Evaluation of different sampling methods for TEM-analysis to characterize single digit aerosol nanoparticles

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Single digit aerosol nanoparticles are used in a wide field of applications. These particles can be applied as catalysts in chemical reactions because of their high specific surface area. To describe the catalytic effect it is necessary to characterize the particle composition, shape and size as well as surface properties and crystal structure. For example TiO_2 nanoparticles that are used to degrade or absorb pollutants in water and soil systems are determined by the aforementioned properties. For 4 nm particles about 50 % of the atoms are on the surface, which changes the surface reactivity significant.

To describe the particle size, investigations down to 2 nm particle diameters are possible with several online-measurement methods such as Scanning Mobility Particle Sizer (SMPS) coupled with counting techniques e.g. Ultrafine CPC or Particle Size Magnifier. While this method is able to determine a particle size distribution, it does not deliver information on the material, form, lattice structure or composition of the produced particles. Transmission electron microscopy (TEM) is part of the techniques needed for characterization. For this purpose, however, it has to be ensured that a representative sample is taken. A drawback of TEM analysis is the quantification of the aerosol concentration from the measured particle number distribution on the TEM grid.

Therefore, the relation between particle deposition on the TEM grid and the initial aerosol concentration was investigated. Depositing mechanisms like diffusion and electrostatic at ambient pressure and impaction at low pressure were studied.

To describe the deposition on the TEM grid the experimental set-up consists of a spark generator to produce spherical particles, neutralizer and differential mobility analyzer (DMA) to classify the particles and a faraday cup electrometer (FCE) to describe the particle concentration before and after the sampling. The neutral particles were analyzed by using a precipitator to separate the charged particles.

After the experiments the mean particle concentration on the grid was determined from the TEM micrographs. Thus, the particle flux, depending on the grid surface and sample time, can be calculated. Then, the particle deposition velocity v* is defined by particle flux divided by initial aerosol concentration. Once the deposition velocity is determined, the characterization of an undefined aerosol concentration, dependent on the deposition method, is possible.

Figure 1 shows particle size vs. depositing velocity. The depositing velocity is increasing with decreasing particle diameter. The highest deposition velocity was reached by electrostatic force (1000 V).

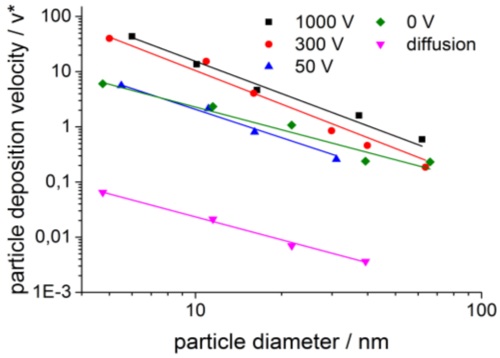

Figure 1. Deposition velocity v* of different sized particles dependent on electrostatic force, charge of the particles and diffusion effects.

The results shown as “0 V” represent the charged particles without an external electrostatic force. This means the deposition only occurs because of diffusion and induced image force near the grid surface. The deposition efficiency of neutral particles by diffusion effects was found to be too low for a solid estimate of the aerosol concentrations or particle size distributions.

However, the particle charge has no influence when using the impactor set-up. Therefore, particles were sampled with the impactor and a size distribution was measured with “DMA and FCE” combination at the same time (Figure 2).

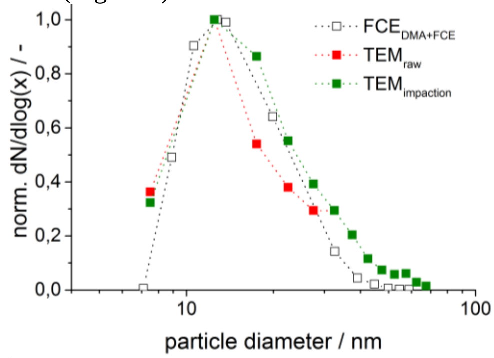

Figure 2. Comparison of the normalized size distributions

It was possible to eliminate the disagreement between “DMA and FCE” and TEM_{raw} (raw data) size distributions by measuring the rebound curves of several particle diameters on the TEM grid. The rebound curves describe the efficiency of particle deposition on a TEM grid dependent on the impaction velocity. The $TEM_{Impaction}$ curve shows the corrected distribution. This offers a good match to “DMA and FCE” measurement. Both particle size distribution and particle concentration can be determined by sampling with impactor on a TEM grid.