TURBISCAN LAB

Redispersibility of ceramic ink characterized with SMLS

KEY **BENEFITS**

VERSATILE

OBJECTIVE RESULTS

ACCURATE

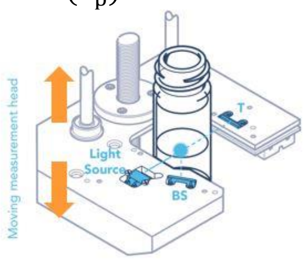
PAINT & INKS

Introduction

Printing inks used in ceramics are constituted of inorganic pigments dispersed in a serigraphic liquid. This liquid gives to the ink some rheological properties necessary for the printing process. A good stability of ink, at rest or in use, is essential because pigments tend to settle and to aggregate. This instability leads to evolutions of the rheological properties of the product, which can make the application difficult or even impossible, and provoke defects in the printing.
For a given pigment, it is necessary to study the stability of the ink as a function of the different serigraphic liquids as the aggregation can be reversible or not due to chemical interactions between the pigment and the liquid. Depending on the stability behaviour, the ink redispersibility can be different.
This note will show, on the example of three inks from the market, how SMLS technology permits to characterize pigment stability and redispersibility.

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 inks samples were analyzed with the Turbiscan®. They are composed of the same pigment in three different serigraphic liquids. The experiments were conducted at 25 °C. Only the backscattering variations (%, ordinate axis) along the tube height (mm, abscises axis) as a function of time (last curved displayed in red) were analyzed.

Results

Raw data

Raw backscattering (BS) data is acquired over a 10-hours period in order to monitor the changes in the sample (Figure 1).

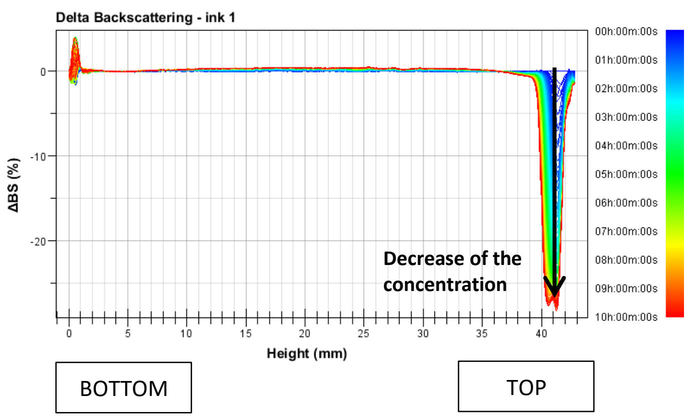


Figure1: Backscattering data for ink 1

A significant decrease of the backscattering signal is observed at the top of the sample over time. This is representative of a clarification occurring at the top of the product. This clarification is due to sedimentation of the pigment in the ink.

Sample stability as a function of viscosity

Monitoring the clarification layer formation allows to calculate the kinetics of sedimentation for each of the three inks. The figure 2 shows the migration rate as a function of viscosity of the sample.

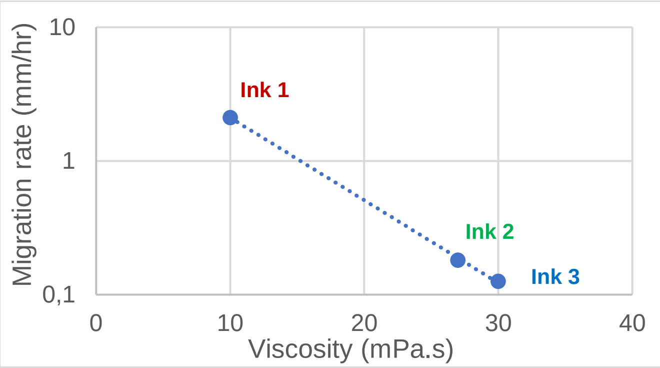


Figure 2: Migration rate as a function of the viscosity

There is a link between the ink viscosity and the migration rate of the pigment. The more viscous the ink is, the slower the sedimentation.

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.
The TSI plots for the three samples are shown below in Figure 3.

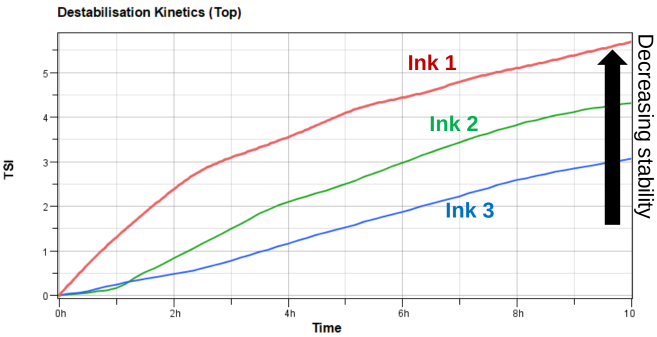


Figure 3. Turbiscan Stability Index (TSI) vs time

The TSI permits to rank the samples such as the migration rate and already after 2 hours of measurement.

Redispersibility of the ink

Generally, pigments are of high density and form a sediment at the bottom of the sample. In this sediment, the pigment can aggregate in an irreversible way.
The redispersibility can be evaluated by comparing the backscattering level at the beginning of experiment and at the end after shaking the sample.

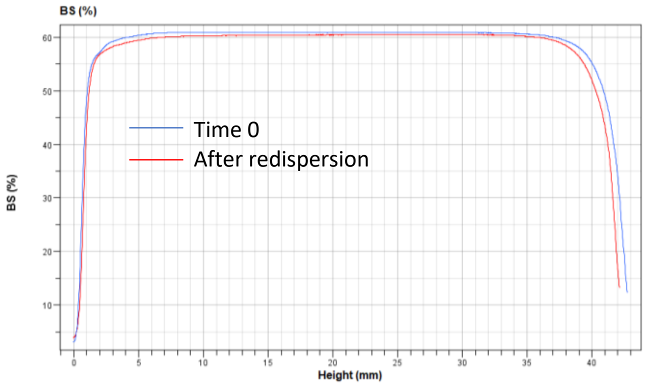


Figure 4: Comparison of backscattered level after preparation and after redispersion for ink3.

In the case when the signal intensity is equal to initial the level, the pigment was properly redispersed (example: Figure 4 for sample ink 3). If the signal is lower at the end of the experiment, the redispersion process was insufficient either because the required force was not met or because the aggregation is irreversible.

Sample	*Ink1*	*Ink2*	*Ink3*
BS level time 0 (%)	61	62	60.6
BS level after redispersion (%)	52	58	60.5
TSI after 4h (after preparation)	3	2	0.9
TSI after 4 h (after redispersion)	5	3	1

The table above compares the BS level and TSI for the sample analyzed after preparation and after redispersion. Only the ink3 is well redispersible and the stability remains the highest compared to other inks.

CONCLUSION

The Turbiscan® detects and monitors migration phenomena in only few hours allowing to rapidly compare the stability of different formulations. Signal comparison before and after sedimentation can also help in characterizing the redispersibility of the pigment sediment and evaluating whether the redispersion process provides required force to redisperse the pigment.
This type of study can be run with any kind of pigment dispersions (paints, ink jet…).