MEAN SIZE DETERMINATION IN REAL AND CONCENTRATED LIQUID DISPERSIONS WITH S-MLS

INTRODUCTION

Nano and micro suspensions are widely used in the industry but their real dispersion state remains unknown or not well characterized in their native and concentrated form (slurries, vaccines…).
Indeed common measurement techniques may alter the dispersed phase or the apparent particle size due to the principle of measurement (microscopy, centrifugation, FFF, sieving, filtration) or the sample dilution (DLS, PTA, laser diffraction). This is especially the case for samples containing agglomerates which may breakdown because of measurement conditions such as shear stress (pumping, flowing, filtration, and centrifugation) or heavy dilutions.
Because the industry requires particles analysis in the native suspension form for product development, regulatory compliance or health and safety tests, here we propose a method to assess the mean particles size and agglomeration state in concentrated media. The measurements were done with well-calibrated polystyrene and titanium dioxide particles.

METHOD

Materials

- Well traceable polystyrene nanoparticles (supplied by an academic laboratory, 60 nm measured by DLS) are analyzed; they are directly provided in water solution with concentration range from 10^{-3} to 10 % v/v.
- Dry titanium dioxide powder (supplied by Marion Technologies, France, 570 nm measured by DLS) is dispersed in distilled water at concentrations from 10^{-3} to 10 % v/v.

Measurement with Turbiscan

Turbiscan is based on SMLS technology for Static Multiple Light Scattering and enables to measure directly the mean spherical equivalent diameter (d), knowing refractive index of continuous (n_f) and dispersed phase (n_p) and the particles concentration (φ) according to the Mie theory:

$$BS = f(\varphi, d, n_p, n_f)$$ with BS for Backscattering Intensity

This technique requires no sample preparation (i.e dilution or stress) and the sample is measured at rest in its native state. The instrument gives good repeatability of 0.5%.

Other Measurements

Comparisons with other methods like laser diffraction, Dynamic Light Scattering (DLS) and Particle Tracking Analysis (PTA) have been done but they require sample dilution and results can be obtained only at very low concentrations (under 10^{-4}% v/v).
Cryo-SEM/TEM seems an appropriate technique for non-dilution measurements. Analyses were realized by Prof. Dganit Danino (Nanotechnology Institute Technion, Haifa, Israel) in comparison to the behavior observed with Turbiscan.
However this technique requires qualification for sample preparation (which has to be done with extreme caution to avoid agglomerates breakdown), sublimation time and selection of micrograph/picture.

RESULTS

Polystyrene nanoparticles

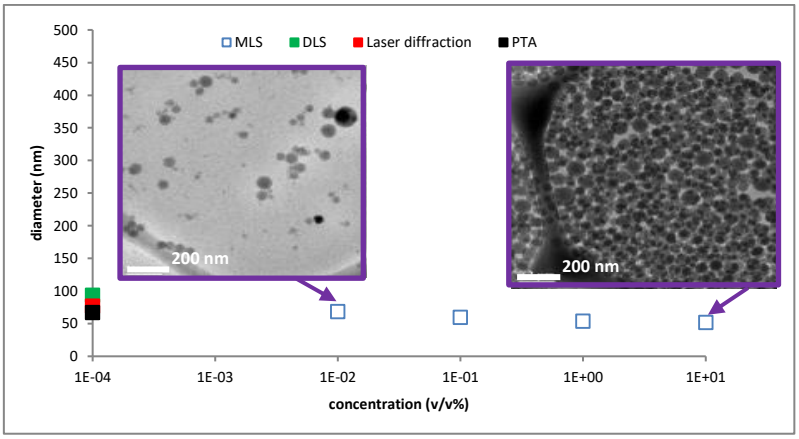

Figure 1: Mean size versus concentration (v/v) for polystyrene particles

Figure 1 shows that the size of the polystyrene nanoparticle is independent of the concentration: particles don't aggregate but remain separated and Turbiscan measurements are completely in agreement with SEM/TEM micrographs at high and low concentrations. Particle size analyses from other techniques (which are made only at very low concentration due to signal limitation) give some results in the same range of the Turbiscan data.

Titane dioxide particles

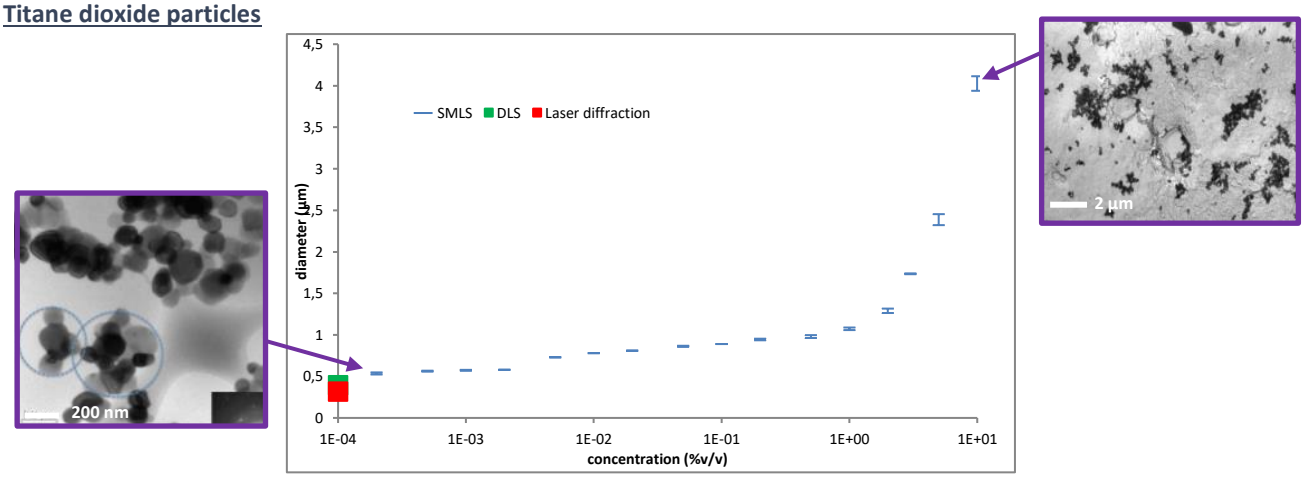

Figure 2: Mean size versus concentration (v/v) for titanium dioxide particles

Figure 2 shows that the titanium dioxide particle mean size increases with concentration, data show a good repeatability of 0.5%. Cryo-microscopy micrographs confirm these agglomeration effects with concentration increase.
S-MLS enables to analyze samples in their native state, it can monitor agglomeration effect with concentration which cannot be measured or observed with other techniques due to concentration limitation or the sample processing.

SUMMARY

A technique based on Static Multiple Light Scattering is proposed to measure mean particles size in a large range of concentration between 0.0001 and 95%, for sizes between 10 nm and 1000 µm with a repeatability of 0.5%, by Turbiscan LAB technology. This technique has the advantage to measure in one click, without sample preparation or dilution, the mean particles size particularly for concentrated suspensions. Other optical techniques such as DLS or PTA can perform this measurement but only at a very high dilution which denatures the agglomerates and give an erroneous size of the native particles. Turbiscan LAB also allows to compare samples in terms on physical stability.