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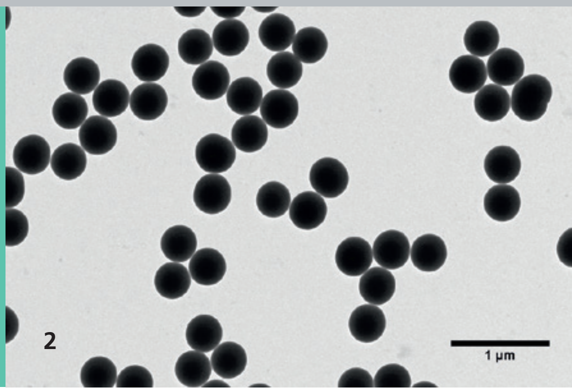

1 *The flow DLS device*
2 *TEM micrograph of silica nanoparticles*

INLINE/ONLINE DYNAMIC LIGHT SCATTERING

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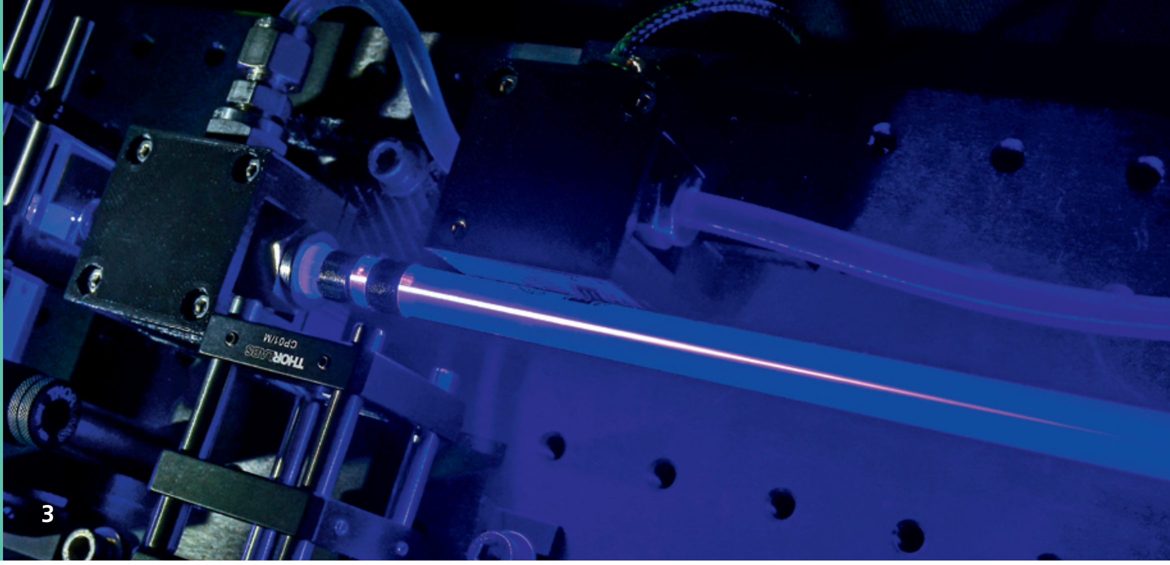
Introduction

Dynamic Light Scattering (DLS) is a very powerful and well-established method for the characterization of particles in dispersion as well as for polymers in solution. Although the correct data analysis is not trivial in DLS, the measurement itself is simple and modern instruments are easy to use. Additionally, the method is not relying on special properties of the analyte as, for example, fluorescence, therefore it is applicable on a wide spectrum of analyte materials and solvents. However, there is a lack of fast and cost effective inline/online particle sizing devices that take advantage of the benefits of Dynamic Light Scattering. Therefore, a new technique for measuring DLS continuously and in flow was developed, patented and implemented in a fully integratable, easy-to-use instrument.

Basic principles

DLS is, as light scattering in general, an ensemble method, meaning that measurements are made on a large number of particles in a small sample volume, the so-called scattering volume. The underlying principle is the Brownian motion of the particles; the primary measurement parameter is the intensity of the light scattered by the sample after being irradiated by a laser light source. In DLS, an autocorrelation function is applied to the data: The current scattering signal is correlated to the signal at previous time points. By this procedure, the scattering signals are "translated" into the diffusion coefficient of the investigated particles which again is converted (by application of the Stokes-Einstein equation) into the mean value of the hydrodynamic particle size. This implies that the result is the mean value of a diameter equivalent to that of a spherical particle with the same hydrodynamic properties as the measured sample.



Active flow compensation

In their data processing, other techniques that are used to measure DLS in flow have to deal with the additional particle movement caused by the sample flow. This leads to the problem that strictly constant flow conditions are required. In contrast, the system developed by Fraunhofer IMM is able to suppress the influence of the flow dependent movement before the calculation of the particle size is started. This substantial advantage is achieved by efficient digital image processing. After this treatment, the following data processing steps are nearly identical to conventional DLS measurements in a cuvette.

High flow rate

The current optical and fluidics setup allows the determination of particle sizes at high flow rates of up to 200 ml/min. The flow does not have to be steady – as long as it is laminar, measurements under pulsing or even alternating flow conditions are possible.

Cost-effective components

The principle of measurement permits the use of low-price image sensors, laser diodes and standard computer hardware as they are found in consumer electronics. Therefore, the pure costs of the components are very low, especially compared to current conventional setups. The flow DLS device consists mainly of OEM parts which can be easily modified, replaced or upgraded, meaning the performance of the instrument is scalable.

Instrumentation and control

For data processing, an integrated barebone PC is connected to the electronics of the system. The flow DLS device auto-calibrates to different particle sizes and concentrations as well as to different flow velocities. No user input is required during operation. The instrument can be connected to an industrial process control system by a serial interface. The measurement results may also be inspected on the integrated touchscreen monitor.

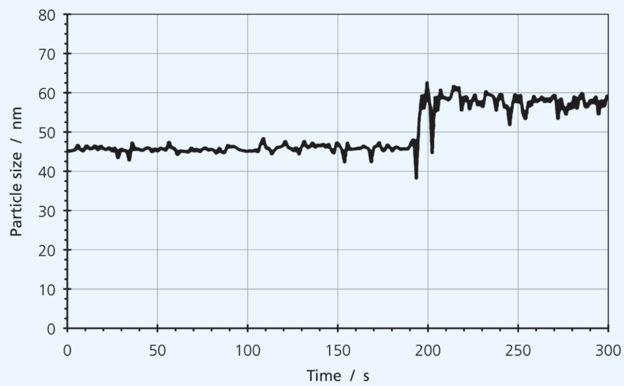
Application example

To demonstrate the potential of the flow DLS device, it was incorporated into a microreactor-based, continuous synthesis setup. Using this setup, vesicular structures from non-ionic surfactants ("niosomes") and functionalized silica particles were produced. In both cases, the flow DLS device served as a real-time monitoring system of the product stream and proved to be capable to detect quick changes in particle size. By checking the particle size up- and downstream of the in-flow work-up routine, the flow DLS device revealed that the product passed the work-up routine unaltered. When implemented into a process control system, a feedback loop can be established to, e.g., constantly control the product properties by automatically adjusting process parameters.

Further applications for inline/online DLS

The flow DLS device is particularly suitable for online monitoring directly at a production line of any process involving particle dispersions, polymer solutions or emulsions of components with differing refractive index. This allows a constant quality control of such product streams. If required, the instrument can also be used as stand-alone device. It then enables quick routine analytics for academic as well as for industrial users and contract laboratories.

3 Close-up view of the inner design (scattering cell)



Flow DLS measurement during continuous synthesis of niosomes; after ca. 200s, the change of the process parameters leads to a clearly detectable increase in particle size.