Quantitative Physical Stability Analysis of Paint, Ink and Coating Dispersions

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STABILITY & SIZE
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Formulation of Coatings

Introduction

- New paint, ink, and coating formulations rely on the formulator to provide optimal properties of the product throughout its entire lifetime.

- Pigments, binders, additives and solvents must be chosen and dispersed in such a way for the formulation to possess ideal properties including stability, viscosity, consistency, and overall quality.

- Many tools are available to chemists in order to analyze such qualities but some gaps remain. Extrapolation of results, correlation of measurements, and subjective analysis may improperly characterize the mixture.

- This can lead to poor performance of the product on an application side and the inability of a sample to redisperse after ageing, among other drawbacks.

Formulation of Coatings

Physical Stability Concerns

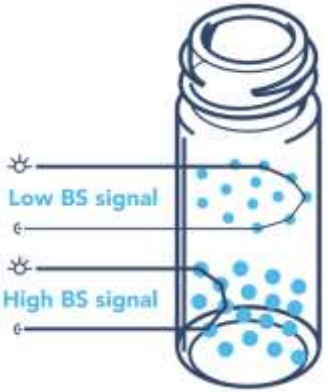
- A few issues are critical when discussing the physical stability and ageing/evolution of formulations related to inkjets, paints, and coatings.
    - Physical appearance (visual phase separation).
    - Redispersion capability once a sample has aged.
    - Prediction of sediment particle packing.
    - General shelf life.

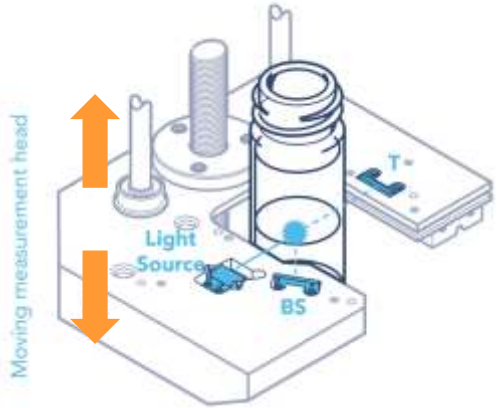
- Additionally, R&D formulators would benefit from a fast method to **quantify** how changes in chemistry effect the formulation.
    - Regulations
    - Different raw material lots
    - New, improved formulations

Static Multiple Light Scattering

Technology and Theory

Static Multiple Light Scattering can be used to monitor particle concentration and size changes inside of concentrated formulations.





Backscattering (BS) is a function of:

d : <u>particle size</u>

Φ : <u>particle concentration</u>

Repetition of the measurement provides:

Δd : <u>change in particle size</u>

Δ Φ : <u>change in particle concentration</u>

Allows for quantification and prediction of destabilization kinetics such as creaming, flocculation, and sedimentation.

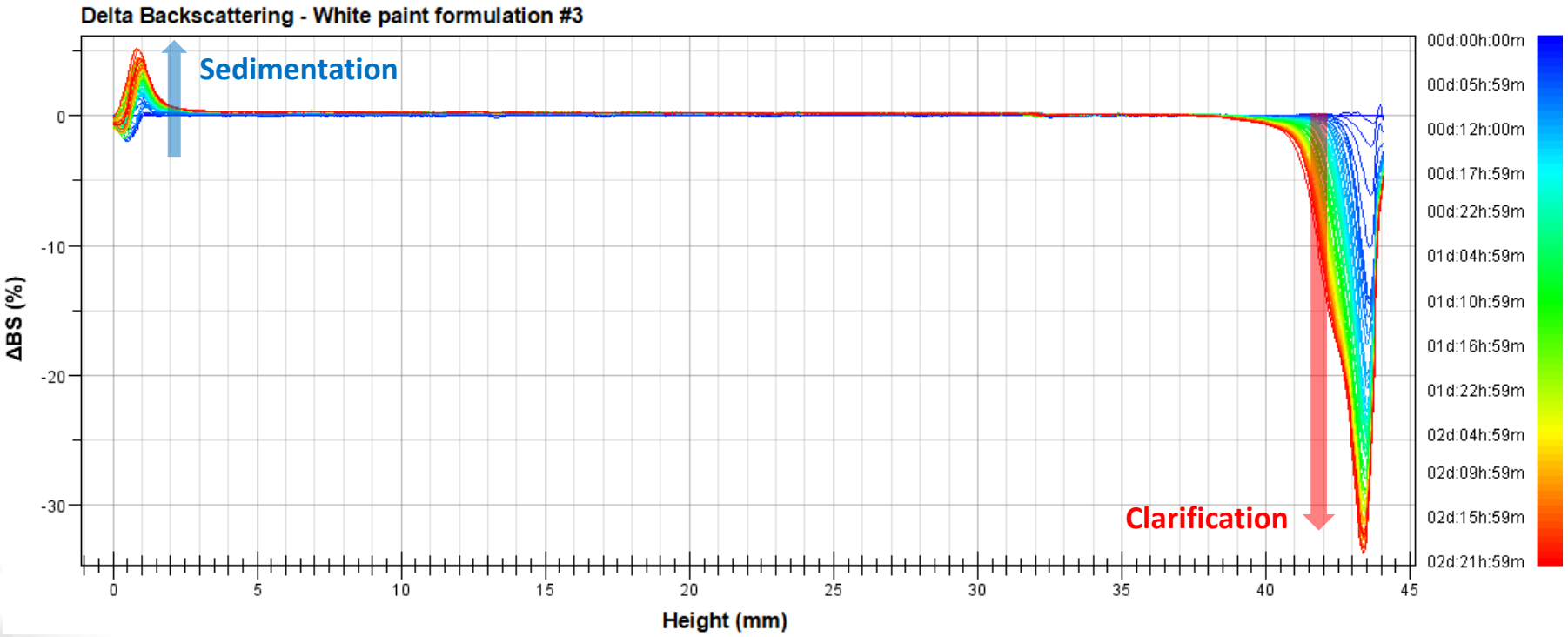
SMLS Applications

Paint & Ink

Pigment Formulas

White Pigment Formulation Modification

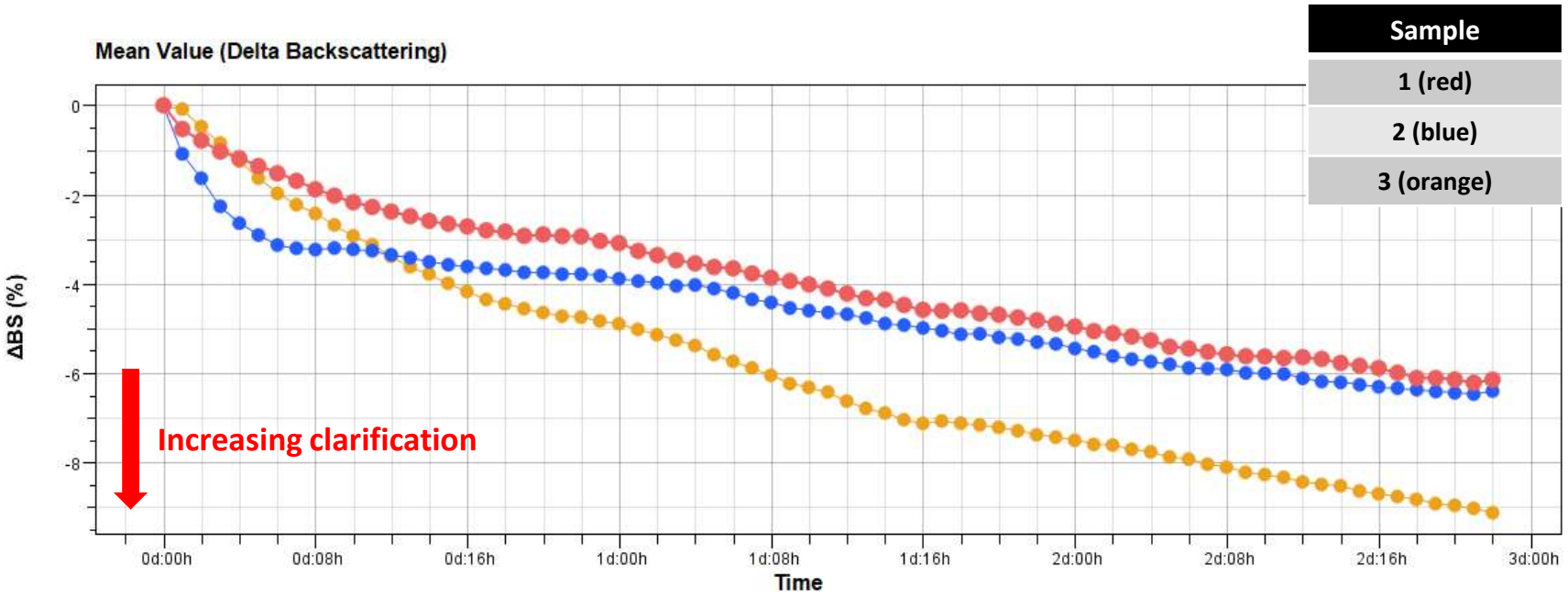
- Three white pigment formulations were analyzed for top-layer clarification as an indicator of overall stability.
- This clarification kinetic can be quantified and the formulations ranked for stability well before any visual phase separation is detected by the naked eye.



Pigment Formulas

White Pigment Formulation Modification

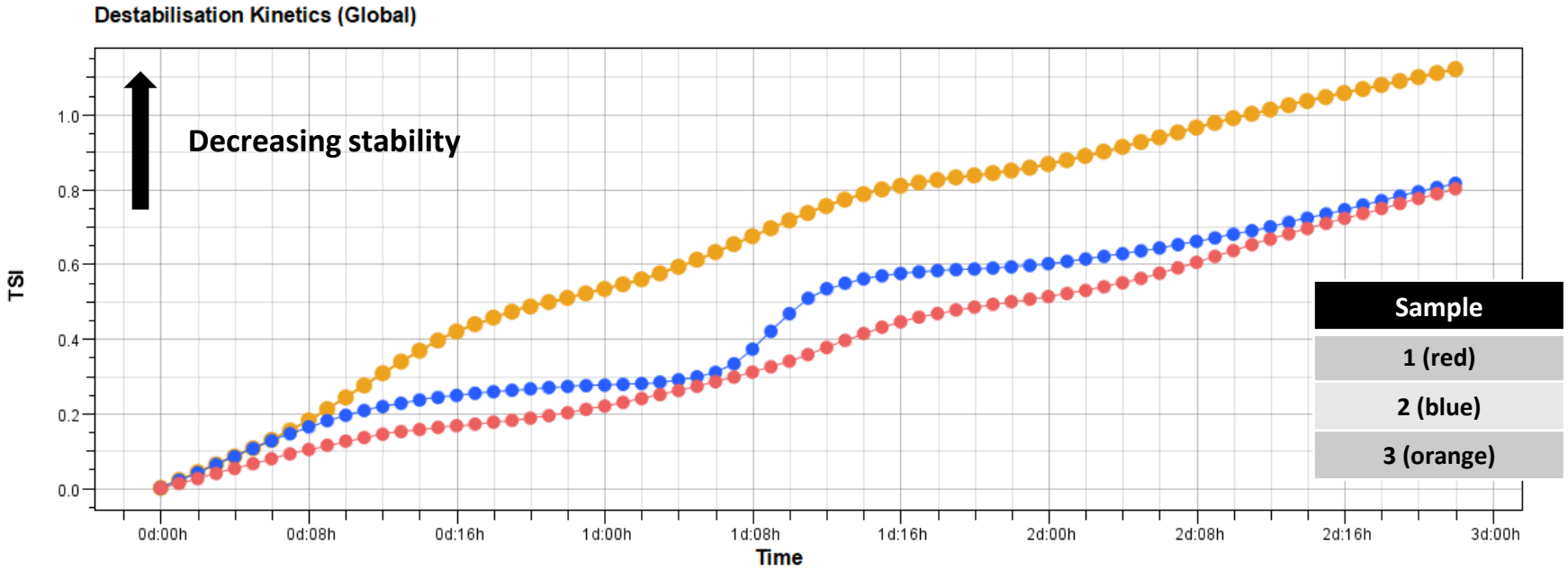
- Light scattered in the clarification/phase separation zone can provide top-of-vial kinetic.
- This allows to quickly and quantitatively determine how the modification in the formula alters the final stability of product.



Pigment Formulas

White Pigment Formulation Modification

- Global stability kinetics (including any sedimentation and flocculation) show clear indication that formulation #3 exhibits greater instability than the other two.
- This formulation should be modified or the process changed to match the stability of the other two formulas.

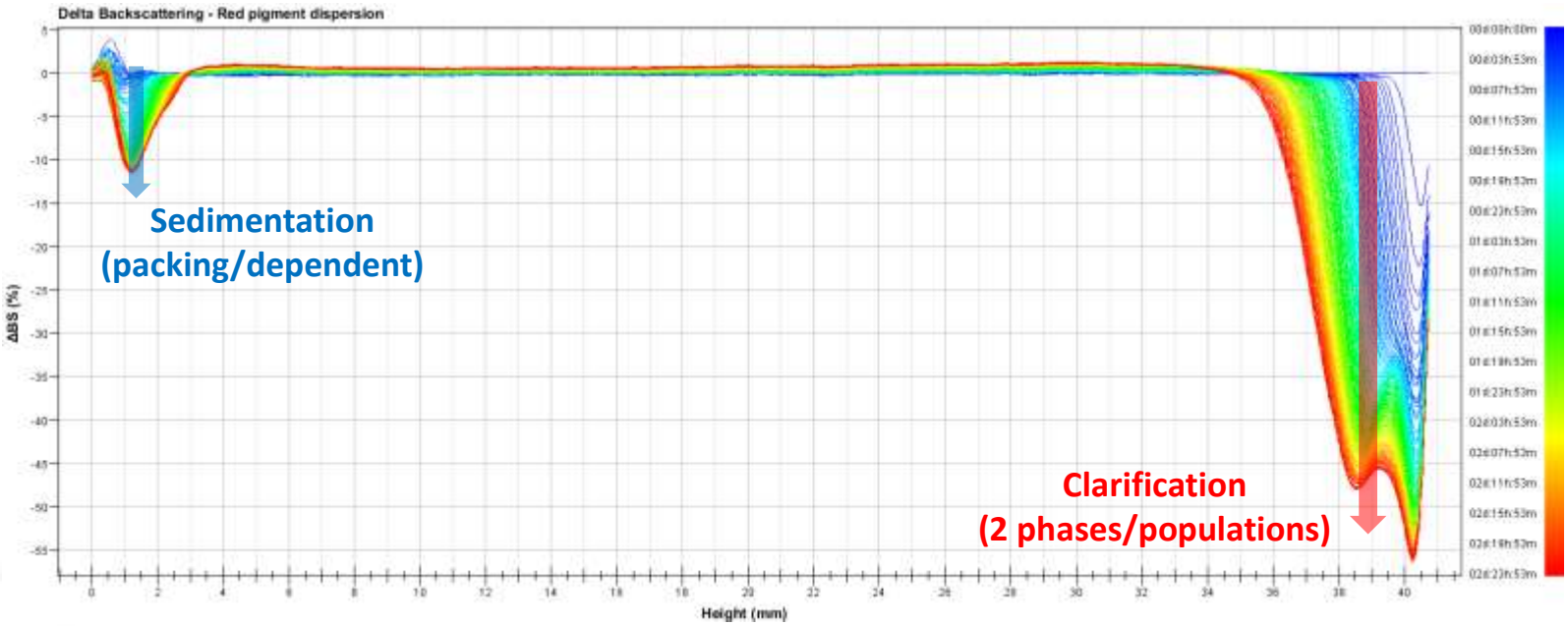


Extremely useful data for formulation control

Inkjet Formulations

Effect of Pigment

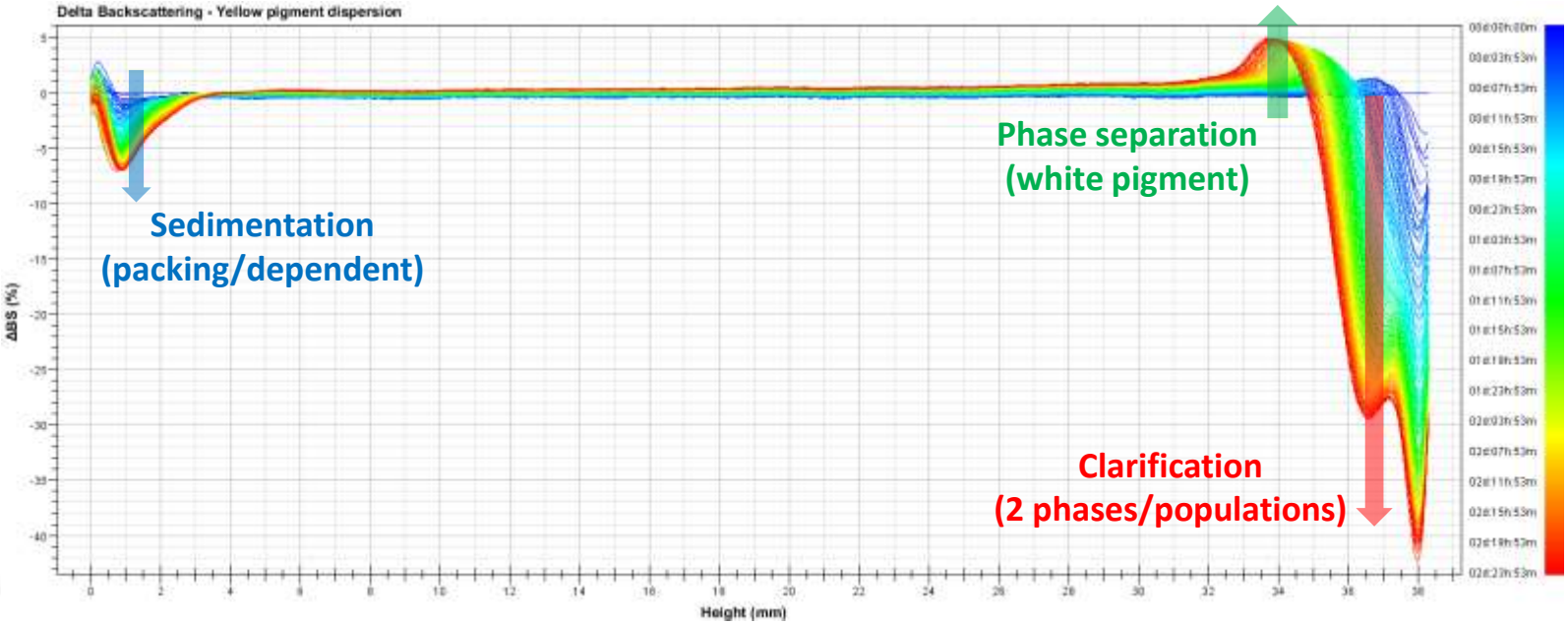
- Inkjet samples are prone to destabilization and packing of the sediment. This hard packing can typically be redispersed by shaking or movement of a printing head.
- Kinetically, the packing is detected as a *decrease* in ΔBS. Even though there is an *increasing concentration*, the particles are *increasing in size* and dominating the signal change.



Inkjet Formulations

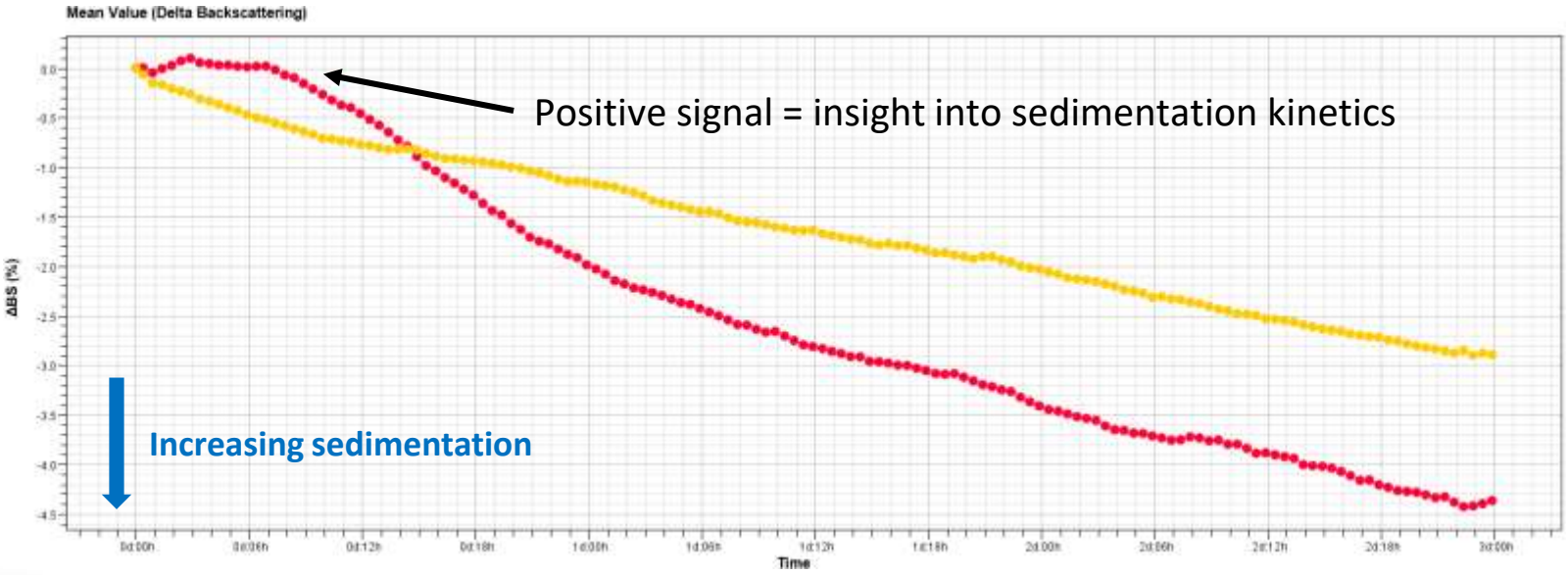
Effect of Pigment

- Samples with different pigments and formulations can be analyzed for the same types of destabilizations.
- This will allow for simple formulation adjustment and to see how different raw materials will behave in the formulation.



Inkjet Formulations
Effect of Pigment

- The mean value of light scattered at the bottom sediment layer is directly correlated to a packing kinetic.
- A few clicks will provide a detailed plot showing the progress of the packing. Note that the two samples are almost the same in this regard.



Advanced quantification of individual kinetics

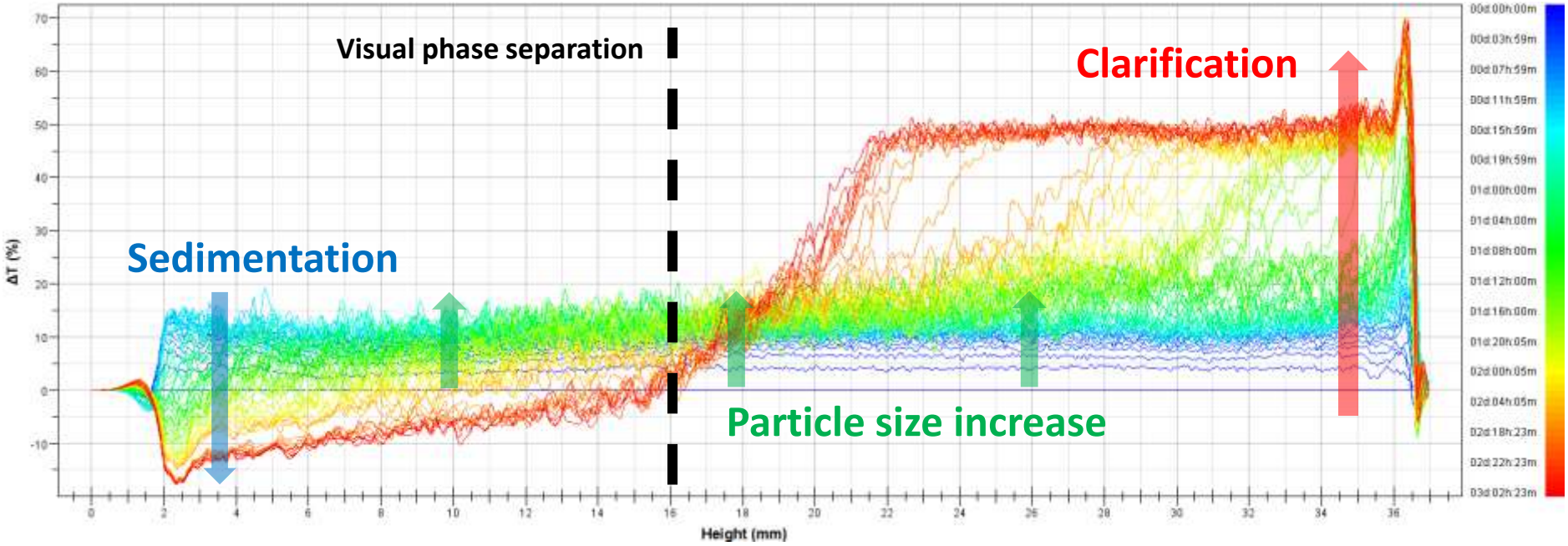
Transparent Material Applications

Refinish Stability

- Clear coats, pre-treatments and topcoat formulations have a degree of transparency, allowing the particle motion of the system to be traced by the transmission detector of SMLS instrumentation.
- Three samples are tested at both RT (27 ˚C) and 50 ˚C to test for particle flocculation, sedimentation, and phase separation.

Transparent Material Applications

Refinish Stability – High T Acceleration



Flocculation is the first kinetic to be seen

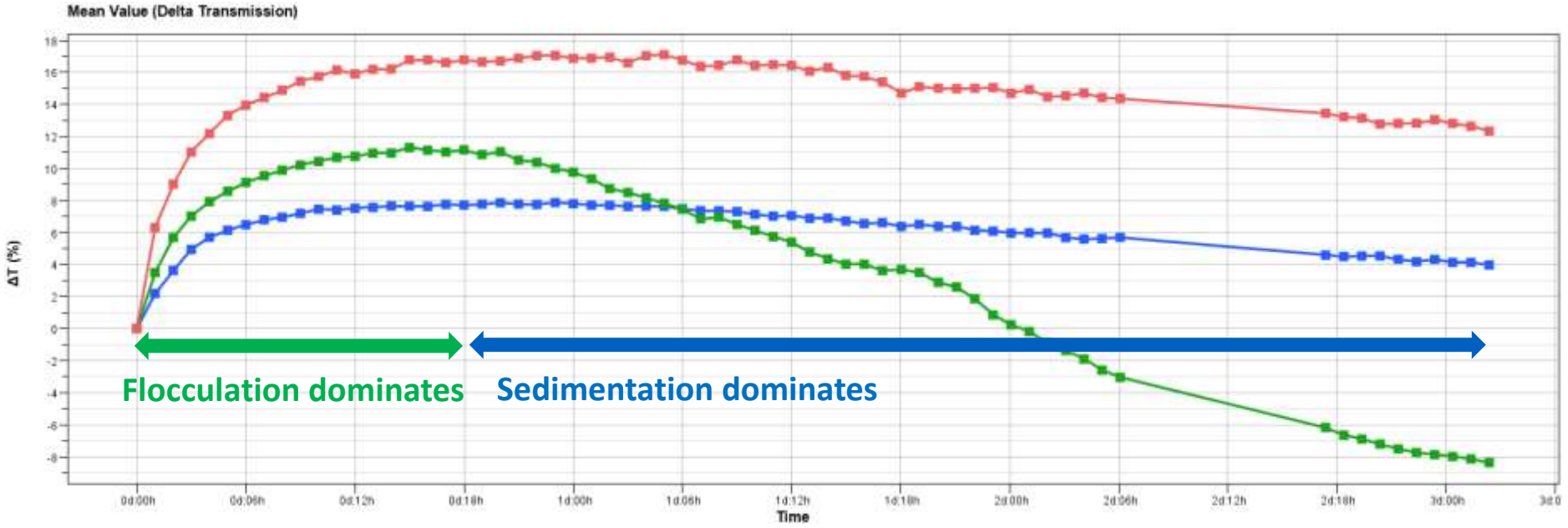
Sedimentation causes a decrease in transmission

All samples exhibit similar behavior – but which are more stable?

Transparent Material Applications

Refinish Stability – High T Acceleration

- Bottom-of-sample analysis shows that flocculation dominates and is then taken over by sedimentation as the ΔT decreases.
- The slope of the graphs provides info into the sedimentation kinetic, even when other signals are interfering in the calculation zone.

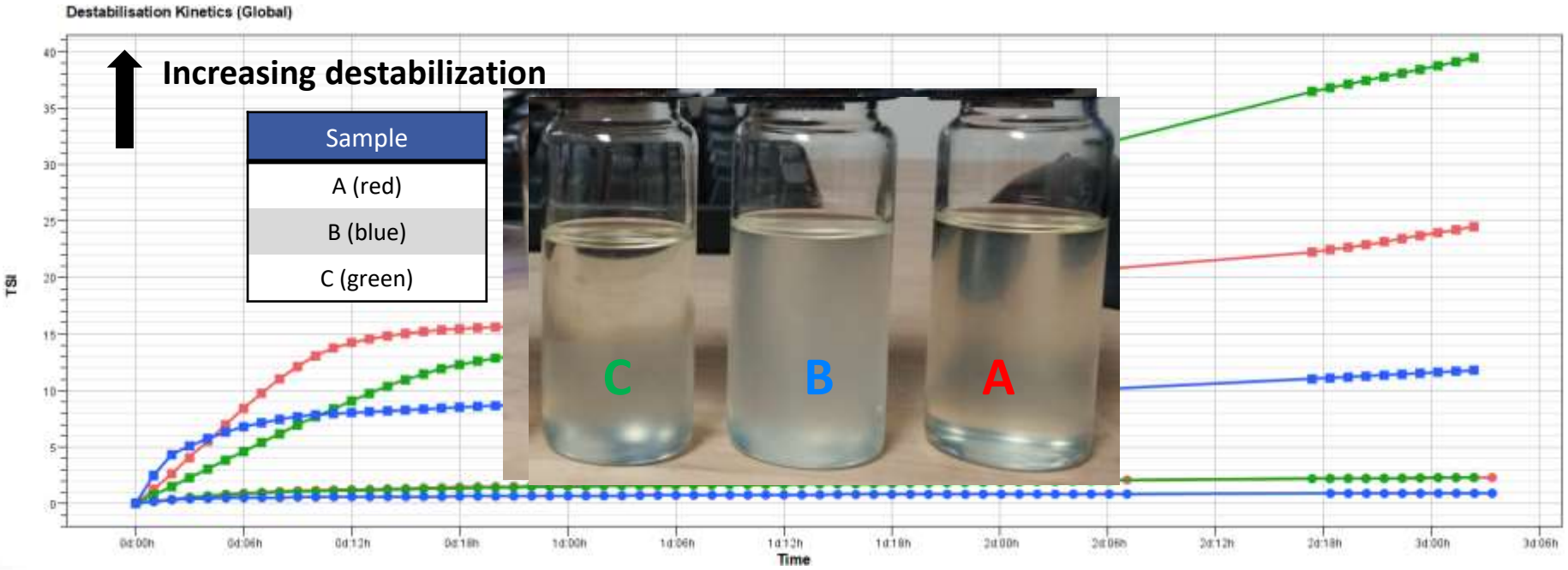


Sample	Slope (18 hr to 3 days)
A, 50 °C (red)	-2.12%/day
B, 50 °C (blue)	-1.80%/day
C, 50 °C (green)	-9.00%/day

Transparent Material Applications

Refinish Stability – High T Acceleration

- Global stability kinetics show a distinct difference between samples analyzed at RT (but the naked eye cannot).
- Plots at 50 ˚C provide a clear distinction between the samples, which shows the same trend from the visual analysis of the samples.

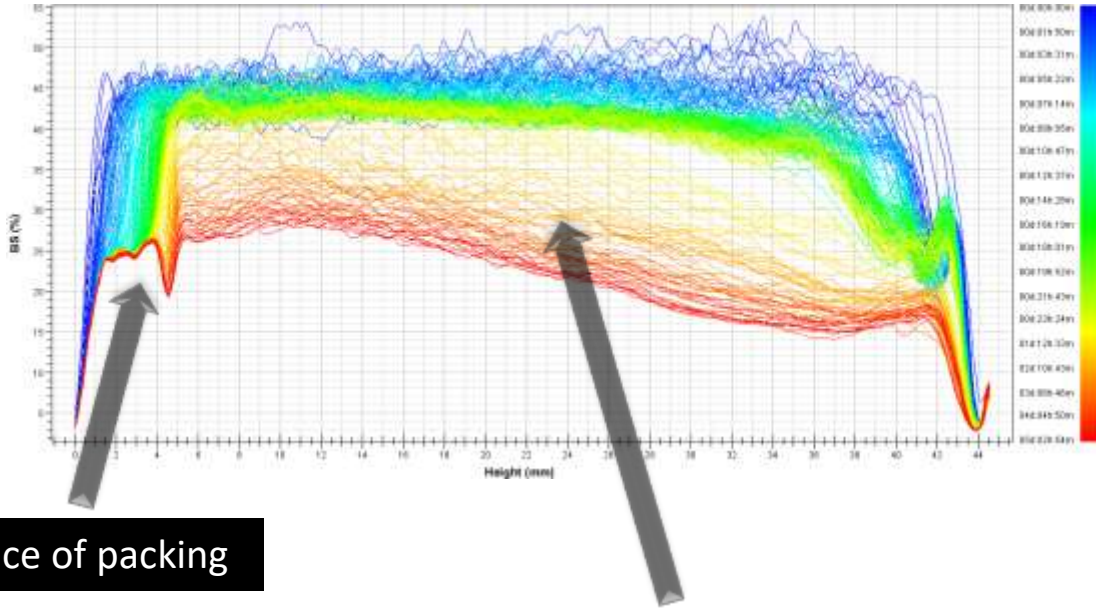


3 days of analysis provide comparative stability data

Shelf Life Projections

Hard Settle Study: Toners

- Toners with the ability to hard pack over their shelf life were analyzed both with SMLS (5 days) as well as by standard methods (6 months).
- The results for both studies correlated exactly with the propensity of the formulations to not produce a hard pack, surely produce a hard pack, or to remain questionable for a hard pack prediction.



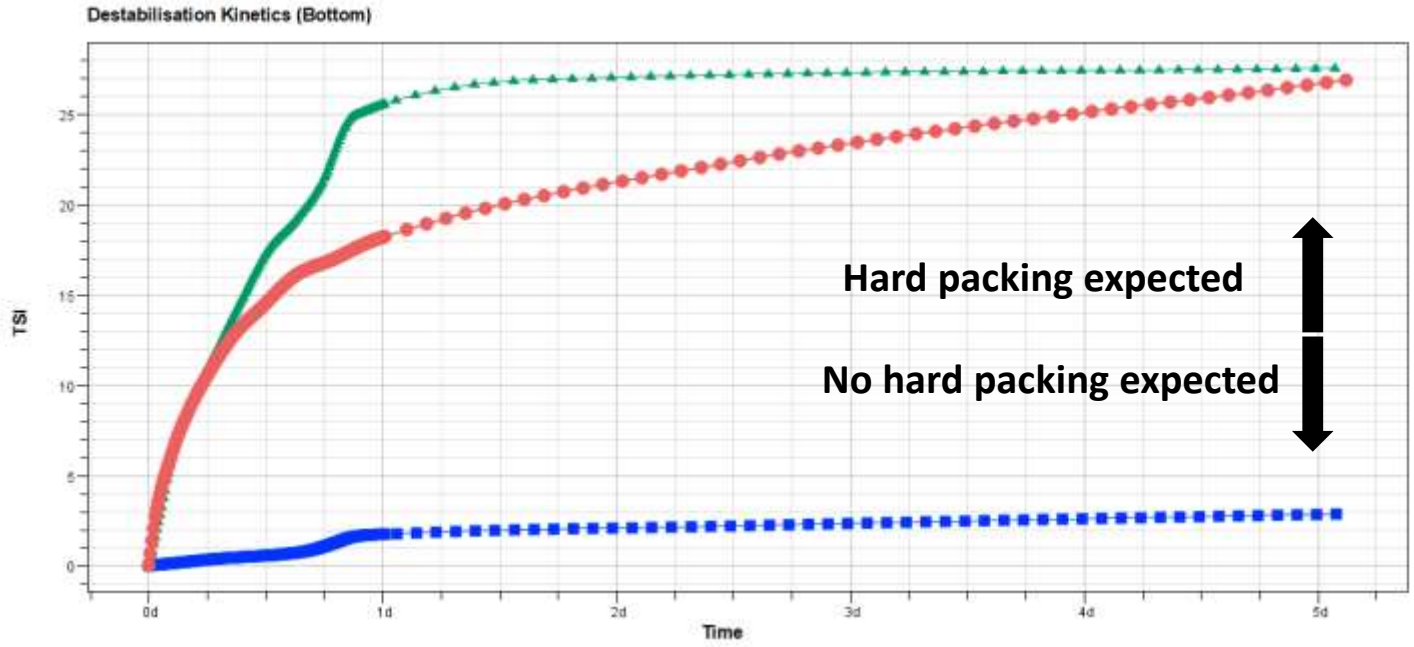
Decrease of BS at the bottom is evidence of packing

Large, broad clarifying layer (but still opaque)

Shelf Life Projections

Hard Settle Study: Toners

- In order to avoid the clarification kinetic in the global stability kinetic at the bottom of the sample (bottom 20% height) was analyzed.
- Hard packing is seen in the red (observed hard settle) and green (questionable hard settle) traces, evidenced by the high numbers.
- Samples that do not hard pack (blue trace) would have very low changes in this region.

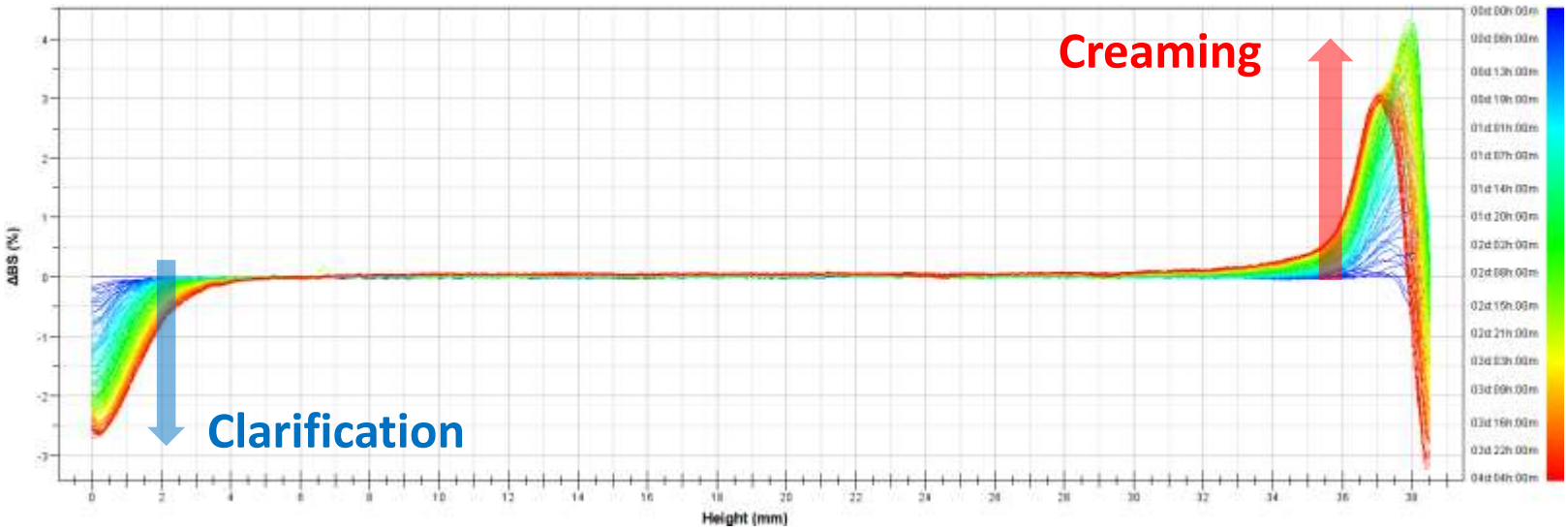


6 month test completed in 5 days with the same results!

Cookware Coatings Applications

Stability of Sprayable Coatings

- White PTFE polymer dispersions for spray coatings show phase separations based upon the components added to the mixtures (analyzed at 45 °C).



Sample	Clarification intensity (ΔBS%, 36 hours)	Creaming phase volume (ΔBS%, 36 hours)	Total stability (36 hours)	Total stability (4 days)
1	-0.47	0.89	0.4	1.1
2	-0.37	0.70	0.3	0.6

Conclusions

Stability of Coating Applications

- Physical stability kinetics of formulations can be analyzed, quantified, and predicted using Static Multiple Light Scattering.

- Concentrated inkjet dispersions, paint and coating dispersions, and clear coat and refinish dispersion all benefit from this type of analysis.

- This allows for fast formulation optimization and accurate predictions into how the chemistry of a formulation affects its performance.

Thank you!
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