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CHIMIQUE On-chip and in situ characterization of nano- to microparticles Suspensions by light extinction spectrometry

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Introduction and objectives

Microfluidics has become an essential tool in many areas, from life sciences to chemistry and environmental analysis. In chemical engineering, microfluidics improves the space-time yields, selectivity, reaction residence times and conversion performances of fundamental synthetic transformations in solution [1]. This makes possible, for instance, to synthetize different nanomaterials with controlled sizes and morphologies, for a vast number of applications [2]. Nevertheless, one of the main current difficulties of lab on chip (LoC) approaches is obtaining real-time, high-performance, quantitative information on processes at the same micrometer scale [3]. To this end, different detection and characterization systems have already been integrated in LoC devices, and among them, those based on optical transduction become of special interest due to their non-invasive character. However, they have mainly been applied for the characterization of continuous (fluid) phases or effective media [4], and their development for the characterization of disperse phases (droplets, bubbles, crystals or aggregates) is still in its infancy.

We introduce and evaluate a new LoC approach allowing for measuring particle size distributions and volume concentrations of optically dilute colloidal suspensions [5]. We have implemented the principle of light extinction spectrometry (LES, which consists in analyzing the variations of the spectral transmittance of a broadband and collimated light beam passing through the particle cloud to be characterized) [6] into a photonic lab-on-a-chip platform [4] with a multiple optical path configuration (Fig. 1, 2). In contrast to classical approaches such as DLS, this simple and effective spectrophotometric method provides absolute particle concentrations and is applicable to both stationary suspensions and dynamic particulate flows.



outlet

Spectrometer

Glass



Figure 2: (1) Glass chip in its (2) housing (a) hatch open); (3) computer controlled injection system; (4) suspension (4) inlet and (5) outlet ports; (6) incoming and (7) outgoing optical fibres; (8) CCD spectrometers (x3); (c-e) Expanded views of (9) the self-alignment channels (x6) for optical fibres; (10) 2D microlenses assemblies (x6) for collimating and controlling optical fibre outputs and inputs; (11) optical channels (x3) with path lengths L=1.0, 3.5 and 10 mm, width 650 μ m and depth 250 μ m; (12) channels endings with parallel optical-grade windows; and (13) air mirrors to avoid probing channels cross-talking during simultaneous acquisition. False colours were added in parts (b, d, e) to highlight the paths of the fluids (light blue) and probing beams (multicoloured). (f) Setup with (2) LoC housing cover removed.



On-chip LES was evaluated (Fig. 3, 4, 5) using six monodisperse particle standards (with NIST certificates) of polystyrene (nano) particles (30 nm, 50 nm, 80 nm, 0.20 μ m and 0.40 μ m), and silica (SiO2) microparticles (0.50 μ m) in aqueous media, with nominal concentrations in mass ranging from 1 to 1000 ppm. The simultaneous combination of different optical paths (Fig. 2b) permits the analysis suspensions concentration changes of up to three orders of magnitude in terms of particle concentration with minute amounts of sample. The obtained results are already promising for numerous applications in various fields of research and engineering.



Results







Figure 3. Example of LES transmissions for suspensions of silica beads of 80nm diameter, measured for two concentrations $(C_v, 5 \text{ and } 250 \text{ ppm})$ with the three optical channels (L).

Figure 4. Mean diameter measured with all channels and for all prepared suspensions versus the mean diameter estimated by SEM analyses on the source suspensions. Figure 5. Volume concentration of various suspensions vs. calculated concentrations (considering manufacturer-specified concentration of the starting suspension).

Conclusion

We introduce and evaluate a new LoC approach to measure particle sizes (from 30 nm to 0.5 µm) and volume concentrations (from 1 to 1000 ppm) of optically dilute colloidal suspensions. This simple and effective spectrophotometric approach is applicable to both stationary suspensions and dynamic particulate flows, with potential applications in various fields of research and engineering. The design of the LoC, and more especially the detection channels, could be further optimized to fit the optical properties, size and concentration of the particles (i.e. the optical thickness of the samples) and to better control the SNR of the transmittance signals. Increasing substantially the optical path length and integrating a microfluidic-controlled optical router for multiplexed analyses with a single spectrometer would also be a benefit for practical applications.

