Combining SAXS and DLS for simultaneous measurements & time-resolved monitoring of nanoparticle synthesis

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Abstract

Time-resolved characterization of nano-particle (NP) synthesis is a promising mean to produce NPs under controlled conditions. Here, an innovative experimental demonstration of a NP characterization tool which combines a laboratory Small Angle X-ray Scattering (SAXS) instrument, a new Dynamic Light Scattering (DLS) device and a microflow reactor is shown. The complementary SAXS and DLS techniques were designed and optimized to meet the ambitious requirements of time-resolved monitoring of NP suspensions while ongoing synthesis. For this purpose, SAXS instrument performance was enhanced by the implementation and optimization of a unique X-ray metal jet source. In parallel, an innovative DLS fiber remote probe head was developed specifically for in situ measurements. DLS measurements were performed directly inside a 2.0 mm diameter glass capillary located inside the SAXS vacuum sample chamber. The combined SAXS and DLS devices were tested separately on commercially available gold NP suspensions of known size. Furthermore, simultaneous SAXS and DLS measurements were performed during the synthesis of silica NPs.

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Figures

Fig. 1. X-ray beam collimation with two SCATEX pinholes.
Fig. 2. Schematic drawing of the OFRH. It incorporates the optics for the main laser and the scattered light. The optics are coupled to optical fibers.
Fig. 3. Optical Fiber Remote Head (top) integrated into the SAXS sample chamber (bottom) with a temperature sensor (1), capillary holder (2) the DLS head (3) and the optical fibers (4).
Fig. 4. Schematic drawing of the production and characterization line of a NP suspension.
Fig. 5. Flow-through capillary holder inside the SAXS sample chamber. The X-ray and laser beam hit the flow-through capillary at the same position
Fig. 6. SAXS measurements (top) and DLS measurements (bottom) on standard gold NPs with radii of 5 nm, 7.5 nm and 10 nm. The SAXS curves were fitted assuming a Schulz-Zimm (solid line) and a Gaussian (red dashed line) size distribution, respectively.
Fig. 7. TEM images of standard gold NPs with diameters of 10 nm, 15 nm and 20 nm.
Fig. 8. Evolution of the mean radius Rm and r during the monitoring experiment
Fig. 9 Representative SAXS curves of SAXS measurements taken from each section. The black lines are the fitted curves.
Fig. 10. TEM images of NPs synthesized in Sections 1,2,4 and 5. For Section 3 no proper images could be made due to agglomeration of the NPs.