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Evaluation of a novel personal nanoparticle sampler†

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This work investigated the performance in terms of collection efficiency and aspiration efficiency of a personal sampler capable of collecting ultrafine particles (nanoparticles) in the occupational environment. This sampler consists of a cyclone for respirable particle classification, micro-orifice impactor stages with an acceleration nozzle to achieve nanoparticle classification and a backup filter to collect nanoparticles. Collection efficiencies of the cyclone and impactor stages were determined using monodisperse polystyrene latex and silver particles, respectively. Calibration of the cyclone and impactor stages showed 50% cut-off diameters of 3.95 μm and 94.7 nm meeting the design requirements. Aspiration efficiencies of the sampler were tested in a wind tunnel with wind speeds of 0.5, 1.0, and 1.5 m s^{-1}. The test samplers were mounted on a full size mannequin with three orientations toward the wind direction (0°, 90°, and 180°). Monodisperse oleic acid aerosols tagged with sodium fluorescein in the size range of 2 to 10 μm were used in the test. For particles smaller than 2 μm, the fluorescent polystyrene latex particles were generated by using nebulizers. For comparison of the aspiration efficiency, a NIOSH two-stage personal bioaerosol sampler was also tested. Results showed that the orientation-averaged aspiration efficiency for both samplers was close to the inhalable fraction curve. However, the direction of wind strongly affected the aspiration efficiency. The results also showed that the aspiration efficiency was not affected by the ratio of free-stream velocity to the velocity through the sampler orifice. Our evaluation showed that the current design of the personal sampler met the designed criteria for collecting nanoparticles ≤100 nm in occupational environments.

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Environmental impact

Concerns about exposure to nanoparticles and the associated health effects have increased because of rapidly expanding applications and production of nanomaterials. Personal samplers capable of collecting nanoparticles are needed to estimate the exposure to nanoparticles in the occupational and ambient environments. This article describes the performance evaluation of a new personal sampler for collecting nanoparticles. Our results found out that the performance of this sampler met the design requirement and was a novel measurement tool to assess the potential exposure to nanoparticles in the environment.

Introduction

Nanoparticles (or ultrafine particles) are those materials with at least one dimension ≤100 nm and have very large surface areas with increased chemical and biological reactivity compared to the bulk material. Nanoparticles can be released in the ambient environment from high-temperature sources, including industrial processes and automobile combustion.[1] Recently, the advances of nanotechnology have produced a diverse range of nanomaterials such as metal oxides, fullerenes, nanotubes, nanowires, and quantum dots.[2] These materials are called "engineered nanoparticles."

The application of engineered nanomaterials has grown rapidly into all sectors of modern society. The explosive development of nanomaterials has raised concerns about potential exposure and associated health effects.[1–4] Several recent studies of exposure assessment in nanomaterial production facilities have shown that the mass concentration of nanomaterials was generally higher than that of the ambient environment. The chemical composition and size distribution of engineered nanoparticles were more defined and specific for each production facility.[4–6]

A potentially important human exposure route for nanoparticles includes exposure *via* inhalation followed by skin and the gastrointestinal (GI) tract.[7] Inhaled nanoparticles deposit with high efficiency in all regions of the respiratory tract by the

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diffusion process.[8–11] Because of the greater surface areas, inhaled nanoparticles showed greater inflammatory responses on a given mass than did larger particles with the same chemical composition.[7] There are many epidemiological and controlled clinical studies of adverse health effects on exposure to ambient ultrafine particles.[7] This has generated great concern for the potential health effects of exposure to these nanoparticles. Because nanomaterials are small in size with large surface areas, many studies have shown that the biological effects of nanomaterials are greater than those of the bulk material of the same chemical composition. In 2005, the National Institute for Occupational Safety and Health (NIOSH) recommended exposure limits of 1.5 mg m^{-3} for fine TiO_2 and 0.1 mg m^{-3} for ultrafine TiO_2 as time-weighted average (TWA) concentrations for up to 10 h day^{-1} during a 40 h work week.

Currently there are no suitable personal samplers capable of assessing the exposure level of ultrafine particles or nanoparticles, but there are several instruments available for monitoring nanoparticle concentrations. A nanoparticle surface area monitor (NSAM) uses diffusion charging of particles followed by electrometer detection to determine the particle concentration.[12] Another frequently used direct-reading instrument is the scanning mobility particle spectrometer (SMPS) that can measure concentration and size distribution of submicron aerosols, including nanosized particles.[13] Other commercially available monitors such as NanoTracer[14] and DiSCmini[15] can also measure nanoparticles in a very short time period. However, none of these instruments collects particles for mass concentration determination.

In order to take samples and determine the mass concentration of nanoparticles, the sampling device needs to separate particles ≤100 nm from the aerosol stream and collect this fraction. Micro-orifice cascade impactors (including the MOUDI and nano-MOUDI) are available to aerodynamically separate particles down to nanoparticle size fractions and collect these fractions, but these samplers are too bulky to be used for personal sampling.

The existing monitoring devices for nanoparticles are either direct-reading devices that do not provide samples for analysis or micro-orifice impactors that are not suitable for assessing personal exposure. There is a need to develop a lightweight, small-volume, personal sampling device that can classify the nanoparticle fraction (≤100 nm) from the aerosol stream and collect this fraction for various analyses.

Recently, a new personal sampler capable of collecting ultrafine particles (nanoparticles) in the occupational and ambient environments has been developed. This sampler consists of a cyclone for respirable particle classification, micro-orifice impactor stages with an acceleration nozzle to achieve nanoparticle classification and a backup filter to collect nanoparticles. The design, calibration of individual stages using sodium chloride aerosol, and effects of loading on collection efficiency have been reported.[16] The initial results indicated that this sampler is capable of classifying the nanoparticle fraction (≤100 nm) from the aerosol stream and collecting this fraction for gravimetric, chemical, and other analyses. This device can be used to accurately assess personal exposure to nanoparticles in terms of mass concentration and physicochemical characteristics. The sampler also has potential applications in the general environment for determining exposures among non-occupationally exposed individuals.

To evaluate a personal sampler, the aspiration efficiency is one of the main criteria.[17–19] Many studies were carried out for the regular personal samplers, especially the Institute of Occupational Medicine (IOM) personal sampler.[20–28] The IOM sampler is used to estimate the personal exposure of workers to airborne micro-sized particles in the workplace. It is designed to collect inhalable particle matter (IPM), the fraction of ambient airborne particles that are deposited in the respiratory tract. The aspiration efficiency of the IOM sampler was tested at different flow rates in different studies.[21–27] For a newly designed personal sampler, the aspiration efficiency must be tested.

The overall objective of this study is to evaluate this personal sampler in terms of stage collection characteristics and aspiration efficiency to ascertain its suitability for collecting nanoparticles. The collection efficiencies in cyclone and impactor stages were determined using silver particles whereas the aspiration efficiencies were measured in an aerosol wind tuned with a wind speed from 0.5 to 1.5 m s^{-1}.

Methods

The personal nanoparticle sampler

A novel personal nanoparticle sampler (PENS) capable of collecting the ultrafine particles (nanoparticles) in the occupational environment was developed as described previously.[16] This sampler consists of a cyclone for respirable particle classification, micro-orifice impactor stages with an acceleration nozzle to achieve nanoparticle classification and a backup filter to collect nanoparticles (Fig. 1A). By applying high and localized

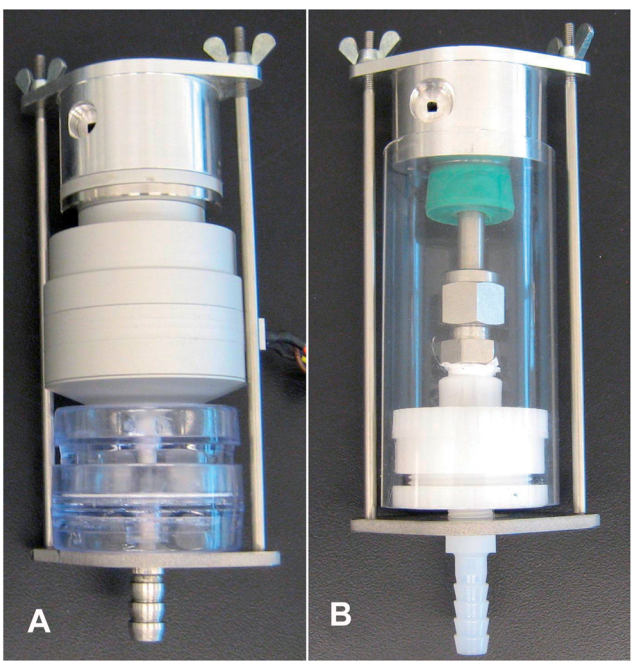

Fig. 1 The PENS sampler before (A)[16] and after (B) modification.

velocity in the nozzle, diffusion deposition of nanoparticles can be minimized in the classifying process, and nanoparticles can be collected in the downstream backup filter. The cutoff diameters are 4 μm and 100 nm for the cyclone and the impactor stages, respectively. To achieve a uniform particle deposition and avoid solid particle bounce on the substrate, a stepper motor was used to rotate the impaction substrate at 1 rpm. The sampling flow rate of the PENS was designed to be 2 L min^{-1}, which is limited by the pressure drop that can be provided by the current personal sampling pump.[16]

The test sampler was a modified version with the inlet configuration changed from that previously reported.[16] This modification was made to improve the aspiration efficiency. PENS was designed as the three main parts to achieve collection of nanoparticles, which also makes a complicated inside structure. However, for an aspiration efficiency test, the inside design does not affect the results. Only the sampling inlet direction and the outside geometry are factors that can change the aspiration efficiency. When the PENS was tested for the aspiration efficiency, particles that deposit inside the PENS cannot be completely washed out because of the complicated inside design. For this reason, the sampler was modified to simplify the inside dimension and keep similar outside geometry as shown in Fig. 1B. The aspiration efficiency will be the particle concentration collected in the modified sampler divided by the particle concentration collected on the reference filters.

NIOSH personal sampler

A NIOSH-designed personal sampler used for collecting bioaerosols[29] consists of two cyclones and a backup filter. The 50% cut-off aerodynamic diameters for the two cyclones are 2.6 μm and 1.6 μm at 2.1 L min^{-1} sampling flow rate. The NIOSH sampler was tested for aspiration efficiency in this study in comparison with the PENS sampler.

Experimental set-ups

Three separate experiments were conducted. The cyclone and impactor were disassembled as individual samplers in order to test their cutpoint diameters. For the aspiration efficiency test, the PENS was modified into the same outer shape with a simplified inside geometry. Details of each experiment will be discussed.

Setup for collection efficiency of the respirable cyclone. Monodisperse sodium-fluorescein-tagged oleic acid particles with a particle size range of 2.5–9 μm were generated by using a vibrating orifice monodisperse aerosol generator (VOAG, Model 3050, TSI Inc., St. Paul MN). An aerodynamic particle sizer (TSI Inc.) was used to monitor and adjust the aerosol size distribution. Only aerosols with a geometric standard deviation (GSD) <1.2 were considered as monodispersed. For 2 μm particles generated by the VOAG, the GSD was >1.2, which is not considered monodispersible. The polystyrene latex (PSL) fluorescent aerosols (Duke Scientific, Palo Alto, CA) were generated by using a medical nebulizer (Hospitak, Lindenhurst, NY) only for this particle size. Fig. 2 shows the experimental setup for the test. Monodisperse aerosols were generated and neutralized by using a Kr-85 source before entering the chamber. When the nebulizer was used as an aerosol generator, the vibrating orifice monodisperse aerosol generator was used as an air dilutor to provide enough air to the chamber.

Setup for collection efficiency of the micro-orifice impactor. The experimental setup for the deposition efficiency test of the micro-orifice impactor is shown in the ESI (Fig. S1†). Silver aerosols were generated using the condensation aerosol generation method.[30] Silver wool was placed in a quartz boat inside a quartz tube of the tube furnace. The furnace was operated at 700–900 °C. Filtered dry air at a flow rate of 1 L min^{-1} carried the vaporized materials into a glass condensation chamber, where the vapor was condensed into nanosize particles. The particles were then introduced into an electrostatic classifier to classify to single charged monodisperse aerosols. An SMPS (TSI) was used to verify the relations between the particle size and voltage of the classifier. The monodisperse particles were then delivered into a mixing chamber where dilute air was provided. The micro-orifice impactor sampled aerosols from the mixing chamber. Aerosol concentrations at the inlet and outlet ports of the micro-orifice impactor were determined by using a condensation particle counter (CPC, TSI). The deposition efficiency of the impactor can be calculated by the ratio of the aerosol concentration in the outlet and inlet of the impactor. The particle size range was 50 to 300 nm.

Setup for aspiration efficiency. All measurements were carried out in a wind tunnel inside of a 4.3 × 3.7 × 3.6 m test room. The wind tunnel consists of an 11 m-long circular duct with a diameter of 1.83 m; a stationary air blender (Blender Products, Inc. Denver, CO), which creates mixing; and a flow straightener, test chamber, and blower (Fig. S2,† ESI). Incoming air filtered by using a high-efficiency particulate air (HEPA) filter is drawn from the specially designed test room into the open-loop flow and exhausted to the same room. The blower used for the wind tunnel (IAP, Inc., Phillips, WI) has a capacity of 1100 m^3 min^{-1} at 1.25 kPa static pressure. The wind velocity in the wind tunnel can be adjusted from 0.5 to 8.0 m s^{-1} by changing the speed of the blower motor. The wind tunnel was

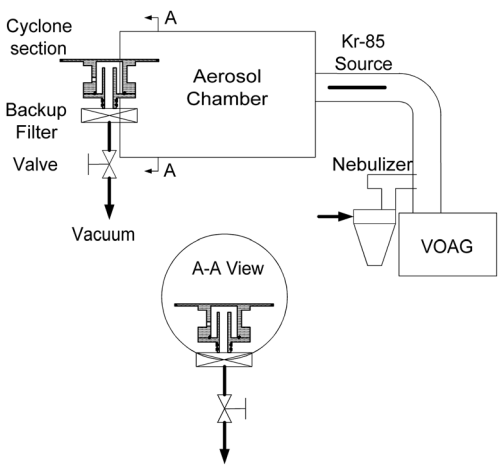

Fig. 2 Schematic diagram of the experimental setup for the cyclone part of the PENS.

calibrated according to U.S. Environmental Protection Agency (EPA) and American National Standards Institute (ANSI) standards. The coefficient of variation (COV) of the wind speeds measured in the middle two-thirds of the test section was found to be less than 5% for all of those wind speeds within the range specified by the EPA and ANSI N13.1 (*i.e.*, 10% over the middle two-thirds of the cross-sectional area). The uniformity of the aerosol concentration in the test section was measured using a 10 μm test aerosol. The COVs of the aerosol concentration were 7.5, 9.1, and 7.5% for wind speeds of 0.56, 2.2, and 6.6 m s^{-1} respectively. The calibration details were described previously by Cheng *et al.*[31]

Test particles. The vibrating orifice monodisperse aerosol generator (VOAG) was used to generate monodisperse, sodium-fluorescein-tagged oleic acid aerosols with the size of 3 to 10 um. For particles smaller than 3 μm, four Hospitak nebulizers were used to generate PSL particles. The VOAG or nebulizers were placed immediately outside of the wind tunnel entrance. The air with generated particles traveled through the length of the wind tunnel and the flow straightener to dampen the turbulence before entering the test chamber. Particles in the range of 0.7 to 10 μm were used in the study. The test aerosol size was determined by using an aerodynamic particle sizer (APS; Model 3310A, TSI Inc., St Paul, MN).

Sampler location and sampling conditions. Three modified PENS and three NIOSH samplers were mounted on a full-size mannequin (170 cm) at a height of 150 cm. One of each type was mounted at the chest of the mannequin, one on the back, and one at a side of mannequin (Fig. 3). Cellulose filters (Type 41, Whatman, Inc., Florham Park, NJ) were used to collect particles passing through the PENS and NIOSH samplers. The samplers were operated at a flow rate of 2.0 L min^{-1} with wind speeds of 0.5, 1.0, and 1.5 m s^{-1}.

Sample and statistical analysis

After each test, particles deposited in the samples were rinsed out by using a solution consisting of 50% isopropyl and 50% distilled water for oleic acid particles and with 100% ethyl acetate for PSL particles. All filters were also placed in the solutions for 24 hours. The relative concentrations of fluorescent tracers in the solutions were measured with a fluorometer (Model 450, Sequoia-Turner Corp. Mountain View, CA). One drop (35–40 μL) of 1 N NaOH was applied into each glass tube, which contains around 6 mL of sample to stabilize the fluorescence. The relative concentration of the fluorescent tracer in the solution was calculated by taking account of the sampling flow rate, sampling time, and dilution factor. The deposition efficiency of the samplers was obtained by dividing the relative concentration of the fluorescent tracer in the device by the relative concentration of the fluorescent tracer in the device and filter. Each data point was an average value of triplicate tests and all error bars in the figure were standard deviations of the triplicates.

The inhalable mass equation for occupational sampling criteria can be expressed in the following equation:[18]

$$\mathrm{IM} = [1 - F(x)], \tag{1}$$

where $F(x) = \int_{-\infty}^{x} \frac{dy}{\sqrt{2\pi}} \exp(-y^2/2)$ is a function of the aerodynamic diameter d_{ae}

$$y = \frac{\ln(d_{ae}/d_{aeo})}{\ln(\sigma_g)}; d_{aeo}\ (\text{at } 50\ \%\ \text{penetration}) = 10\ \mu\text{m};\ \text{and}\ \sigma_g = 1.5. \tag{2}$$

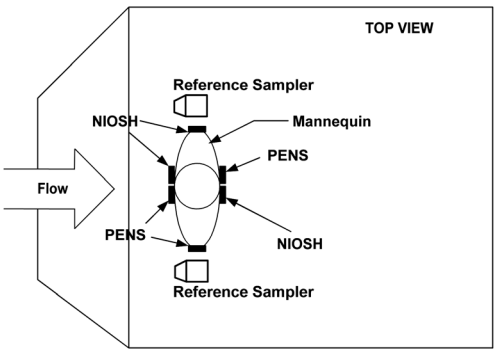

Fig. 3 Locations of the mannequin, personal samplers, and reference samplers in the test chamber of the wind tunnel.

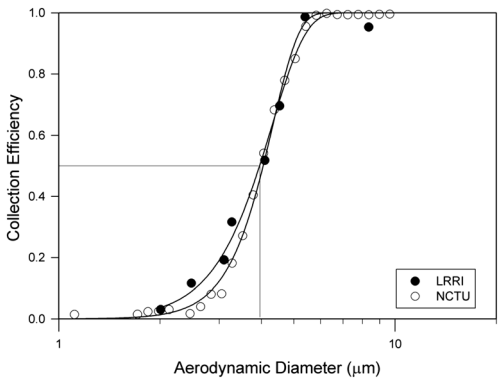

Fig. 4 The collection efficiencies for the cyclone part of the PENS.

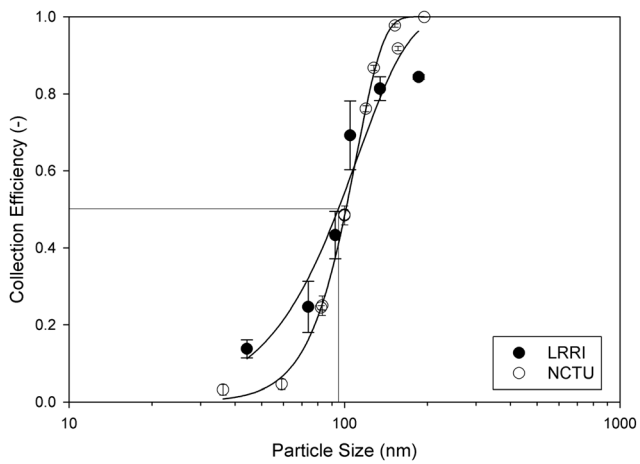

Fig. 5 The collection efficiencies for the impactor part of the PENS.

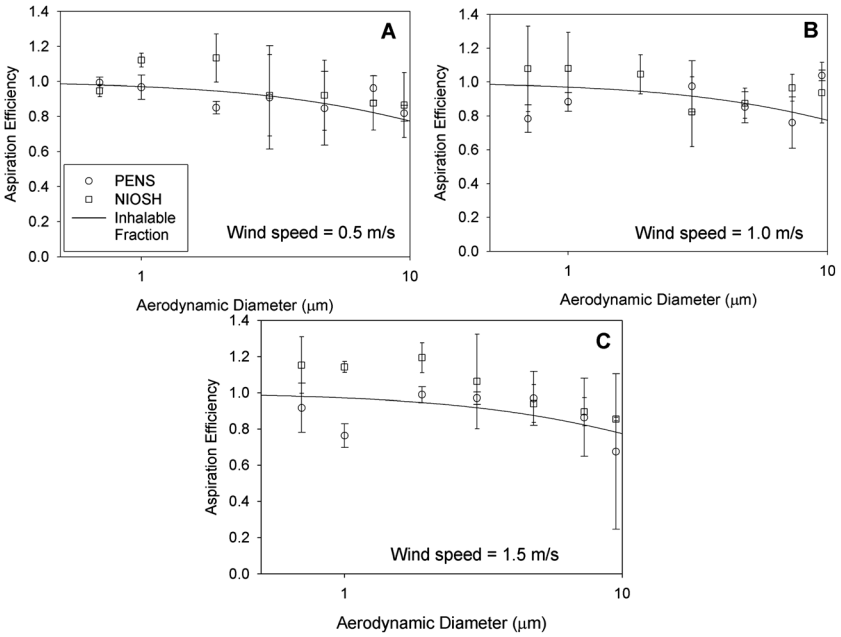

Fig. 6 Orientation-averaged aspiration efficiencies for PENS and NIOSH samplers with a comparison of the inhalable fraction curve at the wind speeds of 0.5 (A), 1.0 (B), and 1.5 (C) m s^{-1}.

The root-mean-square deviation (RMSD) was used for the results in comparing to the inhalable fraction (IF) equation:

$$\mathrm{RMSD} = \sqrt{\frac{\sum_{1}^{n} (Y_i - \mathrm{IF}_i)^2}{n}} \quad (3)$$

where Y_i is the observed value, IF_i is the modelled value, and n is the number of data points. The value of RMSD should be between 0 and 1. All data points are on the equation curve if the RMSD is zero. The smaller the RMSD value, the closer the data point is to the curve.

Results

Cyclone collection efficiency

The cyclone was initially tested at National Chiao Tung University (NCTU), Taiwan, with different methods and particles. Fig. 4 shows the comparison of collection efficiency curves

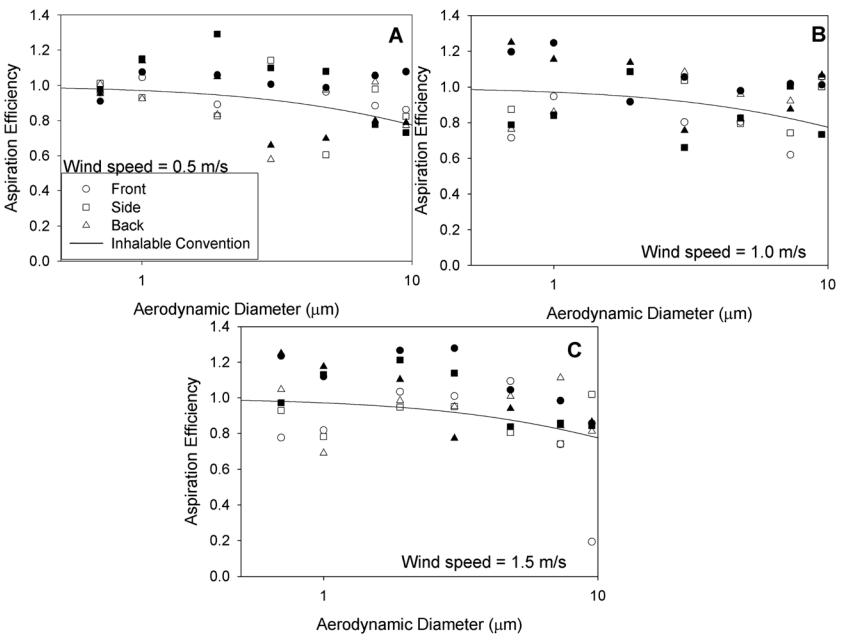

Fig. 7 The aspiration efficiency as a function of particle size for different orientations at the wind speeds of 0.5 (A), 1.0 (B), and 1.5 (C) m s^{-1}. Empty symbols = PENS; blocked symbols = NIOSH.

Table 1 The RMSDs for all wind speeds and wind directions

Wind speed ($m\ s^{-1}$)	0.5		1.0		1.5		0.54
Sampler	PENS	NIOSH	PENS	NIOSH	PENS	NIOSH	IOM[a]
0°	0.18	0.38	0.57	0.39	0.44	0.43	0.30
90°	0.55	0.34	0.32	0.48	0.33	0.28	0.22
180°	0.68	0.46	0.40	0.41	0.44	0.32	0.25
Avg[b]	0.17	0.21	0.34	0.27	0.31	0.29	0.26

[a] The flow rate of the IOM is 2 L min^{-1}. [b] Orientation-averaged.

from the NCTU and current test, showing that the data were in agreement. A fitted curve was obtained by the nonlinear regression routine of SigmaPlot (SPSS Inc., Chicago, IL). Based on the curve, 50% cutpoint diameter of the test is 3.95 ± 0.3 μm, while the result from the NCTU using polydisperse Al_2O_3 particles is 3.92 ± 0.22 μm.[16]

Micro-orifice impactor collection efficiency

In order to obtain the corresponding voltage for each particle size, an SMPS was used to measure particle size distribution at the outlet of the classifier. A relationship between the particle size and voltage was obtained (data not shown). The micro-impactor was tested by adjusting the voltage of the classifier to get expected particle size. Fig. 5 shows the collection efficiency curve of the micro-orifice impactor. With the data fitting, the 50% cutpoint diameter of the test is 94.7 ± 29.0 nm, while the result from the NCTU using oleic acid particles is 101.4 ± 0.1 nm.[16]

Aspiration efficiency

Fig. 6 shows the orientation-averaged aspiration efficiency for both PENS and NIOSH samplers compared to the inhalable convention curve at the wind speeds of 0.5 (A), 1.0 (B), and 1.5 (C) m s^{-1}. The results of three wind directions (0°, 90°, and 180°) are shown in Fig. 7 at different wind speeds. For small particles, it is possible that the particle concentration collected with the sampler is higher than that collected on the reference filters. This will result in the aspiration efficiency larger than 1. Table 1 lists the RMSD for different wind speeds and orientations. The average RMSDs for different wind speeds are also listed. For a comparison, the RMSDs from a previous study for IOM samplers are included. In order to compare with other studies reported in pertinent literature, the aspiration efficiency was described as a function of Stokes number (Stk), a dimensionless parameter, defined as $d_{ae}^2\gamma U$ per $18\eta\delta$, where d_{ae} is the particle aerodynamic diameter, γ is the density of pure water, and η is the viscosity of air. Fig. 8 shows a comparison of aspiration efficiencies for the two test samplers and the IOM personal samplers for a large range of the wind speed (0.4–2.2 m s^{-1}) and velocity ratios (U/U_0 = 0.1–10.5).

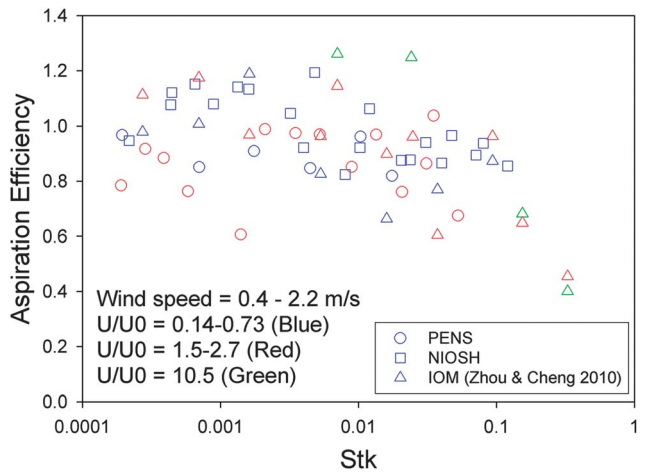

Fig. 8 Personal sampler aspiration efficiency compared with reported data as a function of Stokes number at all wind speeds (0.4–2.2 m s^{-1}).

Discussion

The calibration curves for the cyclone and impactor show the cutpoint diameters of 3.95 μm and 95.7 nm respectively, which is within the designed range. The cutpoint diameters are close to the result obtained by Tsai *et al.*,[16] wherein they used other methods to calibrate the cyclone.

For the PENS sampler, most of the orientation-averaged aspiration efficiency data points were around the curve at a wind speed of 0.5 m s^{-1}. However, the aspiration efficiencies were slightly lower than the convention curve for small particles at 1.0 and 1.5 m s^{-1}. Due to the effects of wind directions, the average data points showed a large bias. To compare the conversion curve, the closest aspiration efficiencies were obtained at a wind speed of 0.5 m s^{-1} with an RMSD of 0.17. For the wind speeds of 1.0 and 1.5 m s^{-1}, the RMSDs to the conversion curve are 0.34 and 0.31 respectively.

For the NIOSH sampler, the orientation-averaged data points were close to the convention curve at the three wind speeds. Most of the data were higher than the convention curve, especially the one at the wind speed of 1.5 m s^{-1}. The RMSDs at these three wind speeds are very close (0.21, 0.27, and 0.29). The closest one was also at a wind speed of 0.5 m s^{-1}.

The aspiration efficiency for both types of samplers was highly affected by the wind direction. The only one that had an RMSD below 0.2 was the PENS at a wind speed of 0.5 m s^{-1} when the sampling inlet was faced to the wind. The RMSD appeared larger than 0.3 for all other conditions. Only small particles (less than 2 μm) have an efficiency close to the conventional curve. Large particles had large deviations to the curve.

The velocity ratio (U/U_0) is another factor that can affect the aspiration efficiency. The samplers tested in this study were designed with a much smaller inlet size than the regular personal samplers. This design results in a small velocity ratio. The range of the velocity ratio was from 0.1–0.7, which is much smaller in comparison to the IOM test (2.7) in a previous study. However, as seen in Fig. 8, there are no significant differences between the studies. This indicates that the velocity ratio may not be an issue within a certain particle size and wind speed.

The next step is to use this sampler to collect nanomaterials such as carbon nanotubes and other nanomaterials to determine how the sampler can do with realistic materials.

Conclusion

Evaluation of a two-stage personal sampler for nanoparticles showed that the cyclone and micro-impactor stages have 50% cut-off diameters of 3.95 μm and 94.7 nm. The aspiration efficiency of this personal sampler and a NIOSH bioaerosol sampler at different wind speeds and orientations were also tested in a wind tunnel. The aspiration efficiencies of both samplers were close to the inhalable fraction curve. The orientation-averaged data had similar RMSDs to the one obtained from the IOM sampler tested previously. Data also showed that the aspiration efficiency was highly affected by the wind direction. The velocity ratio is not a factor that affects the aspiration efficiency at a low wind speed. However, it may be an issue at a high wind speed.

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