

DIRECT EMULSIFICATION SCREENING FAST METHOD & SMALL VOLUME

Introduction

Stabilizers and surfactants play a key role in assuring desired product shelf life. Regulations and new customer habits are challenging formulation scientists to constantly review their stabilizers and additives to fulfill these expectations. Lately, the trends have moved towards greener and more eco-friendly alternatives. The range of new stabilizers is now extremely wide and diversified and so the possibilities for new formulations can be limitless. TURBISCAN, the leading technology to measure stability is a perfect screening and ranking tool. Additionally, the TURBISCAN DNS associated with online capabilities and high-frequency measurement can study the dispersion state directly during the formulation steps thanks to the mixing function TMIX (with only 15mL) or process optimization with the circulation function TLOOP.

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.

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

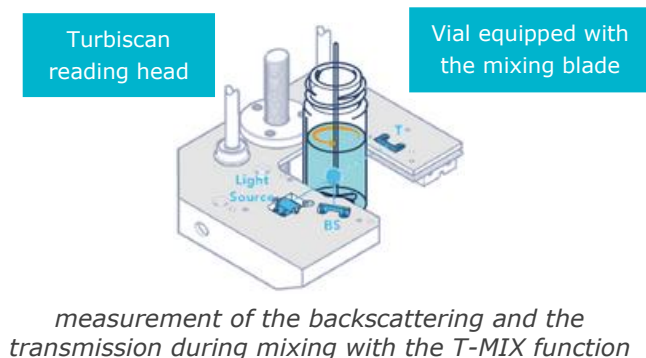
The signal is directly linked to the particle's concentration (φ) and size (d) according to the Mie Theory, with refractive index of continuous (n_f) and dispersed phase (n_p) being fixed parameters. The

measurement of the BS and T can be performed either in 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 samples.

Additionally, the TURBISCAN DNS associates 2 functions for online characterization of the dispersion state and the dispersibility:

- **Mixing function (T-MIX)** for automated fast formulation screening with a stirring bar directly inside the measurement cell.
- **Circulation function (T-LOOP)** for online measurements and scale-up or process optimizations



Experiment and method setup

The aim of the work: Compare the emulsification capabilities (oil droplet size) of two natural surfactants (coco-glucoside and decyl-glucoside) to Tween 20, a commonly used stabilizer

Sample preparation: The oil, water, and surfactant are added to the measurement vials (15mL) for the TURBISCAN analysis. On top of the vial, the T-MIX module is mounted, with a mixer blade and a customized cap for blade guidance.

The shear and mixing speed are directly applied to the emulsion in the measurement cell.

Sample analysis: The mixing rate is fixed at 2000 rpm and the measurement of the BS and T is started immediately, with no delay, and a frequency of 10 measurements/seconds. The mixing is monitored for 10 minutes. Particle size is measured during the emulsification process, without any sampling or dilution.

Using this setup, the amount of surfactant is limited to a few mL, ideal for fast screening and pre-formulation work with **low sample consumption**.

Results

Figure 1 represents the mean size of the oil droplets as a function of time. From Figure 1, the efficiency of each surfactant to successfully emulsify can be studied.

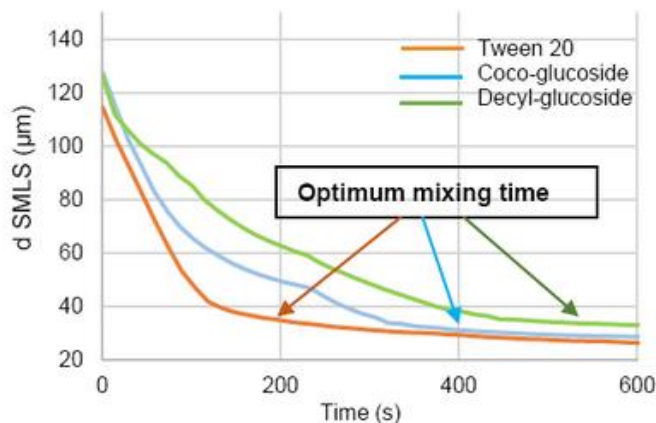


Figure 1. Mean diameter d_{SMLS} for the different surfactants with T-MIX over emulsification time

Interpretation:

- Coco-glucoside is a better alternative than Decyl-glucoside to replace Tween 20 (smaller particle size for emulsion prepared with Coco-Glucoside).
- The efficiency of the green surfactant (time to reach the minimum particle size) is different from the Tween 20. Indeed, at 200 seconds, the emulsion stabilized with the Tween 20 have reached 90% of the final value while it takes from 400 to 500 seconds with the green surfactants

The association of the SMLS technology and the mixing function TMIX provides a unique measurement of the particle size during the mixing step and allows to study of the emulsification efficiency rapidly in a small volume. This helps save time in the pre-processing steps on a lab scale condition.

Does it correlate with my homogenization process?

In order to validate the mixing function TMIX as a fast and easy screening tool for pre-process optimizations, the very same experience was done. Using an ultrasound probe higher shear conditions can be achieved. Figure 2 below compares the final particle size (determined by SMLS) after sonication and reached with TMIX after 10 min.

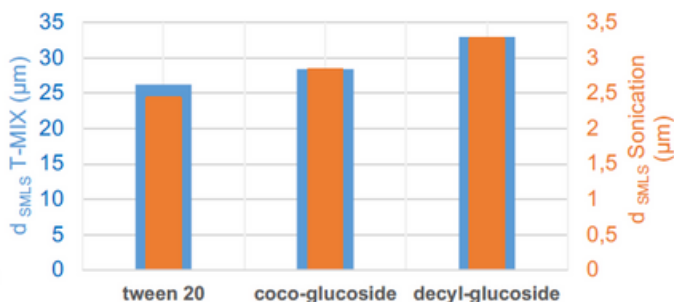


Figure 2. Mean diameter for the different surfactants with T-MIX versus sonication after 10 minutes

As expected, droplet size obtained via sonication is smaller compared to blade mixing, approximately by a factor of 10 (different levels of shear applied). However, the ranking of the surfactants as a function of their efficiency **remains similar**.

Consequently, the mixing function of the TURBISCAN DNS is a **fast** and **hassle-free** screening tool for a direct emulsification study, especially when multiple surfactants are to be compared. The tests were performed in only **10 min** and required **15mL** of sample volume.



Current methods:

- ✗ Large volume (Liters)
- ✗ Long process (30-120min)
- ✗ Multiple sampling for particle size data

VS



Current methods:

- + Small volume (15mL)
- + Fast (10-30 min)
- + Direct reading of particle size reduction

Conclusion

Large panel conditions

Fast (>10min) and low sample volume for efficient surfactant screening

No dilution and on native sample

While other techniques require a high level of sample preparation and dilution or additional forces and their impact is not negligible on particle size.

All-in-one

The TURBISCAN technology can help you select the right surfactant by measuring droplet size with online measurement and stability with the same instrument.

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