TURBISCAN LAB

Hard Settle Study of Toners

- Quantified kinetics of sediment packing -

PAINT & INKS

Introduction

The primary destabilization kinetic in paint and ink formulations is the sedimentation of the suspended particles to the bottom of the sample. In most, if not all, cases, this is unavoidable and redispersion will be required. The main concern is that the redispersion technique (stirring, shaking, etc.) will not be energetic enough to render the suspension back to its initial, homogeneous state. When this is the case, the sediment settles and packs on the bottom of the storage container and the formulation loses its properties and quality. Since packing takes months to complete a fast, quantitative method needs to be developed in order to complete the product development process with confidence that the formula will remain stable and the prediction of this effect is critical.

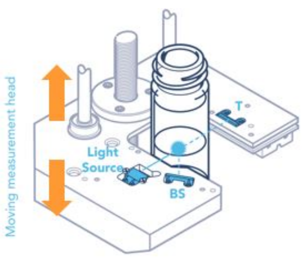
KEY **BENEFITS**

- VERSATILE
- PREDICTIVE RESULTS
- ACCURATE

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 toner samples were analyzed with the Turbiscan®. The experiments were conducted at 50 °C in order to accelerate any destabilization conditions that would cause the formulation to fail and have the particles settle and hard pack. Each sample was then scanned every 60 minutes for 5 days in order to produce the destabilization profiles.

Results

Raw data

Raw backscattering (BS) data is acquired over a 5-day period in order to monitor the changes in the sample and predict the ability of a toner formulation to settle and pack (coalesce). A typical plot is shown in Figure 1.

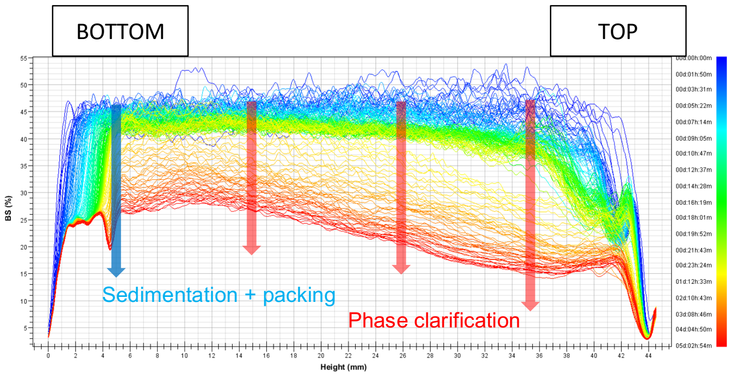


Figure1: Backscattering data from a hard-settling toner

It is seen that an overall phase clarification is occurring at the top and middle of the samples as indicated by the red arrows in Figure 1. This shows that the BS is gradually decreasing over time as the particles are settling.

Left side of the graph (the bottom of the sample vial) is the one to be analyzed for key indicators: particle size and concentration changes. It shows a *decrease* in BS as well, which is a sign that the particles are coalescing in this zone (blue arrow).

The existence and the extent of this hard settling needs to be studied in order to determine permanent formulation failure and to provide concrete details into the shelf life of the product.

Sedimentation kinetics monitoring

By analyzing how the BS% changes over time, the packing kinetic of the particle sediment can be analyzed and quantified. In Figure 2 below, the three samples are plotted to reveal this kinetic.

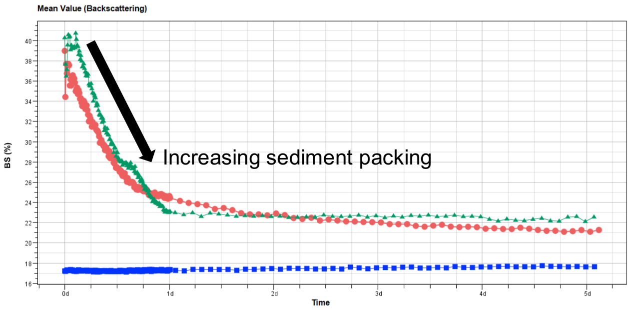


Figure 2: Sedimentation/packing kinetic plot.

Sample A (red) and Sample C (green) both display a large, negative BS trend over the 5-day analysis period while Sample B (blue) displays a rather constant change in BS%. This would reveal that the former two samples are likely prone to packing, possibly of the irreversible kind, whereas Sample B would exist with a long shelf life.

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 order to establish a threshold for failure, the TSI can be used to resolve the overall kinetics of each sample. This one-click calculation can therefore predict long-term shelf life in a matter of days.
The TSI plots for the three samples are shown below in Figure 3.

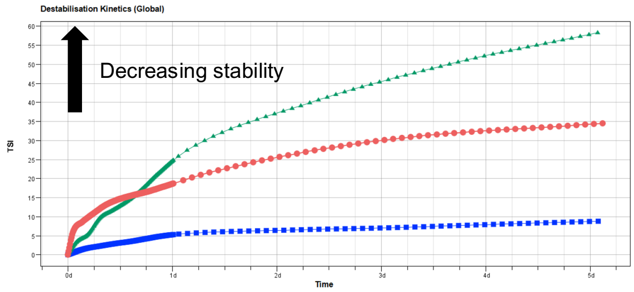


Figure 3. Turbiscan Stability Index (TSI)vs time.

Sample	TSI @ 5 days	Hard pack after 6 months?
Sample A (red)	34.8	Yes
Sample B (blue)	9.6	No
Sample C (green)	58.5	Yes

Table 1: TSI values calculated at specific ageing times for both dispersions.

There is a clear resolution of all samples which allow for easy identification of the global destabilization profiles. In this experiment, samples were separately analyzed after 6 months of storage to determine if hard packing had truly occurred. The results, displayed below in Table 1, show that samples A and C do indeed show hard packing and correlate well with the high TSI values. Sample B does not show hard packing, evidenced by the lower TSI result.

Therefore, the TSI function is able to ***predict in less than 5 days what would otherwise take 6 months*** to confirm!

CONCLUSION

Long-term shelf life studies that are typically conducted by visual or manual analysis are typically subjective by nature and can take weeks or months to complete. By utilizing the advanced kinetics analysis from the Turbiscan, long term analysis can be completed in a fraction of the time while also providing detailed information about the evolution of the samples.