TURBISCAN DNS

Resuspension
Reversible destabilization study

INTRODUCTION

To guarantee performances, many industrial products need to be refreshed or "well shaken" before use. This process is fundamental for many application : coverage in paint, drug dosage, taste of beverages, pesticide dose.... Product resuspension can be a requirement or a just a smart and easy way to extend shelf-life. Method to study this specific ability does not really exist or mostly depends on manual shaking and visual observation leading to low representativity and reproducibility. How to efficiently measure resuspension and sample refreshment ability ?

This note presents a precise monitoring of the resuspension ability with the Turbiscan DNS.

Mixing **Resuspension** **Reversibility**

DEFINITION : Resuspending ability

In some cases, particle migration over recommended storage conditions cannot be overcome and phase separation induced by sedimentation or creaming will occur (paint, inks, vaccines and injectables, beverages...). These phenomena are reversible under some conditions and most commonly by adding energy over a certain period (manual shaking, mechanical stirring...). The challenge is to ensure, once the process is applied, a fully homogeneous dispersion i.e there are no concentration difference between the top and the bottom of the container and no presence of agglomerates or aggregated particles. If so, then initial performances and properties are fully recovered like paint coverage, drug dosage, drink taste...

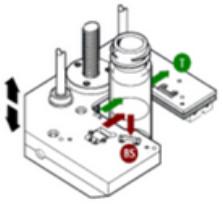
The resuspending ability depends on multiple factors such as particle-particle interactions, cohesion energy, packing of the sediment... Visual observation method gives only an idea about homogeneity but cannot precisely monitor the resuspension capability of a formulation. Particle size measurement can give indication, however, the dispersion should be considered in its native state and this techniques gives no information about the kinetic.

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 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 high frequency for fast time resolved and online measurement.

The measurements are done without any dilution & on native sample up to 95% V/V and from 10nm up to 1mm.

BS and T = f(φ, d, np, nf)

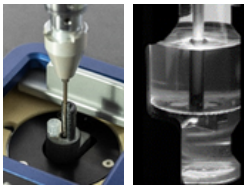


The Turbiscan® DNS ,for Dispersibility and Stability, is composed of 2 modules allowing online measurement of the dispersion state to follow the reconstitution process.

- **T-MIX** *(used for this study)* for mixing and fast formulation screening with a stirring bar directly placed inside the measurement cell. The mixing speed goes up to 2000 rpm.

- **T-LOOP** for online measurements and scale up/ process optimizations.

Turbiscan® DnS



The T-Mix Module

Resuspension
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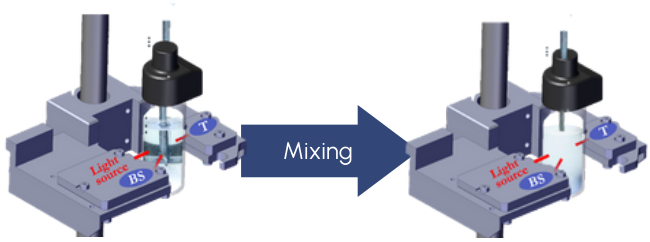
CASE STUDY : RESUSPENDING

As a case study, three white paints were tested. Products were directly poured into measurement cells without any dilution. Paints destabilization over time was studied with the Turbiscan for 7 days at 40°C and sedimentation is easily detected (see note TS_03)

The resuspension study consists of applying a precise and reproducible mixing energy and to monitor the dispersion state in function of time (backscattering level). Indeed, the backscattering level relates on particle size and particle concentration, therefore comparing the backscattering value during mixing to the initial backscattering level reflects to the level of recovery of the dispersion. To study the kinetics, and so the ease of resuspension, the measurement is performed while mixing using the T-MIX function of the Turbiscan DNS. The stirring is done directly inside the measurement cell for a precise and reproductible resuspension process to compare resuspending ability of the different products. The stirring bar was placed in the middle of the cells and the mixing speed set at 800 rpm for a vigorous mixing for 35 minutes.



Phase separation **Start acquiring BS%**

RESULTS

As explained above, to evaluate redispersion quality, the initial BS level is compared to the BS level obtained during the resuspension process. The ease of redispersion, in other words the recovery rate is therefore defined as:

$$Recovery\ rate = \frac{BS_{(redispersed)}}{BS_{(initial)}} \times 100$$

A perfect resuspending ability would give a recovery rate of 100%. Any deviation to this value would mean a different dispersion state hence a lower resuspending property. Thanks to the Turbiscan DNS, the BS is measured during mixing and recovery rate can be calculated as a function of time for a better understanding of the kinetic and final dispersion state.

The recovery rates in function of the mixing time (during 35 minutes) are display on figure 1.

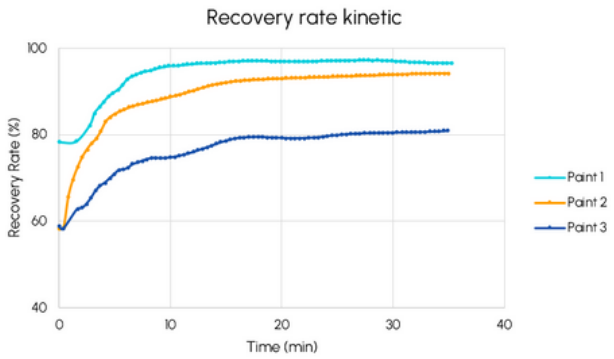


Figure 1. Recovery rate (%) in function of time (min) for the 3 paints

Paint 1 shows the better resuspension ability. Not only reaching a recovery rate > 95%, its recovery is much faster compared to paint 2 and 3. On the other hand, paint 3 shows the worst redispersion ability with a poor recovery rate (approx 80%) and a much slower kinetic. This "bad" resuspension ability can come from a hard settle or caking of the sediment and may have a significant impact on the quality of the final suspension.

The table below summarizes the information from the kinetic with the final recovery rate obtained after 35 min and the time to reach 90% of the final recovery rate (t90).

Sample	Final Recovery rate%	t90
Paint 1	97%	4 min
Paint 2	94%	5 min
Paint 3	84%	11min

Table : time to reach 90% of recovery rate for the 3 paints

CONCLUSION

Turbiscan® technology based on Static Multiple Light Scattering (SMLS) is the leading technology since 1994 to measure stability of formulations and dispersions. The Turbiscan DNS offers extra routes of investigation with resuspending studies thanks to an online measurement of the dispersion state and a reproducible method.

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