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, and cosmetics...

Their dispersibility, which corresponds to their property of being well distributed in terms 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, or any other parameter, particles may aggregate, agglomerate and sediment.

Dispersibility is a multiparametric property, which has to be measured on the native sample, without sample preparation. 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 for two typical applications: titanium dioxide and talcum powder.

PRINCIPLE

Measurement with Turbiscan®

Turbiscan is based on SMLS technology (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: $BS = f(\varphi, d, n_p, n_f)$ 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 a controlled dispersion process, to reach a state as close as possible to 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 is 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 (which is often measured by DLS or TEM...).

DISPERSIBILITY

Case 1 - Dispersion of TiO_2 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 effect.

Two different 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 even primary particles.

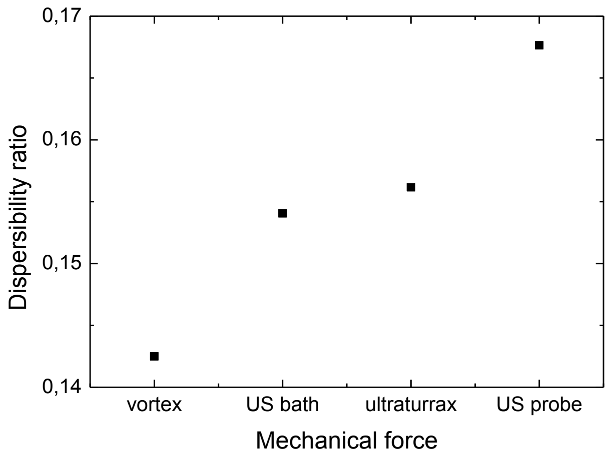

Figure 1: Dispersibility ratio for TiO_2 particles (10% v/v) vs mechanical mixing during 30 minutes

Figure 1 gives the Dr from the size measured with Turbiscan compared to the size given by the supplier (measured in dilute regime).
These results show that the mechanical force influences the dispersibility of titanium dioxide particles. Ultrasound probe is the most efficient way to reduce agglomerates size among the methods tested in this work.

Another chemical way to improve particles dispersibility is to introduce a third component in the dispersion, which is a surfactant. It will change the particles surface properties from hydrophobic to hydrophilic, preventing agglomeration. The Figure 2 shows Dr for TiO_2 with different CTAB concentration.

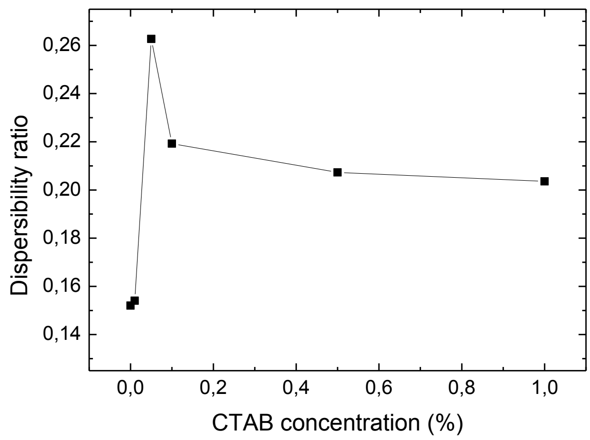

Figure 2: Dispersibility ratio Dr for titanium dioxide with CTAB

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

Case 2 - Dispersant agent to improve talcum powder dispersibility

Introduction of talcum powder in paint contributes to hiding power and whitening effect, in combination with pigments. Talcum powder supplied by Mondo Minerals was dispersed at 30%wt in water, two different dispersant agents were added to the mixture to reduce talcum agglomeration. Dr is calculated for both dispersions, the theoritical diameter taken for the calculation is the d_{50} given by the supplier, measured with Sedigraph.
Figure 3 shows that adding a dispersant enables to reach Dr greater than 1, meaning sizes lower than the theoritical one measured on the talcum powder alone, so the dispersants enable to disperse agglomerates on the native talcum. Dispersant B is a better agent than A to reduce agglomerate formation and increasing dispersant concentration also enables to improve dispersibility.

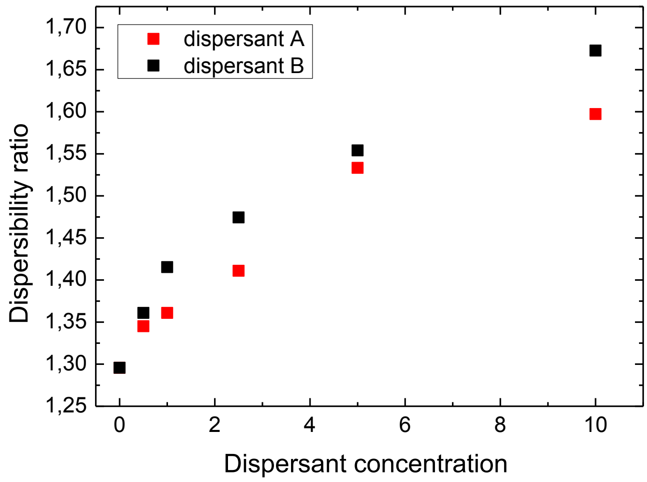

Figure 3: Dispersibility ratio Dr for talc with two different dispersant agents at different concentrations

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 10^{-4} and 95%, for sizes between 10 nm and 1000 µm. This technique has the advantage to measure the mean particles size in one-click, without sample preparation or dilution, particularly for concentrated suspensions and provide to users a level of dispersibility.