Stability of Transparent Primer Formulations

-Characterizing multiple destabilizations simultaneously-

PAINT & INKS

Introduction

Transparent and semi-transparent primers are dispersions that are formulated to be stable for extended time periods. Their formulas can be slightly turbid without being completely opaque but visual analysis of the phase separation of these materials is still subject to guesswork and timeline extrapolation. This physical stability characteristic must be tested in order to determine the integrity of the substance during store shelf ageing, warehouse storage, and transport through various climates. This note will show how Turbiscan® technology permits to evaluate and distinguish primers in terms of stability which was not possible by visual observation.

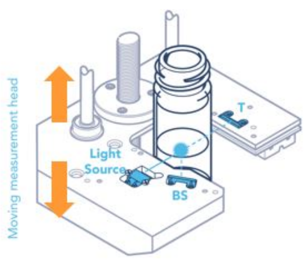
KEY **BENEFITS**

- VERSATILE
- TURBID SAMPLES
- FAST METHOD

Reminder on the technique

Turbiscan® technology, based on Static Multiple Light Scattering (SMLS), consists on sending a light source (880nm) on a sample and acquiring backscattered (BS) and transmitted (T) signal over the whole sample height. By repeating this measurement over time with adapted frequency, the instrument enables to monitor physical stability.

The signal is directly linked to the particle concentration (φ) and size (d) by the Mie theory knowing refractive index of continuous (n_f) and dispersed phase (n_p):



$$BS = f(\varphi, d, n_p, n_f)$$

Materials & Method

Three primer samples were analyzed with the Turbiscan®. The experiments were conducted at both 25°C and 50 °C in order to mimic the conditions these materials may see over their post-production lifetime.

Each sample was then scanned every 60 minutes for 2 days in order to produce the destabilization profiles.

Results

Raw data

The slightly turbid samples are studied in delta transmission mode (ΔT), where the first scan is used as a reference scan and the following data shows the evolution of the sample as compared to this first scan. Figure 1 below displays a representative sample that has aged at 50 °C.

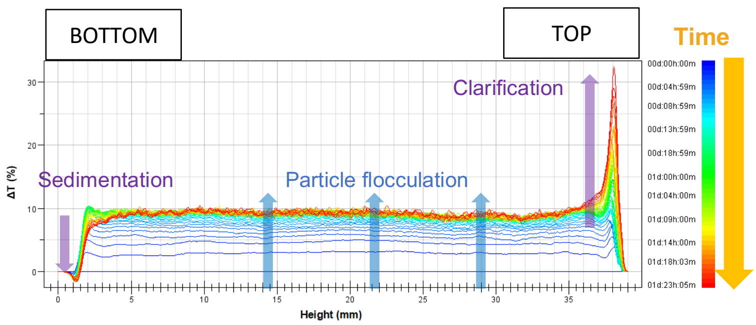


Figure1: Delta transmission data of a transparent primer.

First scans (blue) show early global evolution on the entire height of the sample. This is typically an indication of occurring flocculation.

Following scans (green to red) show at the top of the sample a large positive evolution of the transmission signal with the accompanying negative/decreasing transmission signal at the bottom of the sample (left of the graph).

This indicates that particles are settling to the bottom of the sample as a sedimentation phenomenon and can be suspected that it is a result of early stage flocculation.
Each sample in this note exhibits similar behavior. The Turbisoft program allows for identification and quantification of the kinetics responsible for destabilization.

Flocculation kinetics monitoring

The average value of scattered light can be analyzed in the middle of the sample versus time in order to generate a flocculation kinetic for each dispersion. Figure 2 shows the kinetic comparing the three formulations at 50 °C.

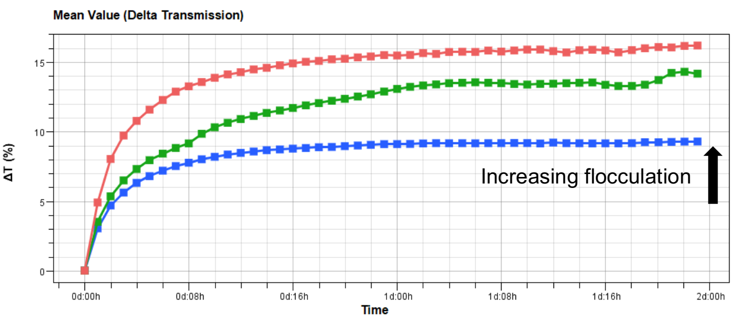


Figure 2: Flocculation kinetics for all samples.

It is important to note that all samples exhibit little or no resolvable flocculation at 25 °C (near 0% ΔT) over 2 days and are difficult to confidently resolve. But at higher temperature, it is seen that samples A (red) and C (green) have higher flocculation kinetics than sample B (blue). Clearly, the higher temperature is causing this to take place but can also expand the resolution that exists in the samples.

Quantifying destabilization with the TSI

The TSI is an algorithm-based calculation within the software that compares all destabilizations and sums them into a single number for easy, one-click ranking and comparison.

In addition to the flocculation kinetics, the TSI will incorporate any other phase migration kinetic (sedimentation, clarification) in order to provide a quick, global raking of sample stability. Figure 3 provides the TSI plot for all samples.

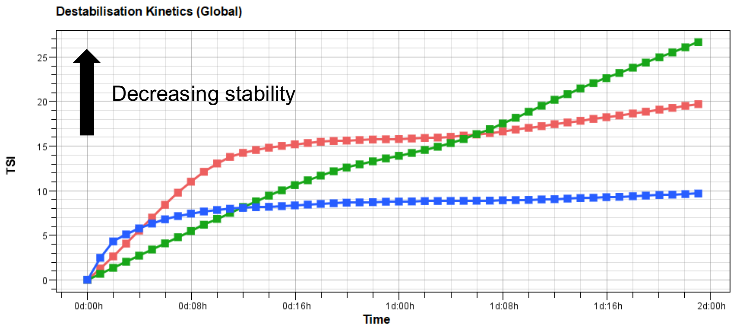


Figure 3. Turbiscan Stability Index (TSI) vs time.

Sample	TSI @ 2 days
Sample A (red)	19.6
Sample B (blue)	9.6 (most stable)
Sample C (green)	26.5 (least stable)

Table 1: TSI values calculated at 2 days.

The lower TSI values of Sample B indicate the particle kinetics are the lowest for this sample. Additionally, this TSI trace has plateaued and indicates that the destabilizations slow and are not subject to large changes over time whereas the other samples have sloped or linear TSI plots as the destabilizations are likely to continue at higher rates.

The higher temperature experiment here will not only decrease the total experiment time by accelerating the particle kinetics but will also allow formulators to see how the samples will behave in high temperature storage, such as warehouses, trains, or trucks. Such data is collected in a simple 2-day experiment with a single click in the TSI calculation.

CONCLUSION

Three primer formulations provided very similar destabilization phenomena but were difficult to distinguish which one was more stable by visual analysis. The Turbiscan is able to provide detailed kinetics of individual destabilization phenomenon present in otherwise visually indistinguishable samples while the TSI feature allows for quick and accurate ranking of the overall stability of multiple samples.