Pigments and fillers (Part 1)

- *Fast and effective raw material screening* -

Introduction

Choosing the correct raw materials for high quality formulation can be tedious and time consuming due to the wide variety of chemicals and alternatives (manufacturers, origins, eco-friendly…). The Turbiscan®, leading technology to measure the dispersion stability, can be the decision maker thanks to its online capability to measure particle size with no dilution. This application papers shows how the Turbiscan® DNS, with its embedded mixing function helps select the most appropriate TiO_2 to reach the desired particle size.

KEY BENEFITS

ONLINE

NO DILUTION

FAST

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How it works

Turbiscan® technology, based on Static Multiple light scattering (SMLS), consists on illuminating a sample with an infrared light source and acquiring backscattered (BS) and Transmitted (T) signals.

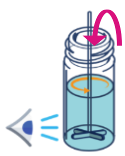
$$\boldsymbol{BS\ and\ T} = \boldsymbol{f}(\phi\,,\boldsymbol{d},\boldsymbol{n_p},\boldsymbol{n_f})$$

The signal is directly linked to the particle's concentration (ϕ) and size ($\boldsymbol{d}$) according to the Mie Theory, with refractive index of continuous ($\boldsymbol{n_f}$) and dispersed phase ($\boldsymbol{n_p}$) being fixed parameters. The measurement of the BS and T can be performed either on scanning mode, to provide homogeneity and stability measurement, or with very high frequency for fast time resolved and online measurement.

The measurements are done without any dilution & on native sample.

Additionally, the TURBISCAN DNS associates 2 function for online characterization of the dispersibility:

- Mixing function (TMIX) for automated fast formulation screening with a stirring bar directly inside the measurement cell. **Subject of this note.**
- Circulation function (TLOOP) for online measurements and scale up or process optimizations. (see Notes Flow 5-7).



Automated or Manual additions

Experiment and method set up

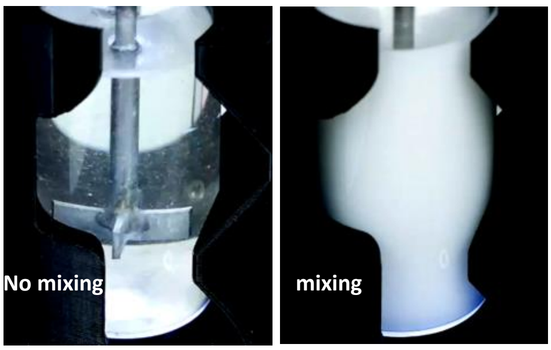
Various types of TiO_2 have been studied with the aim to rapidly screen and select the most adapted powder to reach the targeted particle size.

Sample	Manufacturer	Primary* particle size
TiO_2_1	Pylote	250-300nm
TiO_2_2	Sygma Aldrich	250-300nm
TiO_2_3	Marion technology	250-300nm
TiO_2_4	Kronos 2190	250-300nm
TiO_2_5	Kronos 2971	250-300nm

** provided by the manufacturer*

Samples are prepared at 0.5% wt in deionized water directly in the 20mL cylinder glass vial and placed in the Turbiscan. The TMIX is the mounted on the vial (dedicated cap and stirring blade to apply mixing placed directly inside the measurement vial).

The mixing rate is fixed at 2000 rpm and the measurement of the BS and T is started immediately with no delay and at high frequency (10 measurements/ second) for 30 min.



This setup gave a great advantage with particle size determination under controlled and fixed agitation to study how good is the resulting dispersion.

The measurements are done directly on the native sample, avoiding tedious and long sampling process present with other size measurement methods.

The graph 1 hereunder represents the evolution of the mean particle size in function of mixing time for the different TiO_2 dispersions.

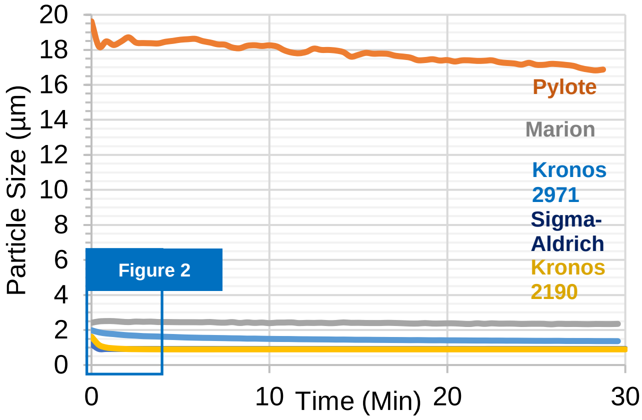


Figure 1. TiO_2 particle size in function of mixing time

The graph 2 hereunder represents a zoom in the first 3 minutes of TiO_2 dispersing into water.

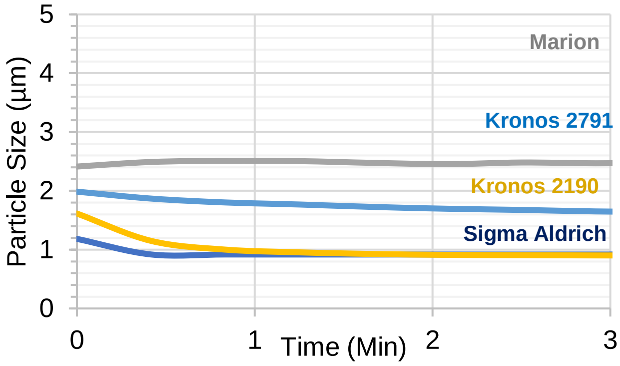


Figure 2. TiO_2 mean particle size in function of mixing time (zoom 3 first minutes)

From this graph, we can rank the TiO_2 upon their ability to reach the final particle size after a given time (30 min) regarding the primary particle size (250-300nm).

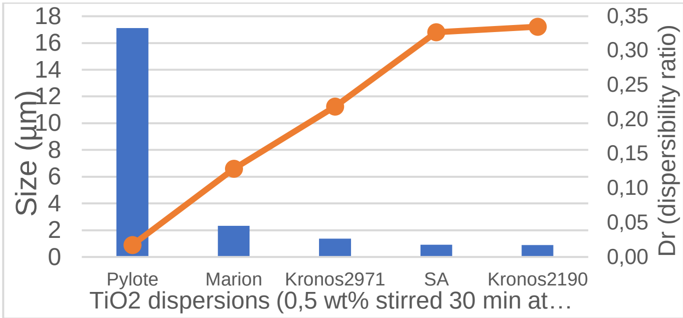


Figure 3. Particle size for the different TiO_2 after 30 min of mixing

Results

Despite a similar primary particle size (250-300nm announced by the manufacturer), the particle size of the different TiO_2 is considerably different once dispersed in water and can be explained by different treatments of the particle surface to help the dispersibility.
The primary particle size is not reached, and smallest particle size are achieved with the Kronos 2190 (900nm) and Sigma-Aldrich (920nm) while other TiO_2 are > 1µm.

Finally, the mixing time has a very limited impact on the final particle size, i.e.: the energy provided is not strong enough to break agglomerates. The final particle size is reached with couple of minutes.

Conclusion

Advantage of using Turbiscan DNS and TMIX function for particle screening

- Saves time:
  Simply weight, stir, and measure directly the native sample without any preparation and

- No dilution and on native sample:
  Other techniques require a high level of sample preparation, dilution or additional forces and their impact is significant on particle size.

- All in one:
  Once the correct raw material is selected to achieve the desired particle size, the Turbiscan technology can also help selecting the correct dispersant agent, adjust process parameters and stability measurement.