 30 September 2011)
- Oberdörster G. (2001) Pulmonary effects of inhaled ultrafine particles. Int Archives Occup and Environ Health; 74: 1–8.
- Oberdörster G, Oberdörster E, Oberdörster J. (2005) Nanotoxicology: an emerging discipline evolving from studies of ultrafine particles. Environ Health Perspect; 113: 823–39.
- Oberdörster G, Stone V, Donaldson K. (2007) Toxicology of nanoparticles: a historical perspective. Nanotoxicology; 1: 2–25.
- Pierce ME. (2006) Comparison of filtration efficiency measurements between TSI Model 8160, TSI Model 3140, and ATI 100P filter test stands. Paper 24. 29th International Society of Nuclear Air Treatment Technologies (ISNATT), 19–21 July 2006, Cincinnati, OH.
- Rengasamy S, Verbofsky R, King WP et al. (2007) Nanoparticle penetration through NIOSH-approved N95 filteringfacepiece respirators. J Int Soc Respir Prot; 24:49–59.
- Rengasamy S, King WP, Eimer BC, Shaffer RE. (2008) Filtration performance of NIOSH-Approved N95 and P100 fltering facepiece respirators against 4 to 30 nanometer-size nanoparticles. J Occup Environ Hyg; 5: 556–64.
- Rengasamy S, Eimer B, Shaffer RE. (2009) Comparison of nanoparticle filtration performance of NIOSH-approved and CE marked filtering-facepiece respirators. Ann Occup Hyg; 53: 117–28.
- Rengasamy S, Miller A, Eimer BC. (2011) Evaluation of the filtration performance of NIOSH–approved N95 filtering facepiece respirators by photometric and number-based test methods. J Occup Environ Hyg; 8: 23–30.
- Rubow K. (1981) Submicron aerosol tiltration characteristics of membrane filters. Ph.D. Thesis, University of MN, pp. 37–8.
- Sager TM, Castranova V. (2009) Surface area of particle administered versus mass in determining the pulmonary toxicity of ultrafine and fine carbon black: comparison to ultrafine titanium dioxide. Particle and Fibre Toxicology; 6: 15.
- Shaffer RE, Rengasamy S. (2009) Respiratory protection against airborne nanoparticles: a review. J Nanopart Res; 11: 1661–72.
- Shin WG, Pui DYH, Fissan H *et al.* (2007) Calibration and numerical simulation of nanoparticle surface area monitor (TSI Model 3550 NSAM). J Nanopart Res; 9: 61–9.
- Stamatakis P, Natalie CA. (1991) Research needs in aerosol processing. Aerosol Sci Technol; 14: 316–21.
- Stanley N, Qi C, Pui DYH. (2010) A new method of filter efficiency evaluation using the nanoparticle surface area monitor (NSAM) for a nanoparticle health relevant filter efficiency measure. Filtration; 10: 40–6.
- Stoeger T, Reinhard C, Takenaka S et al. (2006) Instillation of six different ultrafine carbon particles indicates a surface area threshold dose for acute lung inflammation in mice. Environ Health Perspect; 114: 328–33.
- Tran CL, Buchanan D, Cullen RT et al. (2000) Inhalation of poorly soluble particles II Influence of particle surface area on inflammation and clearance. Inhal Toxicol; 12: 1113–26.
- Waker AJ, Brown RC. (1988) Application of cavity theory to the discharge of electrostatic dust filters by x-rays. Appl Radiat Isot; 39: 677–84.