DISPERSIBILITY CHARACTERIZATION OF PIGMENTS & COATINGS WITH STATIC-MULTIPLE LIGHT SCATTERING

INTRODUCTION

Suspensions with nano and submicron particles are considered of great potential for various fields like pharmaceutical applications, coatings, nanocomposites materials, cosmetic...Their dispersibility, which corresponds to their property of being well distributed in term of size and concentration at a given time after a dispersion process, is of great interest as it influences final end use properties like homogeneity, stability, polishing... Because of size, surface area, concentration, zeta potential, affinity with solvent, particles may aggregate, agglomerate and sediment. Dispersibility is a multiparametric property which has to be measured on the native sample without sample preparation. Also common size measurement techniques may alter the dispersed phase or the apparent particle size due to the principle of measurement or the sample dilution.

In this note, we propose a method to assess the dispersibility ratio for two typical applications: titanium dioxide and talc.

PRINCIPLE

Measurement with Turbiscan®

Turbiscan is based on SMLS technology for Static Multiple Light Scattering and enables to measure directly the mean spherical equivalent diameter ($d_{measured}$), knowing refractive index of continuous (n_f) and dispersed phase (n_p) and the particles concentration (φ) according to the Mie theory: $BS = f\left(\varphi, d, n_p, n_f\right)$ with BS for Backscattering Intensity. This technique requires no sample preparation.

Dispersibility ratio

The dispersibility is defined as a characteristic which quantifies the ability of a solid particle to be spatially well distributed in terms of size and concentration in a liquid after controlled dispersion process to reach a state as closer as possible from the primary particle size or required size.

A quantification of the dispersibility is given with the dispersibility ratio Dr :

$$Dr = \frac{d_{theoritical}}{d_{measured}}$$

The theoretical diameter $d_{theoretical}$ corresponds to the diameter that can be obtained with the highest rate of dispersibility. The measured diameter $d_{measured}$ corresponds to the diameter measured with SMLS on the dispersion without dilution.

When Dr is close to 0, the dispersibility is bad, when Dr close to 1, the dispersibility is good and when Dr becomes greater than 1, the dispersibility enables to obtain particles better dispersed than the size given by the manufacturer (measured by DLS or TEM…).

RESULTS DISPERSIBILITY

Case 1 : Dispersion of TiO2 in water

Titanium dioxide is the most important white pigment used in the coatings industry. It is widely used for its efficiency to scatter the light for coating applications. Nevertheless, bad dispersibility can affect its performance as it will decrease its opacifying efficiency.

Two ways of dispersion have been tested to determine dispersibility of titanium dioxide powder in water: mechanical and chemical. Mechanical stirring or ultrasonication for dispersion can reduce agglomeration among particles by breaking large agglomerates into smaller ones or particles.

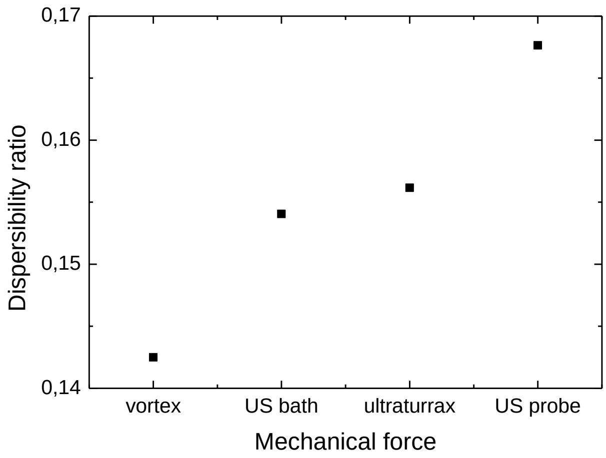

Figure 1: Dispersibility ratio *for TiO_2 particles (10% v/v) vs mechanical mixing for 30 minutes*

Figure 1 gives the Dr from the size measured with Turbiscan compared to the size given by the supplier (measured in dilute regime).

Dr shows that the dispersibility of titanium dioxide particles is influenced by mechanical force. Ultrasound probe is the most efficient to reduce agglomerates size among the methods tested here.

Another chemical way to improve particles dispersibility is to introduce a third component in the dispersion which is a surfactant. It will modify the particles surface properties from hydrophobic nature to hydrophilic surface which prevents agglomeration. The figure 2 shows Dr for TiO2 with different CTAB concentration.

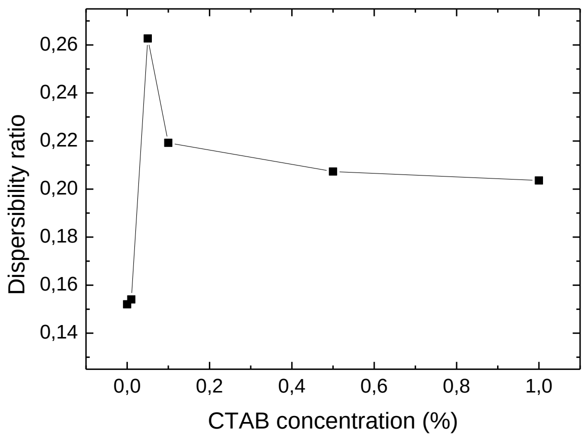

Figure 2: *Dispersibility ratio Dr for titanium dioxide with CTAB*

Figure 2 shows that CTAB surfactant enables to reach better dispersibility ratios than mechanical mixing and so increases titanium dioxide dispersibility in water. A surfactant concentration optimum is reached close to 0.05% corresponding to surfactant CMC.

Case 2 : Dispersant agent to improve talc dispersibility

Introduction of talc powder in paint contributes to hiding power and whiteness effect, in combination with pigments. Talc supplied by Mondo Minerals was dispersed at 30%wt in water, two different dispersant agents were added to the mixture to reduce talc agglomeration. Dr is calculated for both dispersions, the theoritical diameter taken for the calculation is the size d50 given by the supplier measured with Sedigraph.

Figure 3 shows that adding a dispersant enables to reach Dr greater than one so sizes lower than the theoritical one measured on the talc alone, so the dispersants enable to disperse agglomerates on the native talc. Dispersant B is better agent than A to eliminate agglomerate formation and increasing dispersant concentration enables also to improve dispersibility.

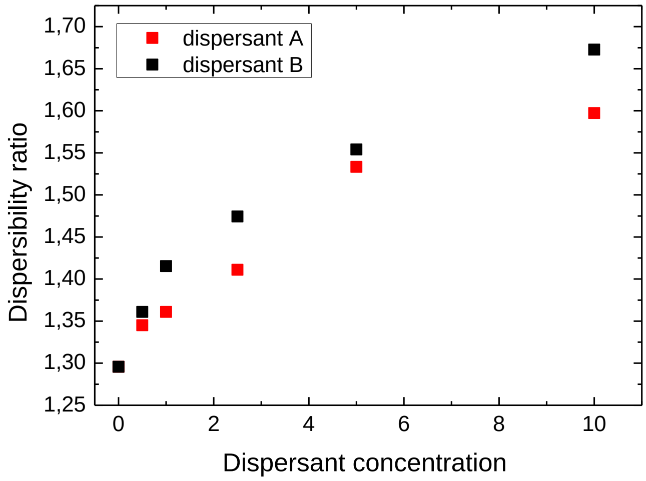

Figure 3: *Dispersibility ratio Dr for talc with two different dispersant agents at different concentrations*

SUMMARY

Turbiscan LAB technology based on Static Multiple Light Scattering (SMLS) is proposed to determine dispersibility of solid component in a liquid. As seen in the examples presented in this note, mechanical action, surfactant or dispersant can improve the dispersibility. This technique enables to work in a large range of concentration between 0.0001 and 95%, for sizes between 10 nm and 1000 µm. This technique has the advantage to measure in one click, without sample preparation or dilution, the mean particles size particularly for concentrated suspensions and provide to users a level of dispersibility.