Characterization, Exposure Measurement and Control for Nanoscale Particles in Workplaces and on the Road

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Abstract. The amount of engineered nanoparticles is increasing at a rapid rate and more concerns are being raised about the occupational health and safety of nanoparticles in the workplace, and implications of nanotechnology on the environment and living systems. At the same time, diesel engine emissions are one of the serious air pollution sources in urban area. Ultrafine particles on the road can result in harmful effects on the health of drivers and passengers. Research on characterization, exposure measurement and control is needed to address the environmental, health and safety issues of nanoscale particles. We present results of our studies on airborne particles in workplaces and on the road.

1. Introduction

The quantity of engineered nanomaterials is expected to grow significantly in the next several years [1]. Analysts have estimated that the worldwide market for nanomaterials will be 700-1000 billion Euros in 2011 (www.nanosafe.org). Nanomaterial manufacturers are in need of fast and online instruments to characterize and measure the produced nanomaterials, and ensure that the concentration, size and morphology are in the expected ranges. Instruments are also needed to monitor emission and release of nanoparticles in workplaces to facilitate occupational safety and health. A number of recent toxicological studies suggest the total surface area of airborne nanoscale particles as a health-relevant exposure metric [2,3]. Researchers argue that the particles react with the biological matrix via the particle surface. The instrument Nanoparticle Surface Area Monitor (NSAM) was designed to measure the lung-deposited surface area of nanoparticles [4,5], and to provide real-time results. Many engineered nanomaterials are in the form of nanoparticle agglomerates [6]. Fast and online measurement for agglomerates is required in many scenarios including measuring fast changing agglomerates, quality control for material manufacturing, monitoring potentially toxic air-borne agglomerates, etc. We have developed the instrument Universal Nanoparticle Analyzer (UNPA), for online measurement of gas-borne nanoparticle agglomerates [6,7]. The principle and applications of UNPA are presented here.

Emission and exposure play important roles for societal impact of nanoparticles. Data are needed about the release of nanoparticles into the environmental and exposure to human. The US National Institute of Occupational Safety and Health (NIOSH) plans to develop recommended exposure limits.
(RELs) for ultrafine titanium dioxide and carbon nanotubes (CNTs) for the period of 2009 – 2012 (NIOSH 2009) [8]. We present emission measurement in an industrial production facility for CNT-imbedded nanocomposites. Exposure to ultrafine particles from engine emissions has drawn increasing concern because of its association with adverse health effects and its potential impact on daily commuters [9,10]. Particle concentrations on the road could exceed $10^6$ particles/cm$^3$, much higher than the ambient background concentration [11-13].

Filtration is one of the most effective ways for particle emission control. To reduce commuters’ exposure to airborne particles, an increasing number of automobiles have been equipped with a cabin air filter. The filters are characterized using particles larger than 300nm following the current International Standards Organization (ISO) standard (ISO 11155-1). However, particles smaller than 300nm comprise the majority of freeway particulate matter by number and surface area [11,14]. Therefore, Qi et al. [15] suggested that the ISO 11155-1 standard [16] to be extended to include particles smaller than 300 nm. We present the results of on-road evaluations of cabin air filters here.

2. Measurement and Characterization of Nanoscale particles

2.1. Nanoparticle Surface Area Monitor (NSAM)

Relatively few techniques are available to monitor exposures with respect to aerosol surface area. The BET measurement based on a gas adsorption method is not suited for a rapid evaluation of aerosol surface area at lower concentration [17]. It can be used only for powders, not for particles in the gasborne state. It does not have on-line capabilities. The epiphaniometer [18] was designed to measure aerosol surface area. This device measures the Fuchs or active surface-area of the aerosols by measuring the attachment rate of radioactive ions. The epiphaniometer is not well suited to widespread use in the workplace because of the inclusion of a radioactive source and toxic lead, and the lack of effective temporal resolution. One of other possible methods is diffusion charging (DC) which measures the surface area indirectly (e.g. [19]). When nanoparticles are exposed to a unipolar ion environment, they acquire a charge level which at least in a certain particle size range is theoretically proportional to the particle surface area [20]. If the particles are deposited on a filter, the electric current, induced by the rate of deposited particles, can be directly proportional to the total particle surface area [21]. The current can be measured by connecting the filter to an electrometer. Instruments using DC include LQ1-DC diffusion charger (Matter Engineering, Switzerland) and TSI model 3070a Electrical Aerosol Detector (EAD). While the LQ1-DC aims at measuring the Fuchs surface area, the EAD is designed for the particle length. Several studies using atmospheric field data, however, have shown that EAD can be used as a useful indicator for the quantity of particle surface area deposited in the lung [22-24].

Fissan et al. [4] observed that the response of EAD as a function of particle diameter is deviating from the charging theory and is closer to the lung deposited surface area, which is the geometric surface area weighted with the lung deposition curves. Based on these results, TSI Nanoparticle Surface Area Monitor (NSAM) model 3550 has been developed. Figure 1 shows a schematic of NSAM that uses a corona discharge to produce positively charged ions and mixes these ions with particles in an opposed flow mixing chamber. An ion trap is located downstream of the mixing chamber. The particles are deposited on a HEPA filter inside a Faraday cup and the current, induced by the deposited particles, is measured with an electrometer.
Fissan et al. [4] found the NSAM response can be adjusted. Between the mixing chamber and particle filter, all excess ions are removed in the ion trap by means of an electric field. Due to the high electric mobility of ions, the voltage can be relatively low. Even though the electric field strength within the ion trap is very low, some charged particles near the electrode of opposite polarity are also removed. Since in the human respiratory tract some particles also get lost before they reach the lung, the ion trap voltage can be adjusted such that the particle losses in the ion trap match those in certain areas of the human inhalation system. It is found that the response function of NSAM matches the surface area deposited in the tracheobronchial region with an ion trap voltage of 100 V, whereas the response function simulates the deposition in the alveolar region with an ion trap voltage of 200 V. The NSAM was calibrated [7] to measure the nanoparticle surface area deposited in two regions, tracheobronchial and alveolar of the human lung.

2.2. Measurement of Agglomerates using the Universal NanoParticle Analyzer (UNPA)

We have developed the instrument UNPA, for online measurement of gas-borne nanoparticle agglomerates [6,7]. UNPA utilizes Differential Mobility Analyzer (DMA), Condensation Particle Counter (CPC) and NSAM to characterize airborne nanoparticle morphology and measure the number, surface area and volume distributions of loose nanoparticle agglomerates. The key parameter measured is the UNPA sensitivity, which is defined as the current $I$ (fA) measured by the NSAM divided by the number concentration $N$ (#/cm$^3$) measured by the CPC.

$$S = \frac{I}{N} \text{ (fA cm}^3\text{).} \quad (1)$$

Charging theories of Chang [25] for aerosol particles of arbitrary shape indicate that the geometric surface area and electrical capacitance of the particles are two important parameters to determine the mean charge of non-spherical particles. The electrical capacitance of agglomerates may be computed using a variational method proposed by Brown and Hemingway [26]. The surface area of loose agglomerates may be calculated using a mobility analysis developed by Lall and Friedlander [27]. Shin et al. [7] combined the above analyses to show that the electrical capacitance of loose agglomerates is larger than that of spherical particles with the same mobility, and loose agglomerates can gain more charges from unipolar charging.
The primary particle size plays an important role in determination of the surface area and electrical capacitance, thus the charges on agglomerates. The UNPA sensitivity is related to the primary particle diameter \(d_p\). We found that the UNPA sensitivity can be correlated to the primary particle size through a power law relation [6]

\[
S = c_2 \left( \frac{12 \pi \lambda \ d_m}{c^* \ d_p^2 \ C_c} \right)^k \ c_1 (d_p)^h
\]

where \(c^*\) is a constant regarding particle orientation, \(\lambda\) is the mean gas free path, \(c_1, c_2, k\) and \(h\) are constants which can be determined from the experimental data. Then the sensitivity data from the experiments can be fitted into (2) to determine the primary particle diameter \(d_p\). Once the primary particle size is determined, surface area and volume of the agglomerates can be calculated.

We used the UNPA to measure silver spheres, and agglomerates of silver, SiO\(_2\) and TiO\(_2\) [6]. SiO\(_2\) and TiO\(_2\) agglomerates were generated by a diffusion burner. The sensitivities for silver spheres and loose agglomerates are the lowest and highest, respectively, among our sensitivity data (Fig. 2). The sensitivities for TiO\(_2\) and SiO\(_2\) agglomerates are between the upper and lower bounds. We also used UNPA to measure gold and nickel nanoparticle agglomerates from a spark discharge generator [28]. We determined the primary particle sizes for agglomerates using the sensitivity data and equation (2). We also analyzed electron micrographs and obtained the primary particle sizes. A comparison of the primary particle sizes from the two methods is shown in Table 1. It can be seen that the agreement for the primary particle size is good and UNPA can measure primary particles in a rather wide size range.

![Figure 2. Measured UNPA sensitivity for silver agglomerates and spheres, SiO\(_2\) and TiO\(_2\) agglomerates.](image-url)
Table 1. A comparison for the primary particle sizes obtained from the UNPA sensitivity analysis and electron microscopy (EM).

|                  | $d_p$ from UNPA sensitivity (nm) | $d_p$ from EM (nm) | standard deviation of $d_p$ from EM (nm) |
|------------------|----------------------------------|--------------------|------------------------------------------|
| TiO$_2$ agglomerates | 22.8                             | 19.9               | 7.5                                      |
| SiO$_2$ agglomerates | 32.5                             | 27.3               | 9.3                                      |
| Au agglomerates    | 5.97                             | 7.90               | 1.47                                     |
| Ni agglomerates    | 4.09                             | 6.56               | 0.98                                     |

Diesel engine exhaust is a major source for on-road ultrafine particles and has profound impact on environment and ecosystem. Diesel aerosols are mainly agglomerates with open structures and composed of primary particles (Fig. 3). We measured diesel aerosols generated from a 4045 John Deere Diesel Engine using UNPA [28]. A schematic of the experiment is shown in Fig. 4. Diesel aerosols were sampled from the exhaust pipe and mixed with dilution air in the residence chamber. Then the diesel aerosols were sent to Nanometer Aerosol Sampler (NAS) for collection of electron micrograph samples, and to UNPA for agglomerate measurement.

![TEM images of diesel particles under the following conditions (a) light load, (b) heavy load.](image)

Figure 3. TEM images of diesel particles under the following conditions (a) light load, (b) heavy load.
We sampled the diesel aerosols under two engine conditions: light load and heavy load. The engine speed, torque and exhaust temperature increased with the load. A catalytic stripper was used to remove sulfur compounds and the soluble organic fraction by passing the diluted diesel exhaust over two heated catalysts. The primary particle sizes of diesel particles measured by UNPA are listed in Table 2. The primary sizes were also measured using a large number of TEM images. A total of 212 primary particles were analyzed in the case of heavy load, and 189 primary particles in the case of light load. The primary particle size from TEM agreed well with UNPA measurement for the heavy load case. In the case of light load, there was about 6 nm difference between UNPA and TEM results, which was still within the standard deviation of the TEM result.

**Table 2.** Comparison of the primary particle size in diesel agglomerates measured by UNPA and TEM.

| Engine load | Primary particle sizes from UNPA (nm) | Primary particle size from TEM (nm) |
|-------------|-------------------------------------|-----------------------------------|
| Light       | 29.5                                | 23.2 ± 6.6                        |
| Heavy       | 24.2                                | 23.9 ± 6.8                        |

For loose agglomerates, UNPA can measure the number, surface area and volume distributions. We show the results for the volume of silver agglomerates in Fig. 5. The distributions based on spheres are also plotted for comparison. The difference of the volume distributions is substantial. The results show that the UNPA measurement can potentially improve distribution measured based on spheres significantly.
Based on direct mobility scanning
Corrected for agglomerate using Lall and Friedlander model

Figure 5. Agglomerate volume distribution from direct mobility scanning and after correction by Lall and Friedlander model (silver agglomerates, primary particle diameter measured by UNPA: 8.4 nm).

3. Exposure Measurement for Production of CNT-imbedded Nanocomposites

The unique properties of CNTs have led to their increased usage in advanced materials. CNTs are used in structural composites for sporting equipment, conductive plastics, electron field emitters, semiconductor devices, etc. As more industries incorporate CNTs into consumer products the opportunities for worker exposure will rise. NIOSH specified identification of the long-term health effects of CNTs in its strategic plan for 2009 – 2012 (NIOSH 2009, [8]). We review our exposure measurement of CNTs in the production of nanocomposites.

We measured exposure in an industrial production facility for CNT-imbedded nanocomposites [28]. In the extrusion operation, CNTs were mixed with melted polymer and other compounds to produce nanocomposite pellets. First polymer was melted and mixed with CNTs. The mixture was extruded from a die into a water bath. Cooled strands passed through a dryer before entering a cutter. The pieces were fed into a second extruder with CNT dispersing agent. At the exit of the second extruder the strands were pelletized. The pellets passed through a shaker and a cyclone for size selection.

A suite of instruments was used to obtain air-borne particle number concentration, surface area concentration, and size distribution data. The instruments were placed in the closed room near the extrusion system. The measurement results are shown in Fig. 6. The measurement duration can be divided into five periods. Period I (from 12:50 to 13:40) accounted for when there was no activity in the room. The total number concentration was low, about 6,000 #/cm$^3$. In Period II (from 13:40 to 13:55), the system was warming up. The particle concentration increased, possibly due to evaporation of polymeric residuals in the extruder. In Period III (from 13:55 to 14:15), the warm up continued and a spike of 10 – 40 nm particles appeared. These particles did not cause significant increase in the lung-deposited surface area because of their small sizes, and were likely polymer residuals from the warm up. In Period IV (from 14:15 to 14:50), the first extruder was running. The particle number concentration increased to about 40,000 #/cm$^3$. In Period V (from 14:50 to 15:00), both extruders were running in the full production mode. The particle number concentration increased to about 90,000 –
100,000 #/cm$^3$ and the particles were mainly in the range of 30 – 90 nm. We believe that volatile polymer fumes were a major particle source. This is corroborated by the observation that the particle concentrations were highest near the extrusion barrel and vapor was visible at the extrusion barrels. Further research is needed to find out the fraction of CNTs in the total particle concentration.

**Figure 6.** Measurement for air-borne particles during the extrusion operation for CNT-imbedded nanocomposites. Upper panel shows the total number concentration and lung-deposited surface area concentration; lower panel shows the evolution of the particle number-size distribution.

**Figure 7.** Examples of SEM images of the CNTs agglomerates collected on filters during the extrusion process. The picture on the right is a magnified local view of the one on the left.
We also used a filter to collect samples for electron microscopic analysis. Examples of the SEM pictures are shown in Fig. 7. We found a number of CNT agglomerates on the filter. The sizes of the CNT agglomerates are in the range of 1 µm to 20 µm. The individual CNTs are much smaller, with the diameter of about 40 nm. The CNTs were already in the agglomerated form when provided by the manufacturer. Such large agglomerates have limited mobility, and are easier and safer to handle and transport. Question remains whether the CNTs agglomerates can be disintegrated in the extrusion process and whether single CNTs can be released. Thus far we have not found single CNTs on the filter samples. Further research will be performed to identify possible released single CNTs.

4. On-road exposure and recirculation control in car cabins

With the filter installed in the test vehicle and driving in freeway traffic, we measured particle number and surface area concentrations of exterior freeway air and cabin air simultaneously and determined the exposure reduction based on the two concentration metrics [15,30]. The detailed testing protocol is as follows. New cabin air filters were installed in the test vehicle before each on-road test to make sure the test data were comparable. Two water-based condensation particle counters (WCPC, TSI 3785) were used to measure the particle number concentration. The WCPC 3785 is able to measure the particle number concentration up to 10^7 particles/cm^3 and has d50% (particle size with 50% counting efficiency) at 5 nm, making it very suitable for such on-road ultrafine particle measurements. A pair of AeroTrak 9000 (TSI Inc.) instruments were used to measure the human lung deposited surface area concentrations. The AeroTrak 9000 is the portable version of the NSAM. NSAM/AeroTrak 9000 was calibrated against neutralized particles [5], thus a neutralizer (Po210 source) was used upstream of each AeroTrak 9000 to make sure the sampled particles are neutralized. The AeroTrak 9000 was set to measure the lung deposited surface area concentration in the alveolar region only. The two WCPCs and AeroTrak 9000 units were tested before being deployed in the on-road tests and showed a high internal precision (R^2 value of 0.99 for both particle number and surface area concentrations). Data collected from both WCPCs and AeroTrak 9000 were in 1 s intervals to provide a high temporal resolution.

All the windows were closed during the on-road tests. For the test in a Saab 93 (2003 model, cabin volume of 2.55 m^3), when the fan is on and the recirculation air is off, outside air is brought in after passing through the cabin air filter and then released to the outside through gaps around the doors and windows due to the positive pressure in the cabin. In this study, fan speed levels 2 (medium) and 5 (high) were tested, with flow rates at 3600 and 7200 liters per minute (LPM), respectively. A WCPC and an AeroTrak 9000 were used to measure particle concentrations of exterior freeway air sampled through a probe mounted on the car window. All on-road tests were conducted when driving on interstates I-35 and I-94 during rush hour (3-6 pm), providing representative data for typical commuters on Twin Cities, USA freeways.

Figure 8 shows the particle concentrations measured outside and inside the vehicle when driving on the freeway with the fan speed at level 5 (high) and the recirculation air off. In the same time span, the data from WCPC (Fig. 8a) and AeroTrak 9000 (Fig. 8b) followed very similar trends for both outside and inside concentrations. The outside concentration varied rapidly due to the change of the vehicle’s position in the traffic and emission status of the surrounding vehicles, while the inside concentration fluctuated much less. The more stable inside concentration was explained by the in-cabin space serving as a chamber that damped down the outside variations.

The effect of the cabin air filter can be quantified by the exposure reduction (ER), which can be defined as

\[ ER = 1 - \frac{C_i}{C_o} \]  

where \( C_i \) and \( C_o \) are time averaged particle concentrations inside and outside the car, respectively (15 min duration in this study). As shown in Fig. 8, data from both WCPC and AeroTrak 9000 indicate an obvious average exposure reduction in the cabin. At high fan speed, the average ER based on number concentrations is 55.0 ± 3.3% (from data of more than 10 replications, the same for all subsequent
data), while the ER based on lung deposited surface area concentrations is 48.9 ± 8.8%. The number- and surface area-based ER are 65.6 ± 6.0% and 60.6 ± 9.4% at medium fan speed, higher than the corresponding values at high fan speed due to a higher filtration efficiency at lower face velocity. The lower ER value for surface area concentration is reasonable because for freeway particles, the maximum particle concentration for the surface area-based size distribution is closer to the most penetrating particle size of the cabin air filter (about 350 nm) than the maximum particle concentration for the number based size distribution.

![Graph](a)

**Figure 8.** Particle (a) number and (b) lung deposited surface area concentrations of freeway air and cabin air observed on December 6, 2007 while driving the Saab 93 on interstate I-35 with the recirculation air off and fan speed at level 5.

The effect for exposure reduction is more pronounced when the recirculation mode is used in the car cabin. The tests were performed in a Toyota Camry (2007 model) with the cabin air filter in the recirculation loop. In-cabin aerosol concentration in the Camry was reduced to below typical office air concentrations (~ 4,000 particles/cm$^3$) in approximately 3 min. Figure 9 shows the time-resolved in-cabin aerosol size distribution during recirculation. It can be seen that particle concentrations of all the sizes were reduced continuously during the recirculation. Aerosol particles < 100 nm were rapidly removed from the air by the recirculation ventilation system. As would be expected from filtration
theory, particles close to the filter’s most penetrating particle size were removed with the least efficiency, but still experienced significant reductions. This result demonstrates that the recirculating air filtration is especially effective in reducing exposure to ultrafine particles.

![Graph showing particle size distribution](image)

**Figure 9.** Measured variations in particle size distribution inside a Toyota Camry, with the air ventilation system in recirculation mode and the air filter in place. $D_p$, particle diameter; $N$, particle number concentration in size bin $\Delta \log D_p$.

5. **Summary**
Nanoscale particles exist in workplaces and on the road and their effects on environment and health of workers and passengers are of concern. We performed research covering particle characterization, exposure measurement and control. NSAM provides real-time measurement for the lung deposited surface area of airborne nanoparticles, which is a health-related metric. UNPA can differentiate compact particles from particles with open structures and can measure the number, surface area and volume distributions of loose nanoparticle agglomerates. We have applied UNPA to measure engineered nanoparticles and diesel exhaust particles and obtained satisfactory results. Measurement at a CNT composite production facility showed that CNT agglomerates were released and there was possible exposure. On-road test results demonstrated that using air recirculation can substantially and rapidly reduce exposure to airborne ultrafine particles.
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