TURBISCAN

Fast and Reliable Creaming studies
Identify & quantify emulsion creaming

INTRODUCTION

Over their lifetime, many industrial products present a creaming phenomenon that usually has undesirable impact on the visual aspect and the end-use properties. The texture, taste and even quality perception of the product might be directly impacted (beverages, syrups, cosmetic creams, lubricants) ... Limiting the creaming requires to anticipate the physical stability during the formulation steps and controlling droplets migration can directly improve the shelf life of the product. This note shows how creaming can be studied, using a quantitative method to rapidly evaluate shelf life and compare formulations. Additionally, to fully understand the mechanisms of creaming, we present detailed calculations that can be done using the Turbiscan® technology

Time saving **Stability ranking** **Objective**

Definition: Creaming

Creaming is a phenomenon that consists of the migration of the dispersed phase to the top of the container over time. It is mainly observed in oil in water (O/W) emulsions

An emulsion is a liquid particle (droplets) dispersed in a liquid phase. These systems are thermodynamically unstable and will likely destabilize until complete phase separation. The reason of the instability is the lower density of the dispersed phase compared to the continuous phase which leads to the migration phenomenon, so called creaming.

The creaming process can be quantified by the migration velocity expressed by the Stokes law :

$$v = \frac{\Delta\rho_p \, g \, d^2}{18\eta}$$

d : particle diameter (assuming spherical)

v : particle creaming velocity

g : acceleration of gravity

η : kinematic viscosity of the fluid

$\Delta\rho_p$: difference between mass density of the particle and the fluid

The analysis of the Stokes Law gives some insight on how to limit the creaming: increasing the continuous phase viscosity, reducing the density differences or by reducing the average particle size.

Creaming: Conventional test

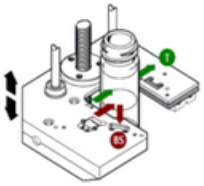
Easy to implement, visual observation remains the most used method for creaming testing, and thus stability analysis. The method is simple but can be time consuming as the variation must be visible to the naked eye to be detected. Additionally, the evaluation of the creaming kinetics is imprecise.

To overcome creaming issues, scientists need an early **identification** and **quantification** to apply the best strategy and measure the benefit of stabilizing agent addition.

Turbiscan®: How it works

Turbiscan® technology, based on **Static Multiple Light Scattering (SMLS)**, consists of sending light pulses (880 nm) into a sample along its height. The reading head scans the sample by moving vertically along the analysis cell and acquire data each 20 µm. Measurements are made over time and variation in the backscattering and transmission levels due to sample instability are recorded.

The signal is directly linked to the evolution of particle concentration (φ) and size (d) by the Mie theory.



BS = f(φ, d, np, nf)

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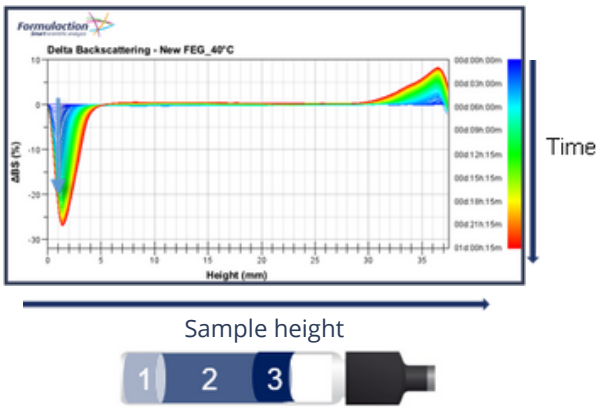
Samples with a particle concentration from 0.0001% to 95% (v/v) and from 10nm to 1mm in particle size can be measured as is.
The instrument enables to monitor changes in physical stability (coalescence, creaming, sedimentation, phase separation, etc...) **without any dilution or any stress** and thus follows ISO TR13097 guidelines.

ISO TR13097 defines scanning and spatially resolving techniques as perfectly suitable for stability analysis and suggests that the acceleration procedures applied to shorten test duration must not denature the initial product.

Creaming with the Turbiscan®

Turbiscan® profiles record the variation in light intensity levels which corresponds to sample instability. The variation (Y axis) is represented as a function of sample height (X axis in mm) over the time. The bottom of the sample is represented on the left part of the graphic and the top of the sample on the right part. The color gradient on the time scale corresponds to each scan time lapse with the **first scan in blue and the last scan in red.** Here, the recorded backscattering level is put in "Delta Mode"- ΔBS. The first scan is used as a reference to follow creaming evolution from sample 's initial state.



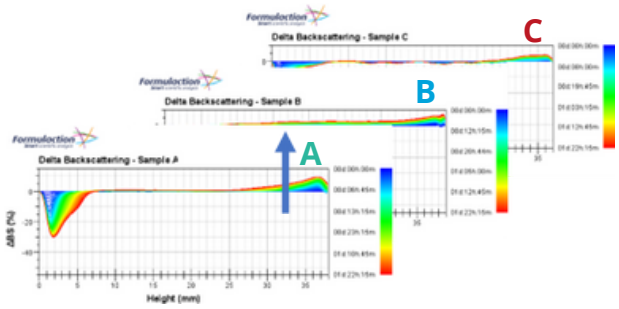
The above profile is an example of a sample analyzed for one day with the Turbiscan®. The graph allows to identify 3 key areas for data treatment:

- At the bottom of the sample (1) - left side of the graph-the Backscattering signal decreases with time, less scatterers are present at the bottom. The emulsion becomes less concentrated. A **CLARIFICATION** layer is formed over time.
- At the top of the sample (3) - right side of the graph- the Backscattering signal increases with time, which means more scatterers are present in this area. The particles migrate to the top and locally change the emulsion's concentration. A **CREAMING** layer is formed over time.
- In the central area (2), the Backscattering signal remains within same level during the test, this indicates that destabilization phenomenon is purely migratory, no size variation is seen in this case.

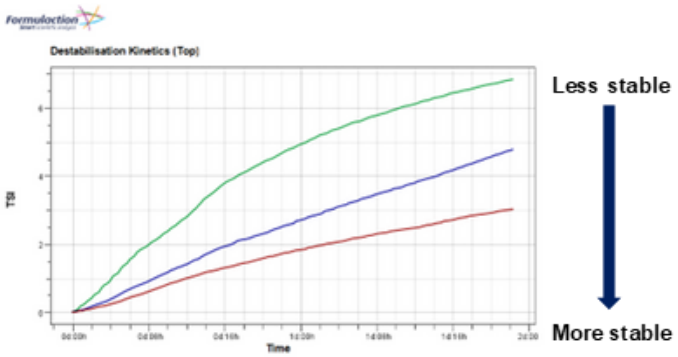
Even in concentrated media, particle migration is clearly identified within the **first hours** while it can take days & weeks to be visible by the naked eye to evaluate stability.

Compare and Select the best Formulation

The **T**urbiscan **S**tability **I**ndex **(TSI)** is an automatic calculation sums all destabilizations into a single number for easy, one-click ranking and comparison. **Therefore, the HIGHER the TSI value, the LOWER the Stability** (for more information on the TSI, download the application note TS-Stab_60-TSI calculations).



Three emulsions (**A**, **B**, **C**) were studied with the Turbiscan®. The emulsions are simply poured, as is, into the 20mL glass vials and measured for 2 days at 25°C. In a single click, the TSI can be obtained in function of time.



The destabilization kinetic of the sample C is slower than for the sample B and A. Therefore, the sample C can be ranked as the **MOST STABLE**. Measurements were performed for 2 days, but samples classification is already possible within the first hours (**4 hours**).

What are the benefits of using the Turbiscan?

Determining the best formulation requires many trials and strategies to limit or stop the creaming. To take a step back and accelerate the decision process, the stability testing must give precise and reliable answer (Go - No Go).

In addition to the fast detection (up to 200X faster), the creamed layer kinetics can be plotted with a single click calculation for an overall understanding and comparison. More detailed calculations are also available to provide a deeper understanding of the migration process to study the intensity, the thickness of the layers, creaming velocities details, and particle size.

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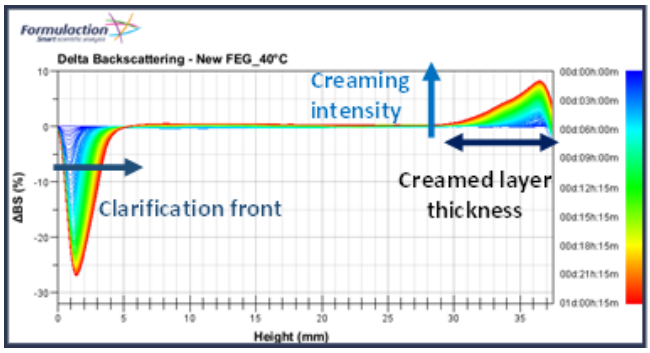
Detailed understanding of the Creaming

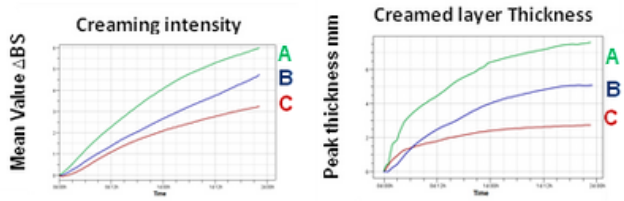
As creaming progresses over time, the clarification front moves forward, and the creaming layer thickness and intensity increase at the top of the vial:



All these parameters can be monotored to understand the creaming process.

How significant is the creaming ?

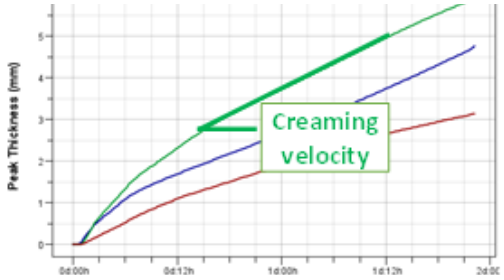
As an example, the progression of the creaming for 3 samples A, B, and C is shown below: the mean values of Backscattered signal - Δ**BS** (left) represent the intensity of creaming and the peak thickness the on the right shows the thickness of creamed layer:



The sample A is the sample with the highest concentration change (creaming intensity) and it generates the thickest creamed layer of 7.5 mm after 2 days. During the first hours, for sample C creaming layer is thicker than for sample B. Then the creaming kinetics evolve, and sample B ends up having a thicker layer than sample C.

How to calculate the exact creaming velocity and particle size of migrating particles ?

The evolution of the **clarification front** enables to follow the creaming velocity of migrating particles.



The slope of each curve corresponds to the speed of migration of the clarification front. In other words, to **particle migration speed**. Particle of sample A are migrating faster than sample B and C.

Based on the Stokes equation, adapted to concentrated media, the migrating particle diameter or so called the hydrodynamic diameter can be evaluated by adding the necessary information.

Sample	Creaming Velocity	Dhydrodynamic
Sample A	0.109	2.60 µm
Sample B	0.084	2.29 µm
Sample C	0.067	2.04 µm

This diameter corresponds to the diameter of the particle in motion. If the clarification front kinetic is not linear (multiple slopes) hence the particle size of each population can be determined to give of the particle size distribution.

CONCLUSION

Turbiscan® technology based on Static Multiple Light Scattering (SMLS) enables to study creaming in **concentrated media without any dilution or mechanical stress.** Creaming is detected **faster** compared to the eye (200x faster). The Turbiscan® allows not only to detect but also to quantify the creaming, either with the Turbiscan Stability Index (TSI) or by plotting detailed kinetics. Based on these reliable results, the strategy to overcome creaming is selected based on an objective choice.