

Using aerosol instruments to study the residual particles formed in the Liquid Flame Spray

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Liquid-fed aerosol flame synthesis techniques are widely used for nanoparticle production in both laboratory and industrial scale. In such techniques, liquid droplets of precursor solution are sprayed into a high-temperature flame, where, ideally, the material vaporizes and goes through the gas-to-particle aerosol route to form nanoparticles. However, incomplete vaporization of the droplets can produce micron-sized residual particles via the liquid-to-particle route (i.e. classic spray pyrolysis). In this unwanted process, material is wasted and the homogeneity of the particulate product decreases.

The formation of the residual particles has been an issue, especially, with some of the inexpensive metal nitrate precursors. In recent studies, the addition of 2-ethylhexanoic acid (EHA) to the solvent has been shown to reduce the formation of the residual particles (Strobel and Pratsinis, 2011), due to the microexplosions within the droplets (Rosebrock *et al.*, 2016). The residual particles have been mainly studied using techniques, such as, electron microscopy and nitrogen absorption. In this study, the mass and number size distributions of alumina and silver residual and nanoparticles are determined quantitatively using on-line and off-line aerosol measurement instruments.

The aerosol particles were produced with the Liquid Flame Spray (LFS, see e.g. Haapanen *et al.*, 2015) technique, which employs a hydrogen-oxygen flame. Aluminium and silver nitrate dissolved in ethanol (EtOH) and EHA at different volume concentrations were used as precursors for alumina and silver particles, respectively. The mass size distributions of the particles collected with a Dekati low pressure impactor (DLPI). Furthermore, a scanning mobility particle sizer (SMPS) and an electrical low pressure impactor (ELPI+) were used to measure the number size distributions of the produced nanoparticles.

The particle size distributions measured with the different aerosol instruments were consistent with each other. With silver nitrate as a precursor, no residual particles were detected, and the addition of EHA had no observable effect on the mass and number size distributions. On the other hand, the mass size distribution of particles generated from aluminium nitrate dissolved in pure EtOH was dominated by the micron-sized residual particles, as can be seen in Figure 1 (red line). With the addition of EHA, the distribution shifted practically entirely from the residual mode to the nanoparticle mode (blue line). The particles that were size classified and collected with the DLPI were also characterized with a scanning electron microscope

(SEM). Figure 2 shows that the micron-sized residual particles (left panel) were spherical, as expected from the liquid-to-particle formation, whereas, the nanoparticles (right panel) formed through the gas-to-particle process were homogenous and highly agglomerated.

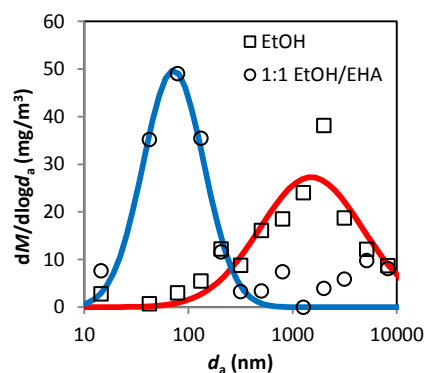


Figure 1. Mass size distributions of alumina particles produced using two different solvent compositions.

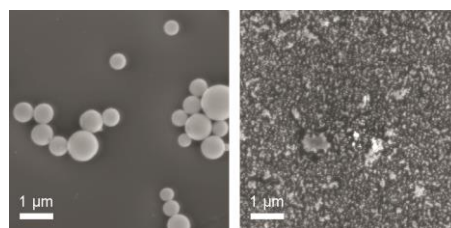


Figure 2. SEM micrographs of alumina residual (left) and nanoparticles (right).

This study demonstrates that aerosol measurements can give valuable information on the particles produced with aerosol flame synthesis methods, especially, regarding quantitative information on the possible residual particles. Furthermore, in some cases, the on-line measurements alone can replace some of the tedious and time-consuming off-line methods. All in all, the results presented in this study are important when thinking about the production costs of nanoparticles at an industrial scale.

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