

## INTRODUCTION

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, the 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 dispersibility of different raw materials and their affinity within the solvent using the Turbiscan® DNS online capabilities.

Raw Material

Screening

Online



## 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, 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 to measure dispersibility?

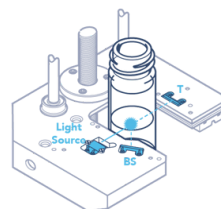
the ISO/TS To 22107:2021 also gives recommendations :

- 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 is 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 **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.



$$BS \text{ and } T = f(\varphi, d, n_p, n_f)$$

$\varphi$ : particle concentration  
 $d$ : particle diameter  
 $n_p$ : dispersed phase refractive index  
 $n_f$ : continuous phase refractive index

The signal is directly linked to particles concentration ( $\varphi$ ) 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 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-MIX module.

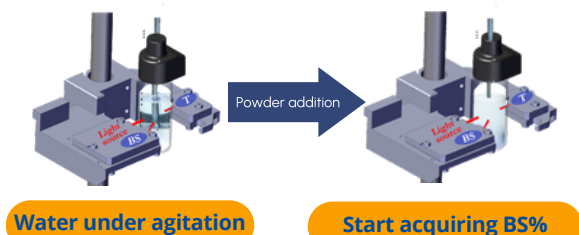


Figure 2. Illustration of SMLS technology combined with T-MIX module

## RESULTS

The final quality of the dispersion mostly results from the de-agglomeration/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 TiO<sub>2</sub> 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 TiO<sub>2</sub> powder (rutile pigment) was added and measured for 4 minutes.

TiO<sub>2</sub> characteristics :

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

Figure 3. Characteristics of the 3 studied TiO<sub>2</sub>

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 measurement 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 TiO<sub>2</sub>\_2 (4.6µm) but decreases the fastest to reach the smallest final particle size (1.32µm). Surface treatment of TiO<sub>2</sub>\_2 should have the best match with the solvent used (Water) compared to TiO<sub>2</sub>\_1 and TiO<sub>2</sub>\_3.

Note: the three TiO<sub>2</sub> powders are given for 300nm. To reach the primary particle size, addition of dispersant (**TDNS\_02**) or higher energy/shear process are necessary (**TDNS\_12**).

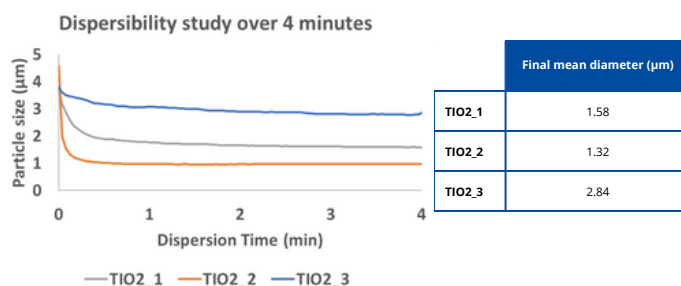


Figure 3. Particle dispersibility study over 4 minutes at 900 rpm

Right after the dispersibility study (after 4 min of mixing), suspension stability was measured. The T and BS values were recorded all over the samples height without any further preparation. Sedimentation and clarification phenomena were observed (See AN....). To compare and rank sample variations, the TSI (Turbiscan Stability Index) was used. TSI algorithm sums all the variations all over the samples height.

**The higher the TSI, the most has evolved the sample therefore destabilized.** The following figure presents the TSI kinetics with the diameter of settling particles.

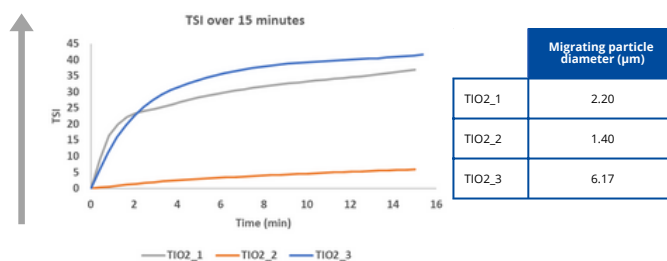


Figure 4. Suspensions destabilization over 15 minutes

In addition to the smallest final particle size, formulation made with the TiO<sub>2</sub>\_2 evolves the less (smallest TSI value over 15 minutes), 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 clear answer for raw materials validation towards dispersibility and stability with a single instrument.

Turbiscan DNS : unique platform for Dispersibility 'n Stability