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Filtration performance down to nano-particles

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FILTECH 2009

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October 13 – 15, 2009

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FILTRATION PERFORMANCE DOWN TO NANOPARTICLES

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ABSTRACT

Man-made nanoparticles escaping into the environment and potentially causing adverse health effects are a serious concern for all interested parties. However, safety issues are currently not fully understood and assessed.

We analyze the nanoparticle removal performance of three different wet-laid fibrous filter media used in both general ventilation and contamination control applications. Media characteristics were evaluated experimentally and their performance measured on a small scale test rig in the 0.1-3.0 μm size range. The data were extrapolated down to 10 nm according to the most recent expressions available for Brownian diffusion. The calculations show that the efficiency at 10 nm is clearly higher than the one at 1000 nm and far higher than at MPPS for the three different media considered here.

In the second part of the paper we analyze the peculiarities of fractional efficiency measurements down to particles with size of few nanometers. Special attention is devoted to the phenomena influencing the test rig design and the measurement procedure. We discuss the capabilities and the cost of the instrumentation currently available on the market for measuring the data needed and for widening the particle size range of the most common standardized current test methods.

KEYWORDS

Brownian Motion, Fibrous Filter, Filter Test, Filtration Performance, Fractional Efficiency, Nanofiltration, Nanoparticles, SMPS

1. Introduction

Nanoaerosols are made of engineered nanomaterials, nanoparticles and nanostructures having one or more dimensions of the order of 100 nm or less. There is much interest in nanosized materials because, at the nano-scale, their physical properties are quite different from the properties of the bulk material from which they are made. However, it has been established for many years that exposure to particles, including nanoparticles, can cause illness in individuals or exposed populations.

The potential risks to health from inhalation of nanoparticles are due to several factors.

- Nanoparticles can reach parts of biological systems which are not normally accessible by larger particles, e.g. the possibility of passing directly from the lungs into the blood stream and to all of the organs, or even through deposition in the nose, directly to the brain (translocation).
- Nanoparticles have, for particle collections with equal masses, much higher surface area than larger particles. If surface area is linked to toxicity this implies potentially higher toxic effects.

- The reduction in size has been shown to relate to increased solubility for some nanomaterials. This effect might lead to increased bioavailability of materials which are considered to be insoluble at larger particle sizes.
- Since nanomaterials and nanoparticles have new and different properties from larger particles of the same material, altered chemical and/or physical properties might be expected to be accompanied by altered biological properties, some of which could imply increased toxicity.
- Some high aspect ratio nanoparticles can be inhaled and enter the alveolar region of the lung and are not easily removed. Their physical dimensions inhibit their removal by lung clearance mechanisms and they do not dissolve in the lung fluids. Hence they remain in the lung for a long period of time, causing inflammation and ultimately disease.

The above issues indicate that more needs to be done to assess the potential risks associated with nanomaterials. In the meantime a cautious approach should be taken in their handling and disposal. The risk depends on the dose of the particles in the organ where disease can occur, and the toxicity of nanoparticles. Dose is hard to assess directly, but can be obtained from the exposure to nanoparticles, i.e. the combination of particle concentration in the air which a person breathes in and the duration of the exposure.

If there are no nanoparticles in the air, no dose will accumulate and, despite the potential toxicity of the particles, there will be no risk to health. Therefore in many working atmospheres the preferred method to control risks is a strong effort to mitigate, manage or reduce exposure. Fibrous air filters can be very effective in removing nanoparticles from air streams and may play an essential role in this strategy. However, no standardized test method for measuring the efficiency of filters in removing particles below the 100 nm size is currently available. Test methods for HEPA and ULPA filters measure the removal efficiency corresponding to the most penetrating particle size (MPPS) or close to it, i.e. usually between 100 nm and 200 nm. The most widely used test methods for general ventilation filters supply no data in the defined nanoparticle range, since they test only down to 200 nm (EN779:2002) and to 300 nm (ANSI/ASHRAE 52.2-2007). The absence of references supplying the measured performance of air cleaning devices in removing nanoparticles makes it difficult to draft any regulation for handling nanoparticles safely.

Even if current research shows that the theory of particle removal by fibrous air filters is valid down to 3 nm, it is reasonable to expect that some factual evidence will be requested by regulatory authorities to trust air filters as a valid mean to minimize exposure risks.

The present paper aims at answering the basic question: should the scope of standardized test methods be widened to include the nanometer size range, or is the information obtained with already available test methods enough for nanoparticle exposure assessment?

We divide this problem in two parts. The first one describes the experimental characterization and performance of three wet-laid fiber glass media and the calculated extrapolation of their efficiencies down to 10 nm according to the most recent theory. The second part describes the peculiarities of efficiency measurements down to nanoparticle size, the phenomena influencing test rig design and measurement procedures, and the costs and capabilities of the instrumentation currently available on the market for measuring the data needed.

2. Measurement of filter media properties

Three types (F6, F8 and H13) of media samples were cut from rolls supplied by the manufacturer, using randomized locations down the length and width of the roll. Variances of measured parameters were found to be small, but random sample selection avoids any systematic parameter biases.

More than 20 scanning-electron microscope (SEM) images were taken of each of the three media types. Approximately 1000x magnification allowed the least diameters to be seen and measured, while preserving enough area of the filter media to allow representative sampling. Again, a randomization technique was used to eliminate bias and simultaneously weight the diameters by the length of each diameter interval present. Parallel lines were scratched across the SEM photographs at randomly-located positions. Wherever these lines intersected a fiber, the width of the fiber was measured in the direction normal to the fiber axis. A special scale was prepared to allow rapid sorting of the fiber diameters at each line/fiber intersection. Several hundred intersections (hence fiber diameters) were included from each media type.

Table 1 - Basic parameters of media tested and values obtained from calculations

Sample	Units	Sample Data		
		F6	F8	H13
Rated velocity (v_r)	m/s	0.0617	0.0617	0.0231
Geometric mean diam. (D_g)	μm	4.110	1.561	0.775
Geometric std. dev. (σ_g)	-	1.921	2.141	2.198
Fractional solids (α)	-	0.076	0.081	0.092
Pressure drop (Δp)	Pa	10	42	121
Effective fiber diameter (d_f)	μm	8.179	3.618	1.512
Measured efficiency at 100 nm	%	10.8	44.9	99.3
Measured efficiency at 1000 nm	%	32.0	93.3	99.5
Calculated efficiency at 10 nm	%	81.8	99.2	100
Calculated efficiency at 100 nm	%	23.9	55.4	99.6
Calculated efficiency at 1000 nm	%	20.2	86.9	99.99986

The fiber-size data were fitted to the log-normal distribution to obtain the geometric mean diameter and standard deviation for the fibers in each media.

The (volume) fractional solids in fibrous media is:

$$\alpha = \frac{M_m \left[\frac{\eta}{\rho_{fiber}} + \frac{1-\eta}{\rho_{binder}} \right]}{L} \quad (1)$$

Fibers of glassfiber filter media have a melting point above the ignition temperature of organic binders. Baking at 500 °C burns the binder out of the media. Weighing media samples before and after baking thus allows the calculation of fiber mass fraction (η). Values of 2450 kg/m³ for fiber density and 1000 kg/m³ for binder density were thought reasonable. The thickness of the filter medium was measured, along with the compression function (the relation between the thickness of the medium and the pressure drop across it) using the technique described in Rivers (2000).

The resistance-vs.-velocity characteristics of flat sheets of media were measured in a test duct which exposed an area of media 300 mm by 300 mm. Complete filter cells from these grades of filter media contain enough media area to reduce the average media velocity below 0.1 m/s and hence the expected resistance of a single sheet of medium to very low, difficult-to-measure levels. Accuracy of resistance measurement

was improved by measuring the resistance of a 10-sheet stack. In the range of interest, resistance is essentially proportional to velocity. Fractional efficiency curves for the three types of media were measured in the same test rig using DEHS synthetic aerosol and an optical particle spectrometer able to provide data in the 100-7500 nm size range.

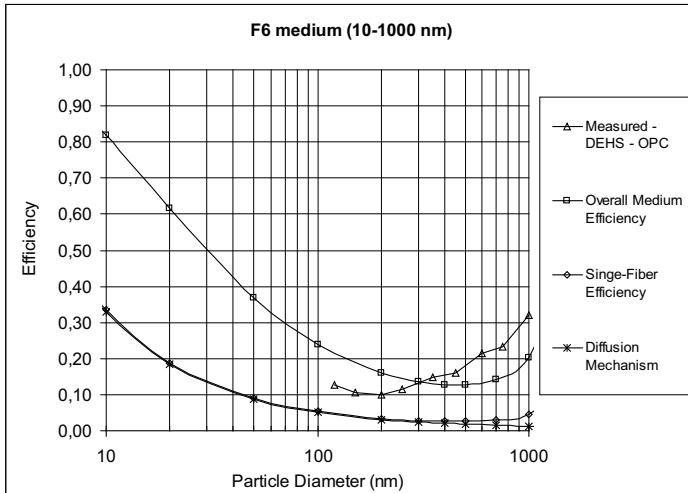


Figure 1 – Fractional efficiency of F6 medium

The single fiber efficiencies due to the deposition mechanisms by which an aerosol particle can be deposited onto a fiber in a filter were computed using the expressions supplied by Hinds (1999). We considered mechanical collection mechanisms only. The common expression for the diffusion mechanism:

$$E_D = 2 \cdot \frac{1}{\sqrt[3]{Pe^2}} = 2 \cdot \left(\frac{d_f \cdot U_0}{k \cdot T \cdot C_c} \right)^{-\frac{2}{3}} \quad (2)$$

was replaced by the more recent expression proposed by Wang (2007):

$$E_D = 0.84 \cdot Pe^{-0.43} \quad (3)$$

Expression (3) yields a lower efficiency due to the diffusion mechanism than expression (2).

Diffusion is the only important mechanism for particles below 0.2 μm , but is of decreasing importance for particles above that size. It is well known that the competing deposition mechanisms are most effective in different size ranges. Hence all filters have a particle size that gives minimum efficiency, usually in the range from 50 to 500 nm. The single fiber approach takes the distribution of the fiber sizes into account indirectly. Since the flow field and collection efficiency associated with each fiber size are influenced by the presence of fibers of other sizes, as a practical means, the effective fiber diameter d_f , based on pressure drop measurements, is used as an approximation for these calculations. Moreover the fibers may be clumped together and the medium may not be uniform. The use of the effective fiber diameter avoids this problem but it turns out to be very different from the geometric

mean diameter of the log-normal distribution describing the actual physical appearance of the medium itself. The results of the measurements and of the calculations are shown in Figures 1, 2 and 3. Media F6 and F8 are meant for use in general-ventilation applications, while H13 is intended for use in contamination control applications.

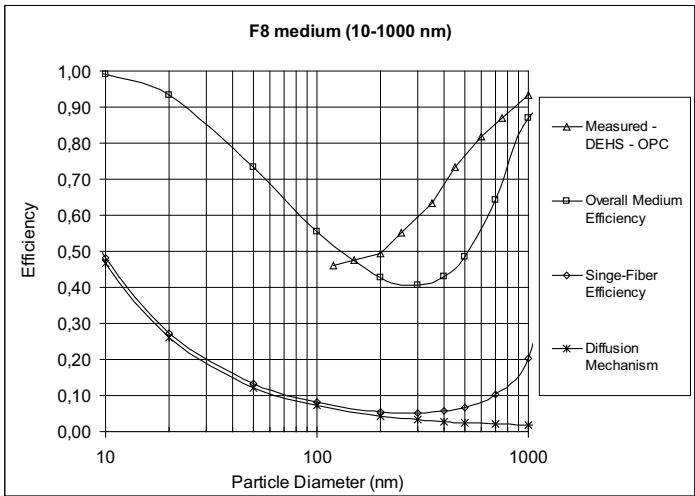


Figure 2 - Fractional efficiency of F8 medium

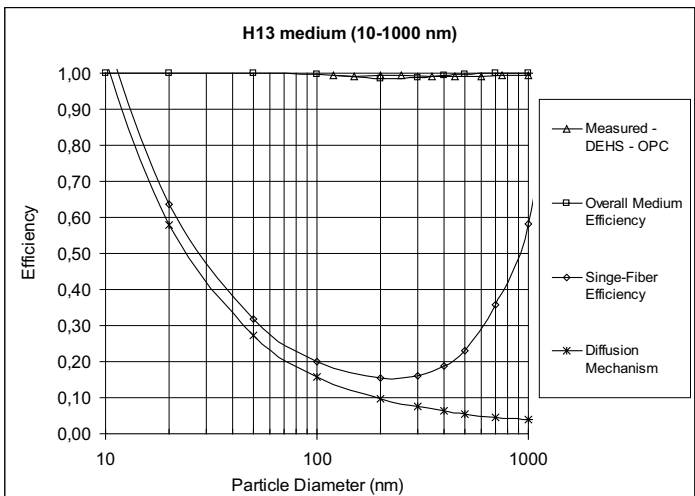


Figure 3 - Fractional efficiency of H13 medium

3. Testing filters at nanometer particle diameters

Current filter testing and standardization practices rely on the higher filter efficiencies predicted at nano-sizes to make claims that filters perform better than specified filter class at nanoparticle sizes. The work reported in this paper has confirmed the predicted higher efficiency at nano-sizes for several filter media. However, building

commercial testing systems for filter performance measurements on nano-size particles poses new challenges. Building commercial test systems involves scaling up the aerosol generating and measurement systems discussed above to handle air flow rates about $1 \text{ m}^3/\text{s}$. The general requirements are discussed below.

Particle measurements: Any filter efficiency test system must have the ability to measure concentrations of particles within specific particle size ranges over the entire size range of interest. High quality data requires that the measurement devices have nearly the same response over the range of interest. Even for current measurement systems, measurements over 2 orders of magnitude in particle size and concentration can become a challenge for commercially available measurement devices. When one considers measurements of particles from 5 nm to 10,000 nm, the challenge may become nearly impossible. The use of more than one measurement device may be required to provide data over the entire size range.

Particle size specific concentrations can be measured either by using particle spectrometers or by size classifiers and particle detectors. Current filter test systems mostly use optical particle spectrometers (OPC). These devices provide particle counts in several particle size intervals. Several commercial devices allow users to select the size intervals. Alternately, particles can be classified according to their size and the classified particles counted by detectors. Electrostatic classification of particles and counting by condensation particle counters (CPC) or electrometers are the most common combinations used. In this case, particles are classified according to their electric mobility, which is directly related to particle size. Classification by diffusion is also a technique for particle size specific measurements, although not commonly used. Where one requires measurement for sizes for which calibrated PSL particles are available, it is also common to use these particles without classification. The advantages of the OPCs are their relatively low cost and their ease of use. Further, most standards relevant to particle contamination and filtration are based on OPC measurements, making data from these devices readily comparable. Their main disadvantage is their detection limitations at smaller sizes. Although the size range of many commercial counters extend down to 100 nm, the response of optical scattering devices drops off significantly for particles under 150 nm. Thus it will be impractical to use these devices for determining filter performance for particles in the sub 100 nm sizes.

Electrostatic classification is the most common method used for particle research and calibration, especially for sub-micrometer particles. Since the size classification can be derived for a given geometry of the instrument, it is also referred to as a reference method. Since the classification is according to the electric mobility of a particle, larger particles carrying multiple units of charge will have double the mobility and, hence, could be classified with singly charged particles with half their size. However, nearly all sub micron particles are singly charged and will be classified according to their size. Commercial electrostatic classifiers account for the effect of multiple charge based on the charge distribution on the aerosol classified. The most common detector for the classified particles is the CPC. Since it can detect nearly all the particles as small as a few nanometers, it is also often called a reference counter. This combination of electrostatic classification and CPC is currently commercially available and is used extensively in nanoparticle research. An alternate means of detecting classified particles is to use an electrometer to collect the particles and measuring the current. Modern electrometers are often more compact and robust than CPCs.

In diffusion classification, penetration of particles through a series of diffusion tubes or screens is measured using a detector, such as the CPC, and the size specific

concentration determined from the data. Although diffusion classification does not depend on the charge on the particle, since particles of all sizes will penetrate to different extent through the diffusion element, the classified aerosol is somewhat poly-disperse. Hence these measurements require extensive data reduction to generate particle size specific concentration. Perhaps for this reason they are not in common use outside of particle research.

Further, as discussed earlier, the efficiency of even lower grades of media can be quite high for nano-size particles. Hence, the downstream counts for these particle sizes will be quite low for many filters, requiring long sample times to obtain statistically valid counts with any of the devices discussed above. Since time is invaluable in product manufacturing, instruments with large sample flows may be needed for nanoparticle measurements.

Particle Losses: Loss of particles during sampling and transport is always a concern since such losses can introduce unknown errors in the data. In current filter testing practice, the focus has been primarily on large particles, typically greater than 2000 nm. Since large particles are lost in sharp bends in tubing, valves, and constrictions, current testing system designs minimize these elements in sampling lines. Where practical, sample lines upstream and downstream of filters are made equal in length in an attempt to equalize the losses, if any, and minimize errors in the computation of filter efficiency. Smaller particles are considered to behave like gases and their losses are generally ignored. However, the losses due to Brownian Diffusion increase significantly for nano-sizes. Much like the higher capture efficiency for nanoparticles in filters, nanoparticles are also more readily "captured" in the sample lines. Hence nanoparticle sampling will require more attention to making upstream and downstream sample lines exactly equal. In addition, a two-stage sampling may be needed if the instrument sample flow rate is low, as is common in many CPCs. In this case, a first stage sample is taken at a high sample flow rate, minimizing the residence time and hence the losses in the sample lines. Then the detection instrument with its much smaller flow rate can sample from this larger first stage sample using very short sampling tubes.

Challenge Particles: The common practice of using poly-dispersed aerosols with particle spectrometers or classifiers will be adequate, in principle, for measurements at nano-sizes. However, as noted above, because of the higher filter efficiency for nanoparticles, poly-disperse aerosols with large concentrations in the nano-sizes are preferred. Vapor condensation generators are best suited for this purpose and are commercially available. Alternately, atomizing solutions of the aerosol material and flashing the solvent will yield a large concentration of poly-dispersed nanoparticles of the solute material. This technique is used in the ASHRAE 52.2 standard for generating nano-size KCl aerosol for neutralizing electrostatic charge in filters.

Other factors: In general, all other good testing practices described in many of the national standards are readily applicable for testing filters down to nano-sizes. These include prescription for system validation, dilution of upstream concentrations, and aerosol neutralization as well as general system configuration.

In summary, the established practices prescribed by the prevailing standards offer a good starting point for testing filter efficiency for nanoparticles. Additional precautions in sampling and measurements are required to obtain valid results. The main upgrade for these systems will be the particle measurement devices, and the design of the sample lines to minimize losses. Further, the need for statistically valid counts may require longer and more expensive testing times. Overall, it is our opinion, based on current market for these particle instruments, that filter efficiency measurements for nanoparticles will add over 30% to the cost of the system.

Conclusions

Our analysis suggests that the additional cost for extending the measuring range down to a few nanometers is not fully justified by the further data obtainable in this way. However, the calculated efficiency depends on many parameters and the accurate characterization of filter media is rather difficult and time consuming. At the same time the technology for evaluating the performance of air filters down to a few nanometers is available on the market and reasonably priced. Interested parties may be willing to pursue efficiency measurements down to a few nanometers, following the nature of air filtration which is mainly experimental.

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