Validating SMPS-measured size distribution of double-mode spherical Silica nanoparticles by Transmission Electron Microscope (TEM)

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Introduction: A nanoparticles size is one of their key physical characteristics that can affect their fate in a human's respiratory tract (in case of inhalation) and also in the environment. Hence, measuring the size distribution of nanoparticles is absolutely essential and contributes greatly to their characterization. For years, Scanning Mobility Particle Sizers (SMPS), which rely on measuring the electrical mobility diameter of particles, have been used as one of the most reliable real-time instruments for the size distribution measurement of nanoparticles. Despite its benefits, this instrument has some drawbacks, including equivalency problems for non-spherical particles (i.e. assuming a non-spherical particle is equal to a spherical particle of diameter *d* due to the same electrical mobility), as well as limitations in terms of its use in workplaces, because of its large size and the complexity of its operation (Kuhlbusch T, Asbach C et al. 2011).

In this study, we aimed to evaluate the performance of the SMPS in measuring the size distribution of double-mode silica particles, by comparing the results with the size distribution calculated from the analysis of TEM images.

Methods: A water-based colloidal suspension containing two populations of silica particles with different sizes was aerosolized using a nebulizer. The aerosol flow passed through a diffusion dryer and was divided into two separate flows: one to the SMPS for on-line size distribution measurement and the other to the electrostatic precipitator with a Formvar-Carbon coated cupper TEM grid inside for particle deposition.

30 SMPS scans were generated (3 days, 10 each) to verify the repeatability and reproducibility of the results. TEM imaging was done by a JOEL 2100 instrument and the particles dimensions were measured using *ImageJ v1.45k* analysis software. The measured diameter range of the Klebosol particles was divided into several *bins* and the number of particles in each bin was counted and used to calculate the size distribution.

Results: Figure 1 presents the size distribution of silica particles measured by the SMPS. As shown, there are several peaks in the graph but not all of them are related to silica particles. They represent solute residues (#1), doubly charged particles (#2) and finally, particle clusters (#3). However, both silica particle populations were also detected by SMPS and are circled in the figure (peaks of interest).

The average mean diameter/GSD measured by the SMPS, for the first and second population of silica particles were (45.1 nm/1.077 nm) and (82.2 nm/1.071 nm), respectively. Table 1 summarizes the results of size distribution measurement using TEM images.

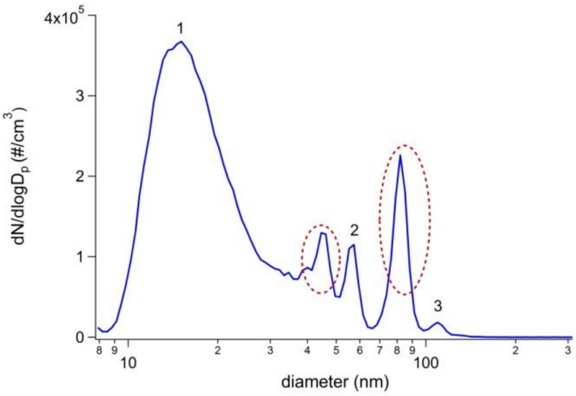

Figure 1. Size distribution of silica particles; SMPS results

Table 1. TEM analysis results

Population	***Size range (nm)***	***Mean diameter (nm)***	***GSD (nm)***	***Number of particles***	***Aspect ratio***
First	31.7-58	42.9	5.38	37	1.055
Second	58.1-89.5	78.9	5.96	68	1.041

According to the results, there is a difference between the mean diameters derived from the SMPS and TEM (5% for the first population and 4% for the second), which could be due to the sampling methods, the use of a neutralizer in the SMPS or statistical errors caused by the insufficient number of particles analyzed using the TEM method.

Conclusions: Despite the minor discrepancy mentioned above, the SMPS results showed a high level of accuracy in measuring the size distribution of double-mode spherical nanoparticles. However, the results might differ for non-spherical particles of a more complicated morphology, since they might have unknown orientations in the SMPS classifier which could result in measurement inaccuracy.

Kuhlbusch T, Asbach C, et al. (2011). "Nanoparticle exposure at nanotechnology workplaces: a review." _Particle and Fibre Toxicology_ **8**(22): provisional version.