Inline particle sizing using Modulated 3D Dynamic Light Scattering

Dynamic Light Scattering (DLS) is the standard tool for sizing of nanoparticles in solution. The theory of this technology is based on the Brownian motion of particles. The thermal motion of the particles can be monitored by means of light scattering. The coherent light scattered from particles carries a fingerprint of the particle motion, which is described by the diffusion coefficient of the particles which is in direct relation to the hydrodynamic radius R_h. As long as the viscosity and the temperature are known, DLS can obtain the hydrodynamic radius of the particles.

The principle is based on the correlation of photons scattered from the sample and detected by a suitable photon detector. It requires that only single scattered photons arrive at the detector. While the criterion of single scattering imposes no problems for solutions of very low turbidity (= low concentration = low scattering), it renders measurement of slightly turbid solution impossible, because this results in undetectable systematic errors. Dilution of the solution below critical turbidities is only as first approach to address this problem, since the dilution may modify the sample. Dilution thus needs to be reduced as much as possible. This can be done with the 3D modulation technology. It suppresses multiple scattering to filter out only the single scattering (figure 1).

With this technology, we were able to show that in all cases studied, measurements with no dilution were possible.

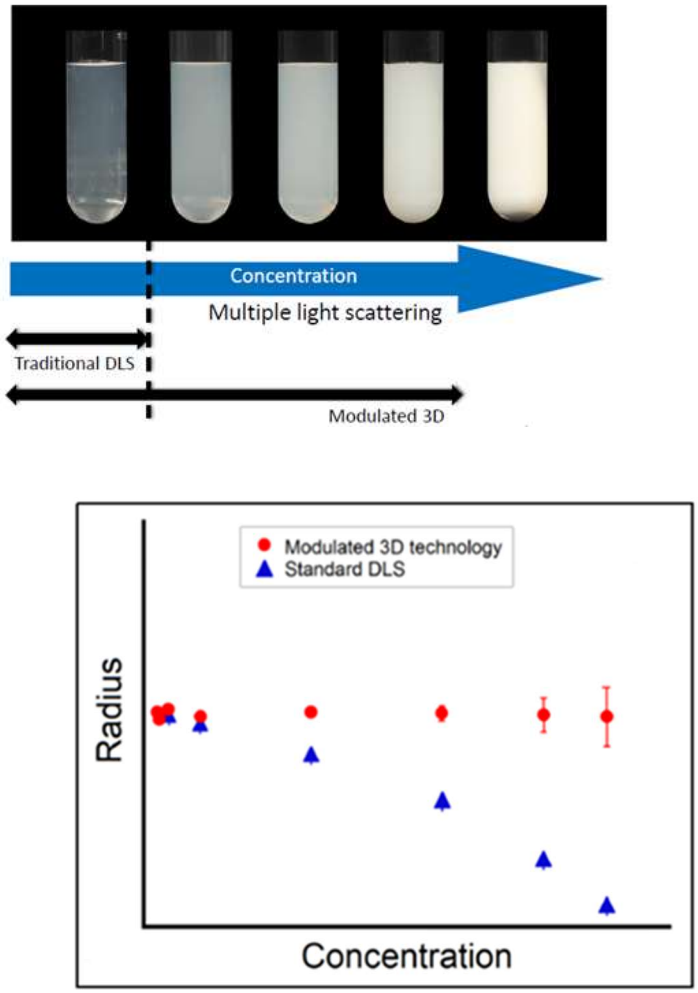

Figure 1: Modulated 3D technology. The triangles indicate the systematic deviation from the true radius at higher concentration.

The newly developed DLS system was successfully validated in a small (2L) pilot reactor environment. The instrument's design was adapted in order to accommodate a flow cell including tubing material to enable online measurements. A sampling loop was implemented and preliminary tests for an implementation of the modulated 3D technology into the pilot reactor setup were successful.

As described above, the DLS system monitors movement of particles. A direct connection of the sample cell to the reactor via a tubing system could easily transmit vibration and cause movement of the particles larger than that caused by Brownian motion. It was thus necessary to decouple and reduce the vibration sufficiently such that the thermal motion of particles in the measured sample volume dominates. Both measurements with flow and without flow First tests of particle size measurements in static mode were in good agreement with reference to a Scanning Electron Microscopy (SEM) and a Malvern DLS instrument.

From the first static validation experiments it was concluded that the measured particle sizes of standardized (NIST) and consortium particles were in the expected size range. The positioning of the flow cell was optimized to improve the accuracy of the measurements during flow.

Particle growth of SiO_2 and TiO_2 particles was successfully monitored online. When online static measurements are compared to dynamic measurements, a comparable outcome in particle size is observed.

Nicole Meulendijks
Research Scientist
TNO
nicole.meulendijks@tno.nl

Dr. Andrea Charles Voelker
CEO
LS Instruments AG
charles.voelker@lsinstruments.ch