

Particle Metrology and Standards

As technology migrates toward smaller physical dimensions, new analytical approaches are required to characterize material properties and to investigate scientifically important issues. In particle technology, these needs become critical in the nanosize regime. Industrial processes involving nanoparticle technology include advanced device manufacture and the development and production of functional materials for microelectronic, pharmaceutical, and biotechnology applications. Our goal is to improve existing metrology and develop new methods and standards for measuring the physical and interfacial properties of nanosize and nanostructured particle systems.

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Standard reference materials (SRMs) are critical for calibrating instruments used for nanoparticle characterization. Primary customers for nanoparticle SRMs are quality control laboratories in the ceramic and pharmaceutical industries. This year, we have initiated the production of a 100 nm to 500 nm particle size distribution (PSD) SRM, the first of its kind (inorganic material system with a broad size distribution), to fulfill a pressing need in the sub-micrometer size range. Interest in this TiO₂-based standard has been expressed from a wide range of industries. The SRM will be certified for PSD using electron microscopy and by X-ray disc centrifuge methods. Additionally, certification of SRM 1021 was completed. This SRM is intended for the evaluation and calibration of equipment used to measure PSDs in the 2 μm to 12 μm diameter range. The size range of this SRM follows that of the coarser beads of existing SRM 1003c.

Another activity involves ultra-small angle x-ray scattering (USAXS) of nanoparticle suspensions and partially-gelled systems. Objectives are to develop

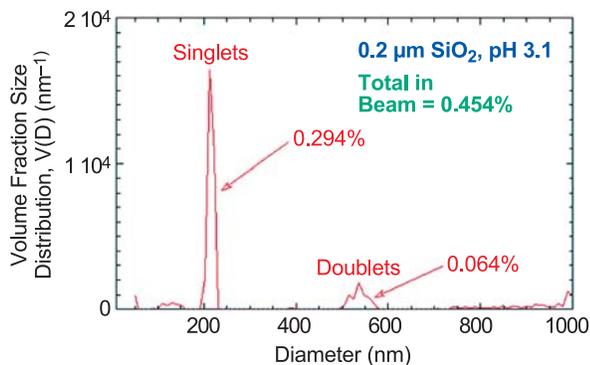


Figure 1: Volume fraction PSD for SiO₂ at the isoelectric point.

sensitive metrological techniques for characterizing metastable and unstable nanoparticle suspensions and to gain new insights into mechanisms influencing their agglomeration and stabilization. Systems of interest include SiO₂, TiO₂, and ZrO₂, studied after stabilization at pH values representing states close to and far from the isoelectric point. For example, Figure 1 shows discrete particle doublets (right-hand peak) in SiO₂ suspensions that form only at the isoelectric pH. USAXS allows such phenomena to be quantified to an unprecedented level. A flow cell for *in situ*, real-time USAXS studies of unstable suspensions is under development.

This year, a new activity was initiated on interfacial property measurements of nanostructured particulates. The goal is to develop the measurement methodology to obtain reliable data to establish a clear relationship between charge development and particle size, crystallinity and interparticle structure. Metal oxides exposed to an aqueous phase develop charge via protonation of surface hydroxyl sites. Surface charge and surface site reactivity play key roles in dissolution, precipitation and growth, corrosion, aggregation, and sorption processes. As particle size shrinks into the low nano-regime (< 10 nm), charge development and reactivity are predicted to exhibit size dependent behavior. Preliminary results indicate some potentially important findings.

- A systematic decrease in the isoelectric point of TiO₂ with decreasing crystallite size was observed; data will be incorporated into modeling efforts by NSF-funded researchers to account for proton-induced charge formation as a function of crystal size.
- Zeta potential measurements indicate that adducts formed between nanocrystalline MgO and molecular halogen species are more stable in an aqueous medium than had been previously predicted; this has important implications for their use as microbial biocides.
- Small-angle neutron scattering experiments suggest MgO may be capable of localized expansion around individual crystallites in response to an aqueous intrusion, without fully disrupting the assembly structure; this would represent a unique attribute not typically associated with inorganic particles.

Contributors and Collaborators

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