# TURBISCAN DNS

# Dispersibility - Case Study
## Milling process optimization

## INTRODUCTION

Dispersibility, the ease of dispersing particles into liquids with a minimum size, is crucial for formulations 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 a first note **(TDNS_11_Dispersibility_Raw Material Efficient Screening)**, dispersion conditions were studied. In this second note, the process conditions to obtain the desired particle size are studied online and without any dilution using the Tubiscan® DNS.

**Process** **Online** **Particle Size**

## 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?

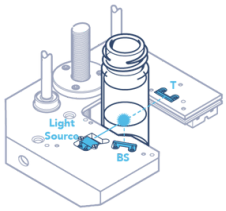
the ISO/TS To 22107:2021 also gives recommendations :

- 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 recomandations 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 and T = f(φ, d, np, nf)*

*φ: particle concentration*
*d: particle diameter*
*np: dispersed phase refractive index*
*nf: continuous phase refracrive index*

The signal is directly linked to particles 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 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.

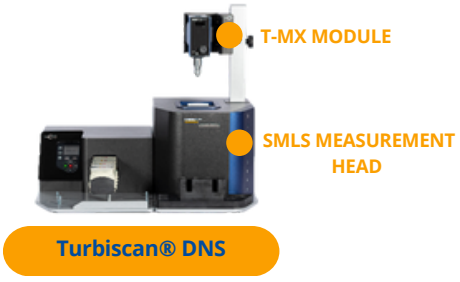



Figure 1. TURBISCAN® DNS

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- **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 peristatic 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.

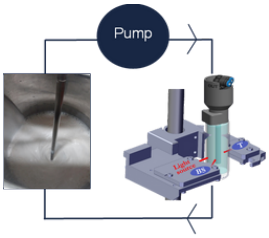


Figure 2. Illustration of SMLS technology combined with T-LOOP module

## RESULTS

The final quality of the dispersion mostly results on the de-agglomeration/emulsification process occuring in the initial dispersion state. During this steps, 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.

### Wet Grinding time

For this study, two particles are studied under wet milling process: 20% wt of calcium carbonate in water and 13% wt sodium bicarbonate in ethanol. Wet milling presents many benefits for both raw material providers and formulation scientists to get high-quality, finely milled raw materials. The wet grinding unit can be directly connected to the T-LOOP module to follow particle size during the grinding process without any dilution. The following illustration shows the measurement principle: SMLS technology combined with the T-LOOP module and a Wet Grinding Unit.

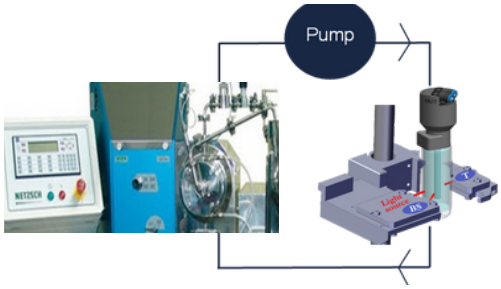


Figure 3. Illustration of SMLS technology combined with the T-LOOP and a Wet Grinding Unit

The following figures show the mean diameter while milling for the calcium carbonate in nm (on the left) and for the sodium bicarbonate in µm (on the right).

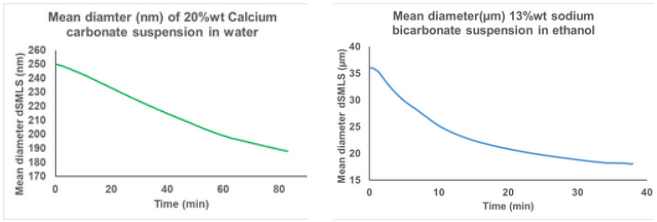


Figure 4. Mean diameter over time while wet grindind

In both cases, particle size decrease is not completed over milling time (no plateau observed). However, thanks to the online measurement, the milling can be stopped if the desired particle size is obtained or the process can be optimized to reach this target.

### How to deagglomerate particles with Ultrasounds ?

The mean particle size of 0.1wt of silica nanoparticles (fumed silica) in suspension was studied by varying the ultrasound amplitude (20%, 30%, 40%).

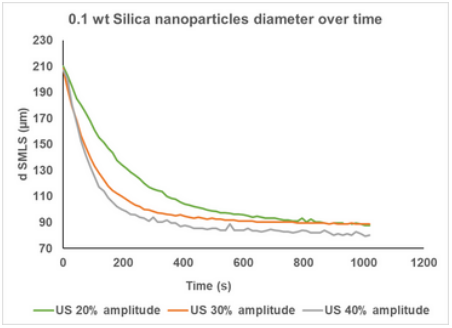


Figure 5. Mean diameter over time by varying ultrasound amplitude

Based on these results, increasing ultrasound amplitude enables to increase the de-agglomeration rate and reach a plateau earlier (400s for 40%, 600s for 30% and 800s for 20%). For 20% and 30% the same final particle size is reached with a faster kinetic for 30%.
40% amplitude is more efficient and enables to reach the smallest final particle size (80.3 µm).

**CONCLUSION**

With the study of online particle size during the dispersing process, the Turbiscan DNS provides unique information for fast optimum process definition in a single measurement using a single instrument. Turbiscan DNS : unique plateform for Dispersibility 'n Stability


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