

Particle Size measurement of High-concentration Pigment Samples with DLS Technology

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Introduction

Pigments are either organic or inorganic coloring materials. In the manufacturing process, pigments need to meet many technical criteria, including ease of application, color strength, durability or light and weather fastness, all of which are closely related to the size of the pigment particles.

In this application note, dynamic light scattering (DLS) technology was utilized to measure the particle sizes of two high-concentration pigment suspensions. Due to the high concentration and poor light transmittance of the sample, a capillary sizing cell with an inner diameter of 1 mm was used for the DLS measurement.



Theory and Instrumentation

DLS measures the intensity fluctuations of the sample due to Brownian motions of particles. The diffusion coefficient D is obtained and related to the particle size, i.e., the hydrodynamic diameter D_H , by the Stokes-Einstein equation.

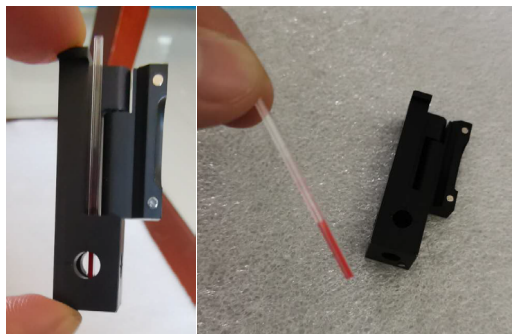
$$D = \frac{k_B T}{3\pi\eta D_H}$$

Where k_B is the Boltzmann constant, T is the temperature, and η is the dispersant viscosity.

In this study, the pigment samples were characterized by the BeNano 90 Zeta (Bettersize Instruments Ltd.), which adopts a 10mW laser with the wavelength of 633nm. In addition, in the BeNano 90 Zeta, single-mode optical fibers are used for signal transmission to maximize the signal to noise ratio; high-speed correlators are utilized such that the fast-decay correlation functions of small particles can be calculated effectively.

Experiment

A red and a yellow pigment were pre-dispersed in purified water for the particle size measurements. The measurement temperature was set to be $25\text{ }^\circ\text{C} \pm 0.1\text{ }^\circ\text{C}$ through the built-in temperature control system of the BeNano 90 Zeta. Each sample was measured at least three times to ensure a low standard deviation of the results.



Results and discussion

Correlation functions of two pigment samples were calculated through their scattered light signals and the overlap correlation functions of multiple measurements are shown in Figure 1 and Figure 2.

As shown, the repeatability of the correlation function is good, indicating the samples remain stable during the measurement. Neither the color nor the high concentration of samples affected the measurement results.

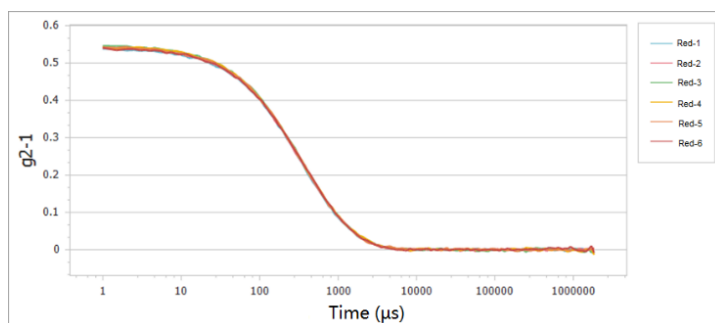


Figure 1. Correlation functions of red pigment sample for six measurements

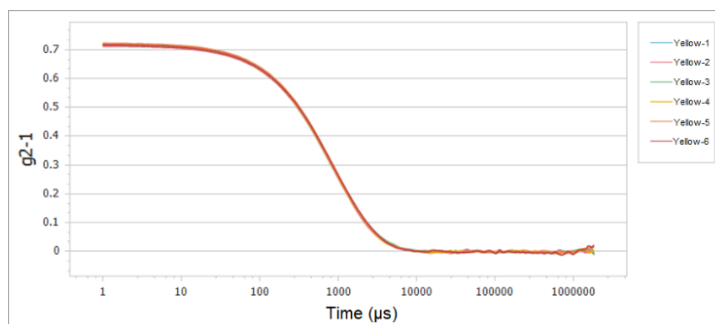


Figure 2. Correlation functions of yellow pigment sample for six measurements

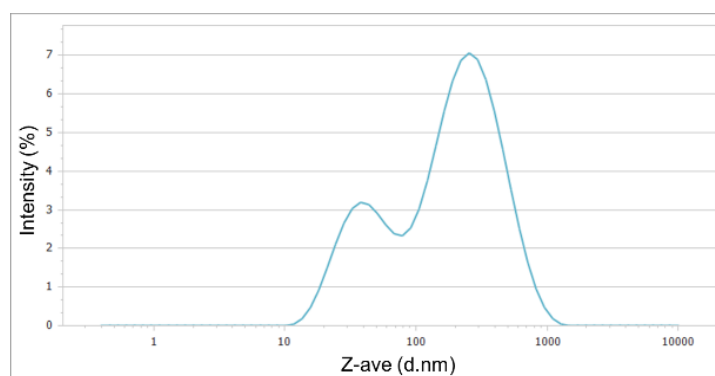


Figure 3. Particle size distribution of red pigment sample

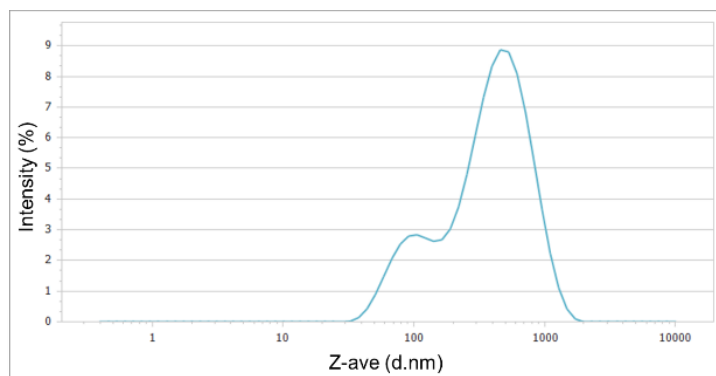


Figure 4. Particle size distribution of yellow pigment sample

Sample	Z-ave (nm)	PdI
Red Pigment	112.49 ± 1.42	0.528
Yellow Pigment	267.54 ± 2.69	0.391

Table 1. Particle sizes of two pigment samples

Figure 3 and 4 suggest that both particle size distributions of red and yellow pigments were wide with two peaks detected. The peak of small particles was at around 40nm, whereas the peak of large particles was at hundreds of nanometers.

As can be seen in Table 1, though both pigment samples were nanometer-sized, the yellow pigment sample had a larger particle size but a narrower particle size distribution than the red pigment sample.

Conclusion

The particle sizes of high concentration pigments (red and yellow samples) had been characterized successfully by the DLS technology of the BeNano 90 Zeta. Using the capillary sizing cell compatible with the BeNano 90 Zeta, even samples with high concentrations and low transmittance can be analyzed to yield reliable and accurate results.



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