Applying image analysis to support the development of particle sizing methods

PARTICLE SHAPE

PARTICLE SIZE

Dispersion

Dispersion is a key process in particle characterization. Getting a sample to a stable and reproducible state of dispersion is therefore an important part of method development. When measuring particles suspended in liquid, dispersion can be aided by surfactants, mechanical stirring and the application of ultrasound. In this application note we will focus on how the Mastersizer's Hydro Sight accessory (Figure 1), can aid method development by visualizing the sample dispersion. This allows you to see whether agglomerates are present in your sample and how stirring and ultrasound can disperse these into primary particles.

Figure 1: Mastersizer 3000 with Hydro Sight and Hydro EV

Case study: Toner

In this example the sample is a powdered toner. The particle size and size distribution of a toner is critical to producing high quality printed images. This case study looks at the process of dispersing a toner sample in water for measurement on the Mastersizer 3000, and how the Hydro Sight accessory can be used to visualize this process and gain a better understanding of the sample dispersion.

The sample was pre-dispersed in de-ionized water with a surfactant and then added to Hydro EV using de-ionized water as a dispersant. The Hydro Sight was connected between the Hydro EV and the Mastersizer 3000 and provided images of the dispersion at the same time as it was measured by the Mastersizer.

Following the dispersion process

Dispersion of the sample begins under the action of the Hydro EV pump and stirrer and initially we see some very large agglomerates in Hydro Sight images, accompanied by a wide particle size distribution measured by the Mastersizer 3000 (Figure 2). The action of the stirrer in the Hydro EV provides some dispersion energy and we begin to see loosely bound agglomerates break up. We can follow this process in a number of ways. The live trend in the Mastersizer 3000 software allows us to follow the decrease in particle size and increase in obscuration as agglomerates are dispersed into primary particles. We can also use the Hydro Sight Dispersion Index (DI) and Relative Standard Deviation (RSD) to follow this process, and identify when the sample is fully dispersed.

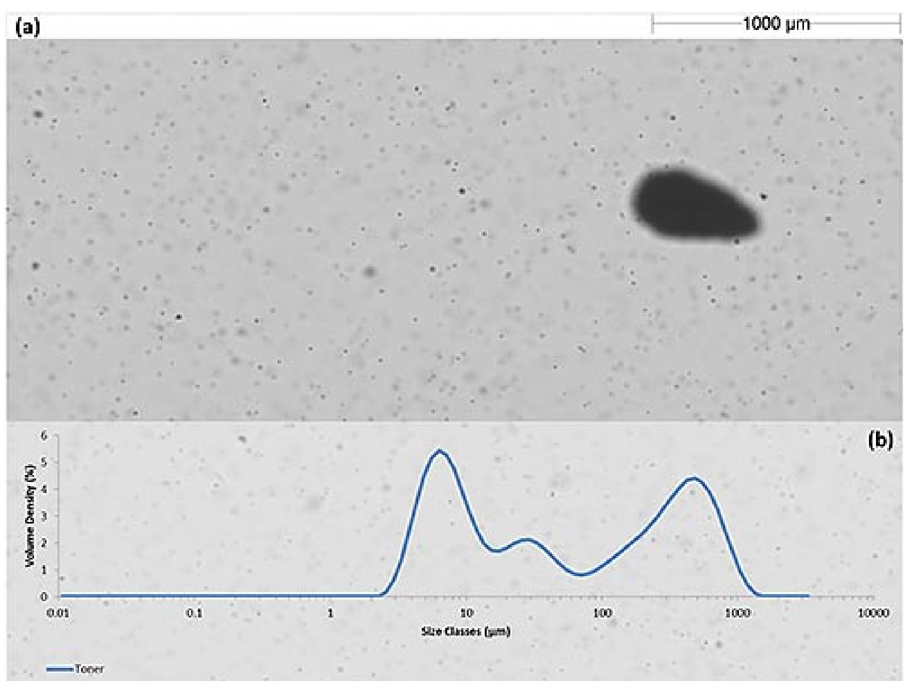

Figure 2: (a) Hydro Sight Image of toner in an agglomerated state (b) Mastersizer 3000 particle size distribution of agglomerated toner.

The dispersion index and relative standard deviation

The DI is a measure of the disorder within an individual image frame. A frame showing a high number of particles has a high degree of disorder, and a high DI. A frame containing very few particles is less disordered and has a low DI. Changes in the DI value therefore indicate a change in dispersion is occurring. We can also use the variability in the DI to track dispersion processes. For example, as a sample reaches full dispersion the size distribution narrows and becomes more stable the DI becomes more stable and it's variability, RSD, reduces.

As the sample disperses we expect the DI to increase as agglomerates are dispersed into primary particles and the number of particles per frame increases. In addition, as the sample reaches full dispersion the particle size distribution becomes narrower, the sample is more homogenous, and so the RSD of the dispersion index is expected to decrease.

Figure 3 presents a plot of the DI and RSD during the dispersion process for the toner sample. The DI increases as the sample is stirred and loosely bound agglomerates are dispersed, increasing the number of particles per frame. There is also a sharp increase in the RSD which reflects the changing state of the sample. During the first application of ultrasound there is a further significant increase in the DI, and another sharp increase in the RSD as the sample disperses further. The second ultrasound application shows a smaller increase in the DI indicating a small amount of further dispersion. The third ultrasound application does not show any increase in DI, indicating that the sample is fully dispersed, which is also confirmed by the stability of the RSD.

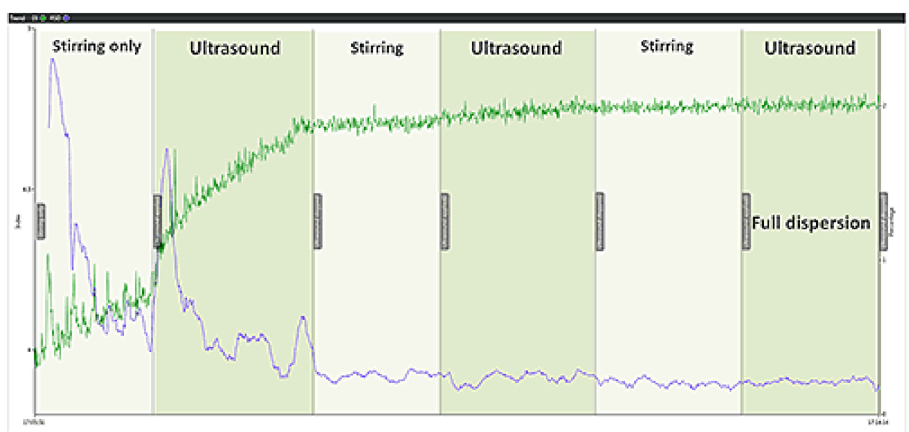

Figure 3: Following the dispersion trend using the Dispersion Index (DI) and Relative Standard Deviation (RSD)

The images displayed by the Hydro Sight after ultrasound show only fine particles, confirming that the sample is fully dispersed, (Figure 4). In addition the particle size distribution reported by the Mastersizer 3000 shows a narrow distribution of primary particles with a median size of 6μm.

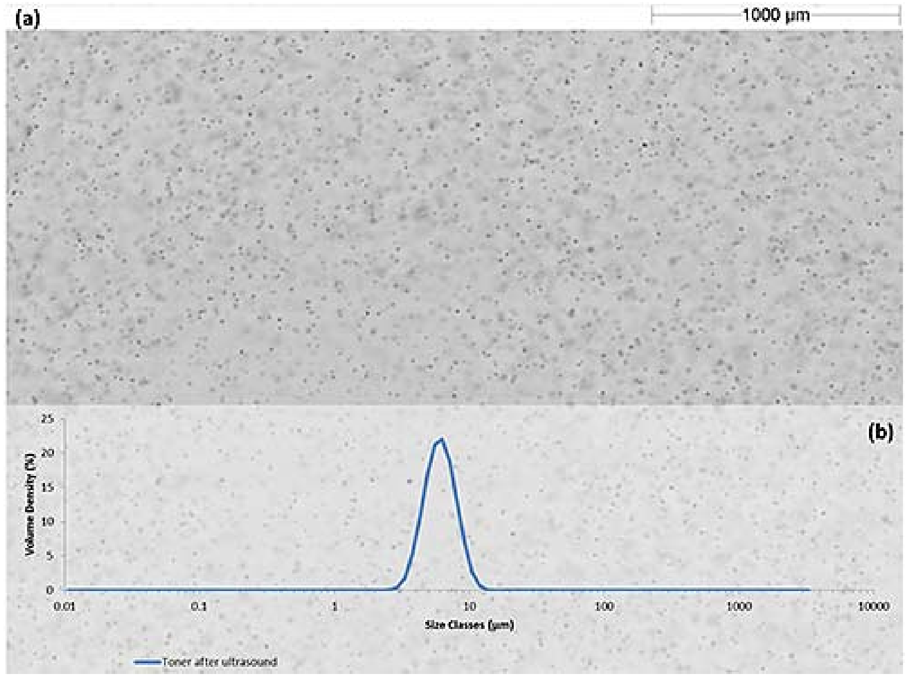

Figure 4: Hydro Sight Image of the dispersed toner sample (b) Mastersizer 3000 particle size distribution.

Conclusions

In this application note we have shown how the Mastersizer's Hydro Sight accessory can be used to visualize the state of dispersion of a sample. We have also followed the dispersion process using the Hydro Sight's dispersion index and relative standard deviation. Figure 5 shows Hydro Sight images of the beginning, middle and end of the dispersion process for toner. This additional information increases our understanding of the dispersion process and aids in the process of developing robust methods for laser diffraction measurements.

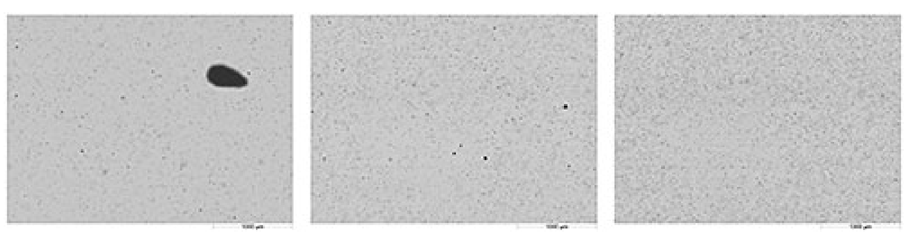

Figure 5: Hydro Sight Images from the beginning, middle and end of the dispersion process.

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