TURBISCAN DNS

Raw material optimization (Part 2)
automated dispersant screening

INTRODUCTION

Suspensions are solid particle dispersed into a liquid phase. Due to the high energy of contact, dispersing solid particles remains a challenge and it is key to ensure final properties of the formulation. To ensure a good dispersibility, additives (known as dispersant) can be added to keep particles individually dispersed in liquid rather than allow agglomerates to form. Many questions need to be answered: Which dispersant is the most suited? What is the optimum concentration to ensure the best performance/cost ratio? What is the shelf life of the final formulation?
The Turbiscan®, the leading technology to measure the dispersion stability & shelf life, helps formulators in the decision-making process thanks to its “online” capabilities. This application papers shows how the Turbiscan® DNS with the TMIX, can help select the most appropriate dispersant for a TiO2 suspension and to reach the desired particle size.

ONLINE | **NO DILUTION** | **FAST**

HOW IT WORKS

Turbiscan technology, based on Static Multiple light scattering (SMLS), consists on illuminating a samplewith an infrared light s ource and acquiring backscattered (BS) and Transmitted (T) signals.

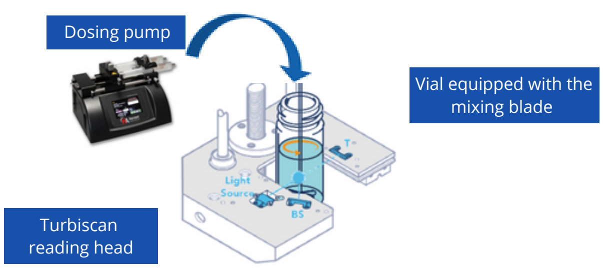
BS and T = f(φ, d, np, nf)

The signal is directly linked to the particle’s concentration (φ) and size (***d***) according to the Mie Theory, with refractive index of continuous (***nf***) and dispersed phase (***np***) being fixed parameters. The measurement of the BS and T can be performed either on scanning mode, to provide homogeneity and stability measurement, or with very high frequency for fast time resolved and online measurement.

The measurements are done without any dilution & on native sample.

Additionally, the Turbiscan DNS associates 2 functions for online characterization of the dispersion state and the dispersibility:

- Mixing function (TMIX) for automated fast formulation screening with a stirring bar directly inside the measurement cell. **Topic of this note.**
- Circulation function (TLOOP) for online measurements, scale up and process optimizations.



measurement of the backscattering and the transmission during mixing with the TMIX function

EXPERIMENT AND METHOD SET UP

TiO2 particles are prepared in pure water* at 0.5wt% and the dispersion is then placed in the Turbiscan® measurement cells equipped with TMIX function. Dispersant is automatically added to the dispersion using a syringe pump for controlled concentration
screening. The process takes place under agitation at 2000 rpm.

Formulation screening:

3 dispersants have been selected (SDS, CTAB and Decylglucoside) with the aim to screen the concentration from 0% to 1.2%. The concentration of the dispersant is applied automatically via autonomous syringe pump. Thus, the measurement is fully automatic and only lasts 30 min per dispersant.

The measurements of BS and T are done:

- **Under agitation** and while changing the dispersant concentration.
- At **high frequency** (10 measurements/second) thanks to **T-Fast** software.
- Directly on the **native sample**, avoiding tedious and long sampling process present with other particle size technologies.

* the application note TDNS-01 explains how the Turbiscan® can be used to screen and select the most suitable raw materials.

RESULTS

The graph 1 below presents the evolution of the mean particle size, measurement by SMLS in function of the dispersant concentration and under agitation.

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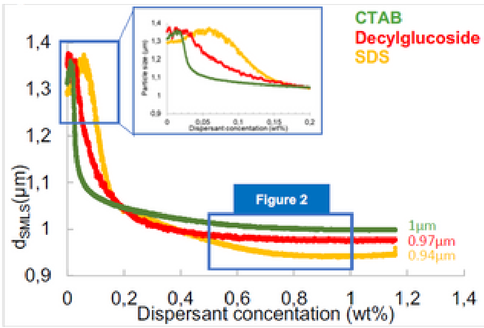

Figure 1. Particle size (µm) as a function of dispersant concentration %wt.

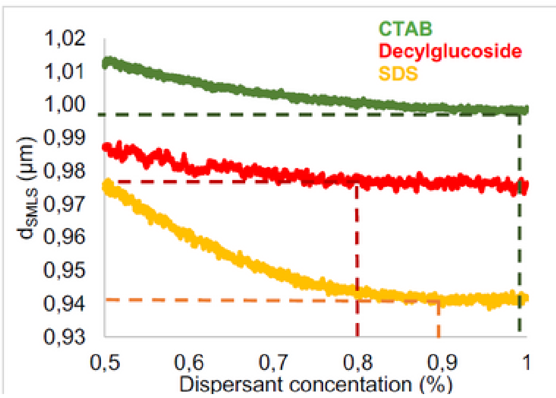

Figure 2. Zoom on particle size (µm) as a function of dispersant concentration (%wt) with optimal concentration.

Such study helps formulators select the most effective dispersant and optimize the cost vs performance ratio. SDS can be considered as the best dispersant for this TiO2 particles, as it results in lowest particle size. However, SDS outperforms at higher concentrations (highest efficiency = 0.9%). which may have an impact production costs and even on other properties.

In the case of limiting the concentration is priority, for cost or any other viable reasons, CTAB can be considered for lower concentrations (below 0.2%). In this last configuration, the impact on the final particle can be somehow acceptable with a final particle size of 1.1µm compared to 0.94µm with the SDS at 0,9% wt.

Dispersant	Dr	D-SMLS (µm)	[Optimal Dispersant] (%)
SDS	0.32	0.94	0.9
DG	0.31	0.98	0.8
CTAB	0.30	1.00	1.0

Table 2. Dispersibility ratio (theoretical size = 300nm), particle size (D-SMLS) and optimal dispersant concentration.

By providing fast and easy answer based on reliable and quantified data, the Turbiscan® and the T-MIX module provide a unique solution to understand the dispersion quality. Formulators and chemists can take fast decision on how to optimize formulation with fast screening directly in the measurement cell. The method ensures the shortest time-to-market.

CONCLUSION

Saves time

Simply weight, stir, and measure directly the native sample without any preparation

No dilution and on native sample

Other techniques require a high level of sample preparation, dilution or additional forces and their impact is significant on particle size.

All in one

Once the correct raw material is selected to achieve the desired particle size, the Turbiscan technology can also help selecting the correct dispersant agent, adjust process parameters and stability measurement.