



DISPERSIBILITY – CASE STUDY

RAW MATERIAL EFFICIENT SCREENING

Context

Dispersibility, the ease of dispersing particles into liquids with a minimum size, is crucial for formulas' end-use properties. Indeed, this operation could impact the final suspension quality like paint opacity, drug delivery, and shelf-life... Dispersibility depends on formulation parameters: raw materials, formulation, and the dispersing process. Methods for dispersibility characterization usually require strong sample modification (dilution, sampling, external stress), making studies long, tedious, and not always reliable.

In this note, a method is presented to screen the dispersibility of different raw materials and their affinity within the solvent using the TURBISCAN DNS online capabilities.

What is dispersibility?

According to the Technical Specification ISO/TS 22107:2021 entitled "Dispersibility of solid particles into a liquid", here is a general definition established for dispersibility:

"When the raw particulate material is a powder, "dispersibility" is often used to indicate the ease of bringing a powder into a dispersion by achieving uniform spatial particle distribution and, if, aimed "deagglomeration".

The ISO/TS describes the process to disperse powders:

- First, the introduction of particles into the media "wet the powder (source material) with the liquid (continuous phase)"
- Then, product homogeneity "obtain a uniform distribution of mass throughout the liquid volume (primarily for mixing, not size reduction)"
- Finally, "decrease (reduce) the size of agglomerates of the source material to the application specific criteria for size or size distribution, or down to constituent particles or primary particles if desired".

To summarize, for a specific formulation with defined experimental conditions, dispersibility is divided into 2 processes: homogeneous distribution of the particles (DISTRIBUTIVE process) with the smallest size (DISRUPTIVE process).

Dispersibility should not be confused with shelf-life, the ability of the dispersion to present acceptable variation over a desired amount of time.

How do we measure dispersibility?

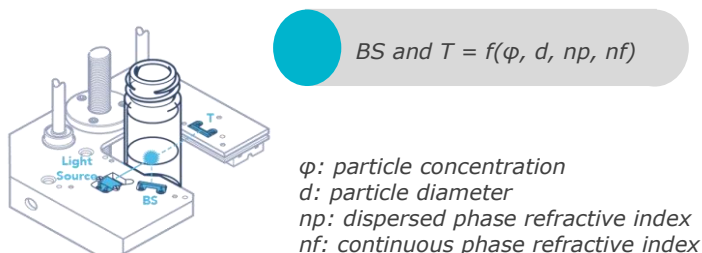
The ISO/TS To 22107:2021 also gives recommendations:

- Sample must be measured in native state "dilution, mixing,... or any other chemical treatment are deprecated since they may induce changes in the dispersion state".
- A direct reading and online measurement are suitable hence the "time of analysis following sample preparation, which shall be conducted during or immediately after processing" i.e.: an online measurement is a perfect match.

The TURBISCAN DNS fulfills all these recommendations thanks to the **native** and **online** characterization of the dispersion state via **Static Multiple Light Scattering** technology (SMLS).

TURBISCAN: How it works

TURBISCAN technology, based on Static Multiple light scattering (SMLS), consists of illuminating a sample with an infrared light source and acquiring Backscattered (BS) and Transmitted (T) signals.



The signal is directly linked to particle concentration (φ) and size (d) according to the Mie

Theory. Measurements can be performed at high frequency for fast time-resolved and online dispersibility measurement, or on scanning mode to provide homogeneity for stability measurement. Samples are studied in the native state up to 95% V/V and for particle size measurement from 10nm up to 1mm.

The **TURBISCAN DNS** for **Dispersibility** and **Stability** is composed of 2 modules allowing the online measurement for dispersibility study.

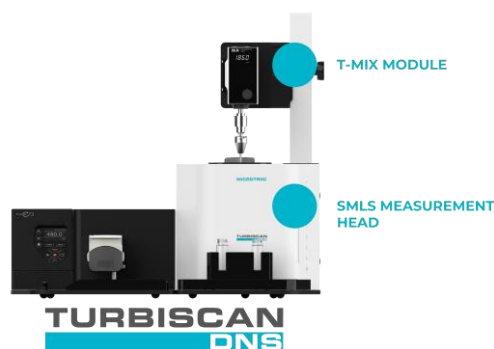


Figure 1. TURBISCAN DNS

- **T-MIX:** measure particle dispersion directly inside the measurement vial with a stirring blade adapted inside (mixing speed up to 2000 rpm).
- **T-LOOP:** measure online dispersibility from your process with a peristaltic pump creating a Loop and/or optimizing your dispersion process.

The following illustration shows the measurement principle: SMLS technology combined with the T-LOOP module.

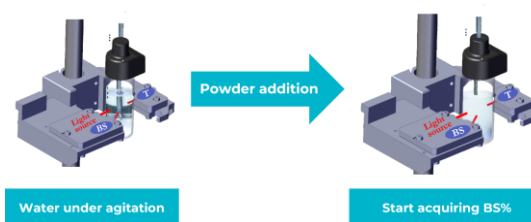


Figure 2. Illustration of SMLS technology combined with T-MIX module

Results

The final quality of the dispersion mostly results from the deagglomeration/ emulsification process occurring in the initial dispersion state. During this step, particles face shear energy to reach the smallest particle size.

Formulation scientists usually select the process parameters and the formulation to reach the smallest particle size hence the best dispersibility. While the process must provide enough energy to de-agglomerate the particles, the formulation will help to minimize the particle-solvent interaction energy and prevent the re-agglomeration of the particles.

Which particle type or additives are the most suitable for the formulation?

For this study, three different TiO₂ were studied with the TURBISCAN DNS. Measurements were performed with the T-MIX module. First, 15 ml of deionized water was added into a glass vial, while mixing at 900 rpm, 1% (w/w) of TiO₂ powder (rutile pigment) was added and measured for 4 minutes.

TiO₂ characteristics:

	TiO ₂ surface treatment	Density (g/cm ³)
TiO ₂ _1	Al/Si	3.9
TiO ₂ _2	Al/Zr	4.1
TiO ₂ _3	None	4.2

Figure 3. Characteristics of the 3 studied TiO₂

The following graphic represents the mean particle size evolution measured online during mixing for 4 minutes. Using this configuration, the TURBISCAN DNS can make up to 10 measurements per second, ideal for precise kinetics.

After 1 minute, the final particle size is nearly obtained for the three samples highlighted with the fast and kinetic measurement. The initial

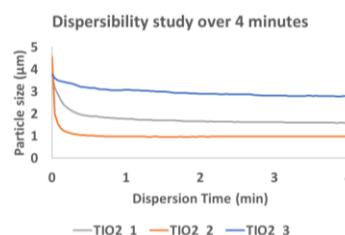
particle size is the highest for TiO₂_2 (4.6µm) but decreases the fastest to reach the smallest final

Particle size (1.32µm). Surface treatment of TiO₂_2 should have the best match with the solvent used (Water) compared to TiO₂_1 and TiO₂_3.

	Final mean diameter(µm)
TiO ₂ _1	3.9
TiO ₂ _2	4.1
TiO ₂ _3	4.2

Figure 4. Particle dispersibility study over 4 minutes at 900rpm

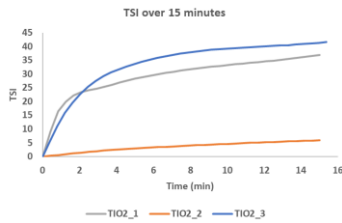
Note: the three TiO₂ powders are given for 300nm. To reach the primary particle size, the addition of a dispersant or a higher energy/shear process is necessary.



Right after the dispersibility study (after 4 min of mixing), suspension stability was measured. The T and BS values were recorded all over the sample height without any further preparation. Sedimentation and clarification phenomena were observed. To compare and rank sample variations, the TSI (Turbiscan Stability Index) was used. TSI algorithm sums all the variations all over the sample's height. **The higher the TSI, the more evolved the sample therefore destabilized.** The following figure presents the TSI kinetics with the diameter of settling particles.

	Migrating particle diameter(μm)
TiO ₂ _1	3.9
TiO ₂ _2	4.1
TiO ₂ _3	4.2

Figure 5. Suspension destabilization over 15 minutes



In addition to the smallest final particle size, a formulation made with the TiO₂_2 evolves the less (smallest TSI value over 15 minutes) and, hence is the most stable.

However, for samples 3 and 1, particles tend to agglomerate especially for 3. With agglomeration and denser particles, sedimentation is faster (higher TSI values).

Conclusion

With the study of both dispersibility and stability, the TURBISCAN DNS provides unique information for fast screening and a clear answer for raw materials validation towards dispersibility and stability with a single instrument. TURBISCAN DNS: a unique platform for Dispersibility 'n Stability



TURBISCAN RANGE

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