DIRECT MEAN SIZE MEASUREMENT IN NATIVE CONCENTRATED DISPERSIONS WITH STATIC MULTIPLE LIGHT SCATTERING

INTRODUCTION

Nano and micro suspensions or emulsions are widely used in the industry but their real dispersion state remains unknown or not well characterized in their native and concentrated form. Indeed, it is well known that aggregation and agglomeration may exist in concentrated regime.

Static Multiple Light Scattering (S-MLS) is a well-adapted technique to determine samples mean diameter without dilution and external stress, which could modify their real state. Common measurement techniques may alter the 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.

Here we propose to use the SMLS technique principle to assess the mean particle size and to highlight the range of analysis of this method and its assets.

METHOD

Static Multiple Light Scattering technology (SMLS)

Turbiscan instrument, based on Static Multiple Light Scattering, consists in sending a light beam (880 nm) on a sample and acquiring backscattered and transmitted signals. Combining both detectors (BS & T) enables to reach a wider concentration range. The backward reflected light comes from multiple scattering as the photons are scattered several times by many different particles (or droplets).

This signal is directly linked to the mean spherical equivalent diameter (d), according to the Mie theory:

$$BS = f(\varphi, d, n_p, n_f)$$

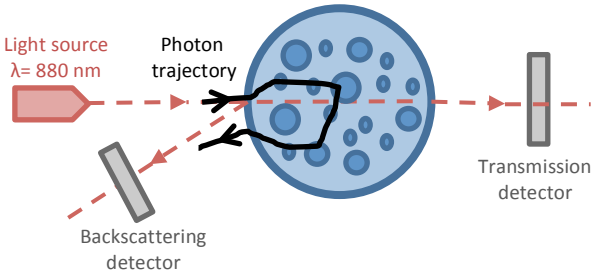


Figure 1 : Schematic representation of Turbiscan device

knowing refractive index of continuous (n_f) and dispersed phase (n_p) and the particles concentration (φ).

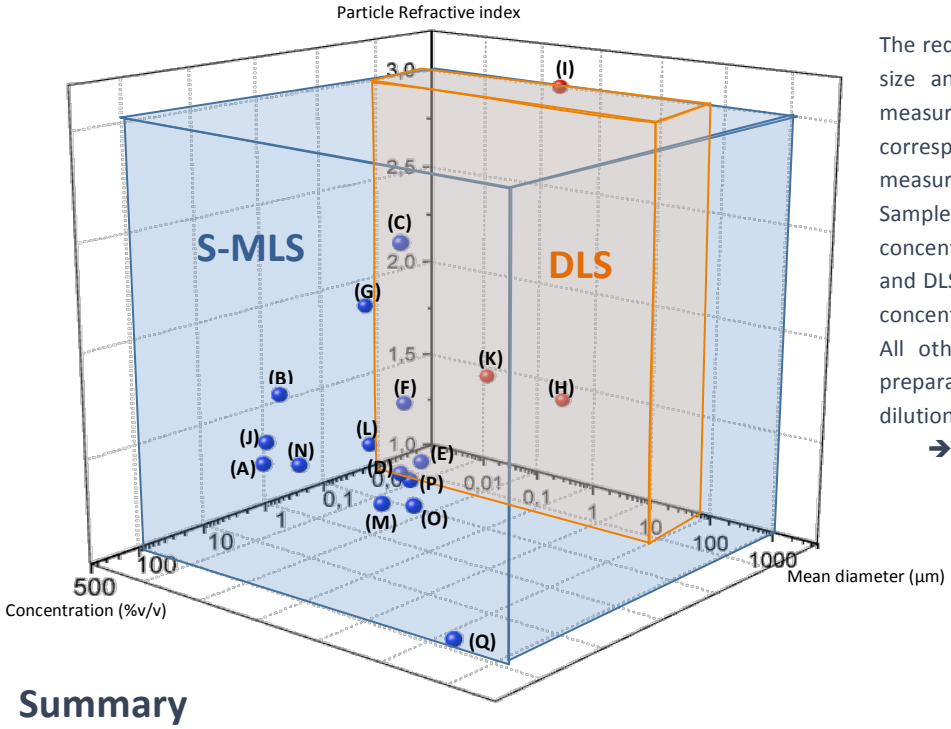
The mean diameter is given with an accuracy of +/-5% on a concentration range between 10^{-4} and 95% and for a size range between 10 nm and 1000 µm.

MATERIALS & RESULTS

A large samples panel in terms of refractive index, concentration and size was analyzed with the device. These samples correspond to industrial samples. Their characteristics are described in the table next page.

Sample	Particles	Concentration (%v/v)	Refractive index (dispersed phase)	Refractive index (continuous phase)	SMLS diameter (µm)
A	Ludox	27.8	1.48	1.33	0.050
B	Al_2O_3 liquid dispersion	11.1	1.76	1.33	0.035
C	TiO_2 powder particles	10	2.57	1.33	3.7
D	Talc powder particles	10	1.53	1.33	3.55
E	SiO_2 powder particles	1.9	1.48	1.33	1.42
F	$CaCO_3$ powder particles	0.37	1.62	1.33	0.135
G	ZnO liquid dispersion	0.18	2.01	1.33	0.013
H	SiO_2 low concentration	0.0019	1.48	1.33	0.500
I	TiO_2 powder particles	0.001	3	1.33	0.206
J	Polystyrene	29	1.59	1.33	0.060
K	Polystyrene	0.008	1.59	1.33	0.087
L	Bovine Serum Albumin	2.9	1.351	1.33	0.025
M	Healthcare emulsion	27	1.448	1.381	4.99
N	Emulsion	24	1.51	1.381	0.180
O	Emulsion with sunflower oil (surfactant sodium caseinate)	21.5	1.46	1.33	12.87
P	Emulsion with sunflower oil (surfactant tween 20)	5.4	1.46	1.33	2.7
Q	Hair foam	60	1	1.403	154.60

The figure 2 gives the data and the range of analysis that can be reached with SMLS compared to the one reached with DLS.



The red zone (DLS) corresponds to the area (of size and concentration) reachable with DLS measurements without dilution. The blue zone corresponds to the area reachable with SMLS measurements.

Samples H, I and K are in the range of size and concentration that can be reached with SMLS and DLS directly on the sample as they are low concentrated samples.

All other samples can be analyzed without preparation with SMLS whereas they require dilution to be analyzed with DLS.

➔ MLS covers a much wider area than any other techniques like DLS, PTA…

Summary

Static Multiple Light Scattering is well adapted to measure mean particles size in a large range of concentration between 10^{-4} and 95%, for sizes between 10 nm and 1000 µm, by using Turbiscan LAB technology. This technique has the advantage to measure the mean particles size in one-click, without sample preparation or dilution, particularly for concentrated suspensions, but also to quantify the sample's stability at the same time.