Scope

To achieve optimum performance of paint products (gloss, hiding power, separation stability etc.) a high level of pigment deagglomeration is of paramount importance. Deagglomeration performance is determined by:

- Selection of the processing device for high shear deagglomeration and dispersion
- Processing parameter energy input, duration
- · Selection and concentration of additives

Given this high complexity there is a need for efficient screening tools for process development and quality control. This application note demonstrates how a multisample analytical centrifugation system can be used to control and quantify the result of the deagglomeration treatment.

Even more it allows for high resolution determination of the particle size distributions.

For general application of multisample analytical centrifugation for testing stability and measuring quality of paint products see application note *Paint products - Accelerated stability and quality testing.*

How does multi-sample analytical centrifugation work

The LUMiFuge® / LUMiSizer® instruments employ the STEP-Technology®, which allows measuring the intensity of the transmitted light as function of time and position over the full sample length simultaneously. The data is displayed as function of the radial position, as distance from the centre of the rotation.

Up to 12 different samples can be measured in one test run providing high volume data output.

By means of the available analysis modes "Integral Transmission" (clarification speed) and "Front Tracking" (separation velocity) the separation behaviour of the individual samples can be compared and analysed in detail.

Why determination of particle size distribution is often insufficient in measuring deagglomeration

Simple measurement of the particle size distribution often fails to directly measure the degree of deagglomeration in real world paint products. Usually dilution is required, which can alter the dispersion structure. Looser agglomerates will break-up and will appear as fine particles, however, in the concentrated paint formulation they still will act as agglomerates.

Figure 1 compares the particle size distributions of pigment dispersions differing in the degree of deagglomeration. Sample A represents the pigment suspension (50 % m/m) after dispersion of the pigment in water. Sample B and C were deagglomerated by wet milling for 1 and 15 hours, respectively. From this figure one hardly could expect efficient treatment. The overall particle size distribution is changed only slightly, the content of particles $> 1\mu m$ is reduced to a small extent. Nevertheless, after 15 hours of wet grinding the final quality is achieved, which is in line with the product specifications.

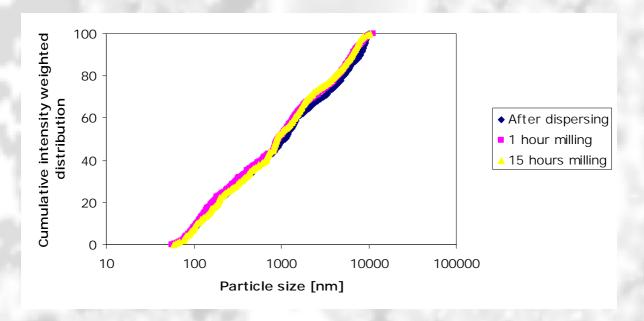


Fig. 1 Pigment dispersions – effect of duration of wet milling on the cumulative intensity weighted particle size distribution, determined with LUMiSizer (dispersions diluted to 0.25 %m/m)

Direct measurement of deagglomeration of original undiluted samples

Multisample analytical centrifugation can be used to compare the dispersion properties of different samples under strictly identical conditions. Fig. 2 compiles the kinetics and extent of separation during centrifugation at 2300 g for the pigment dispersions with different degree of deagglomeration. The movement of the interface supernatant-dispersion is depicted (given as distance from the centre of rotation). Increasing the extent and duration of deagglomeration the sediment height is reduced (packing density increased).

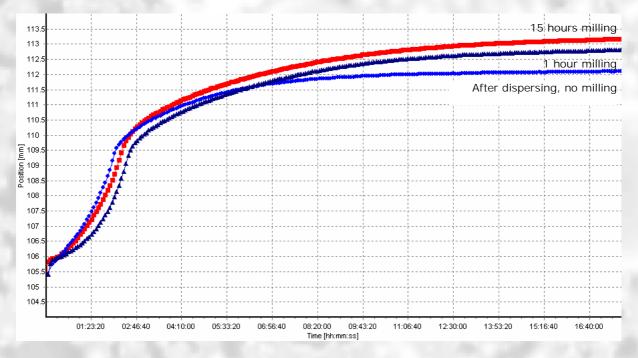


Fig. 2 Comparison of the kinetics and extent of separation during centrifugation, depending on the duration of wet milling. Position of the cell bottom is at 130 mm.

Reducing the number of (irregular) agglomerates a denser packing can be achieved. Therefore packing density (sediment height) can be used as a direct measure of deagglomeration. Further, a direct comparison can be made with a standard sample in line with the product specifications.

Conclusion

- Multisample analytical centrifugation is an efficient tool for the characterization of paint products with high throughput.
- Packing density (sediment height) can be used as a direct measure of deagglomeration. This way screening for selection of the processing device, processing parameter, and of efficient additives can be managed.

References

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- Dispersion Stability and Particle Characterization by Sedimentation Kinetics in a Centrifugal Field, D. Lerche, J. Dispersion Sci. Technol. 23 (5), 699-709, 2002
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