TURBISCAN DNS

Measuring resuspending properties
Another route against sedimentation

INTRODUCTION

Sedimentation of solid particles is a very well know phenomena and several different strategies are available to avoid or slow down particle settling (reducing particle size, optimizing viscosity, adding better dispersants). Another route to counteract the sedimentation is to resuspend already settled dispersion. This gives the user the possibility to refresh the formulation by adding energy (manual or giving stirring speed) and thus extend the period of formulation's use. The esuspending process should however be studied and optimized. Thus, many questions are to be asked: How to measure the resuspending capabilities? Is manual shaking enough? Can the same dispersion state be achieved? This paper provides a methodology to study resuspending capabilities via the Turbiscan DNS.

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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.

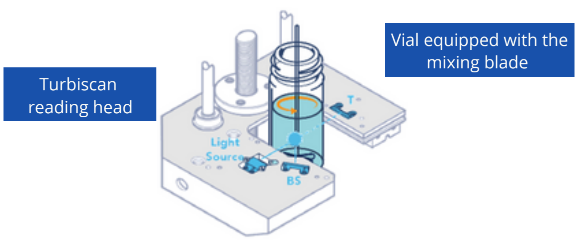
BS and T = f(φ, d, np, nf)

The signal is directly linked to the particle's concentration (φ) and size ( ) according to the Mie Theory, with refractive index of continuous ( ) and dispersed phase ( ) 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 associated with 2 function for online characterization of the dispersion state and 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 optimization.



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

EXPERIMENT AND METHOD SET UP

In the application notes TDNS_02 and TS_64, the Turbiscan was used to determine the best dispersant between SDS, CTAB and DG for a TiO2 powder dispersion in water at 0.5wt%. Both the ability to disperse the particles and the final stability of the suspension were studied. The initial particle size (measured directly during the mixing with TMIX function) and stability particle size (measured after agitation is stopped and sedimentation is observed) were compared. The table 1 summarizes the results of this studies.

		Particle Size (µm)	
Sample	**Optimal Dispersion conc. [%]**	**Initial dSMLS**	**After Mixing dSTAB**
DG	0.9	0.98	1.00
CTAB	0.8	1.02	2.00
SDS	1.0	0.94	25.10

Table 1: Optimal concentration for minimum size for studied dispersants.

Despite similar dispersibility capabilities of CTAB, SDS, and DG, it was found that particles start agglomerating during sedimentation (TS_STAB_64) in the case of SDS and CTAB (table 1). While all 3 dispersants are good candidates to disperse TiO2 in water, DG is a better candidate as stabilizing agent as it kept similar particle size during sedimentation.

For resuspending studies of TiO2, the same samples are left for 7 days at rest and at room temperature and retested with TMIX function. The use of this function ensures reproducible mixing method and direct measurement of the resuspending capabilities. The mixing speed is set at 2000rpm for 5 min.

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To study the resuspending ability, the level of BS right after the resuspending process is compared to the BS obtained on the fresh sample via the Redispersion ratio (see below.

$$\text{Redispersion ratio} = \frac{\text{BS (fresh)}}{\text{BS (redispersed)}}$$

A perfect resuspending ability would give a Redispersion Ratio of 100%. Any deviations to this value would mean a different dispersion state hence a lower resuspending property. The measurement is done without any sampling and directly in the sample cell thanks to the T-MIX module that allows to control applied energy.

RESULTS

The figure 1 below represents the redispersion ratio.

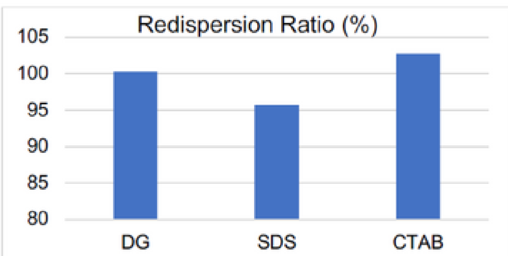

Figure 1. Redispersion quality of 7-days old samples expressed as ratio between BS of fresh sample and redispersed sample.

Globally, the redispersion of the samples are good (between 95% and 102%) meaning that with the mixing protocol it is still possible to retrieve similar dispersion state based on the 7 days of storage.

Once samples are resuspended, the mixing is stopped and the Turbiscan is used in "scan mode" to study the settling properties once again. In the scan mode, the Turbiscan acquired T and BS all over sample height, repeatedly and for 10 hours. As previously observed (TDNS_03) a large clarification is observed and the kinetics of the evolution of clarified phase thickness is calculated and compared to the initial kinetics (figure 2).

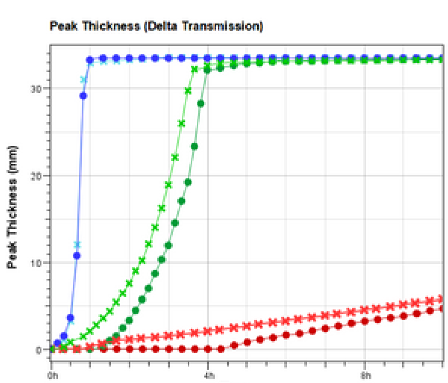

Figure 2. Peak thickness of freshly prepared sample (●) and redispersed settled samples (×)

Despite a redispersion ratio close to 100%, i.e. a similar dispersion state compared to the fresh suspension, the sedimentation kinetics are slightly different for DG and CTAB once resuspended. This can be due to a double mechanism. Firstly, the resuspension is less effective in breaking down all agglomerates/aggregates formed in the sedimentation cake over the 7-day period. Secondly, the surfactant cannot prevent agglomeration after the second mixing, which is shown by bigger particle sizes (calculated with Stokes equation).

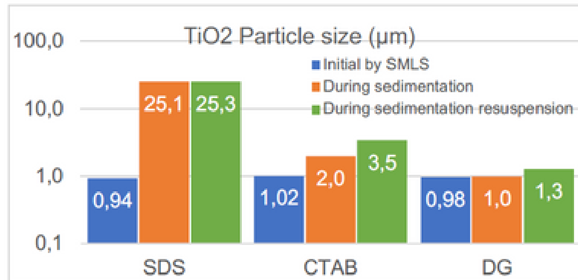

Figure 3. Particle size by SMLS **(dSMLS)** of freshly prepared suspension and the Stokes diameter during sedimentation **(dSTAB)** right after preparation and after resuspension **(dSTAB).**

Studying the resuspending ability of suspension can be a new route to ensure the perfect dispersion in the eyes of the user with a simple 'shake before use' label with limiting additive quantities.

CONCLUSION

The Turbiscan technology has been used for decades for fast and quantitative stability studies. The T-MIX module offers the ability to continue the nvestigation beyond stability studies, toward resuspending properties and extending the period of use. The measurements are done in native sample, without dilution and thanks to the T-MIX module, the Turbiscan provide a reproducible and robust mixing method to study the resuspending capabilities.