Quantitative Particle Size Distribution Analysis by Laser Diffraction and Differential Centrifugal Sedimentation

A comparison of techniques

Both Laser Diffraction (LD) and Differential Centrifugal Sedimentation (DCS) are used for quantitative particle size distribution analysis of samples in the sub micron region. LD can be described as a low resolution, wide dynamic range technique which is robust, fast and easy to operate. DCS, is a high resolution particle sizing technique, and is now similarly robust, fast and simple to use. Historically however, DCS has been limited in dynamic range; but with the introduction of speed ramping, the dynamic range capabilities of DCS are now much improved.

Figure 1 below demonstrates the LD and DCS analysis of a nominal 1 and 30 µm PMMA standard. A more or less equal quantitative response is demonstrated for LD and DCS, illustrating the quantitative capabilities of both techniques. However a fundamental difference between LD and DCS becomes apparent, since for both mono-disperse populations LD demonstrates much broader distributions. Moreover, DCS has been able to resolve the small satellite peaks at 1 micron relating to the clusters of 2 or 3 spheres.

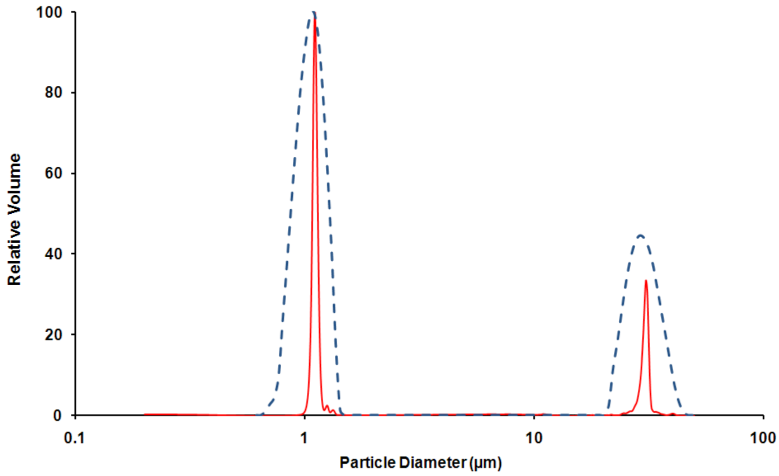

Fig.1 - *Differential particle size distribution of a mixture of 1.03 and 30.0 µm PMMA mono-disperse standard diluted in 0.1% Tween 20 in water and measured by DCS under 2000 rpm to 22000 rpm speed ramping conditions (calibration with 1 µm PMMA) in 4-12% (w/w) sucrose in 10% (w/w) PEG 20000 (——), and measured by LD (- - -).*

The reduced size resolution of LD is well known, and for most state-of-the-art LD instruments the minimum reportable peak width is typically in the range of 20-25%, therefore DCS is capable of achieving a far better resolution than LD. If high resolution is required, the decision to use either LD or DCS is not arbitrary. This is shown in figure 2 (below) for the speed ramping DCS and LD analysis of a mixture of 5 PMMA standards.

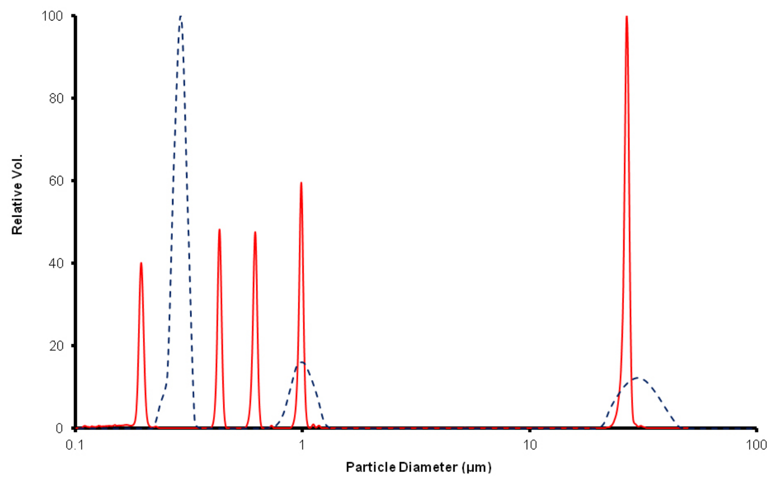

Fig.2 - *Differential particle size distribution of a mixture of 0.20 µm, 0.43 µm, 0.65 µm, 1.03 and 30.0 µm PMMA monodisperse size standards as measured by DCS (—) experimental conditions as figure 1; and by LD(- - -).*

Clearly DCS shows individual peaks for the 5 populations of PMMA spheres, and as before in figure 1, resolves the small satellite peaks that relate to the clusters of 2 or 3 spheres at 1 µm. LD demonstrates a more or less equal response for the 1 and 30 µm samples, but is unable to resolve the other PMMA populations.

Even if the standard size range of the LD instrument is extended by means of a second light source with a lower wavelength (blue light detection) as shown in figure 3, only the smallest population of 0.20 µm spheres is resolved, whereas the 0.43 and 0.65 µm populations still remain unresolved.

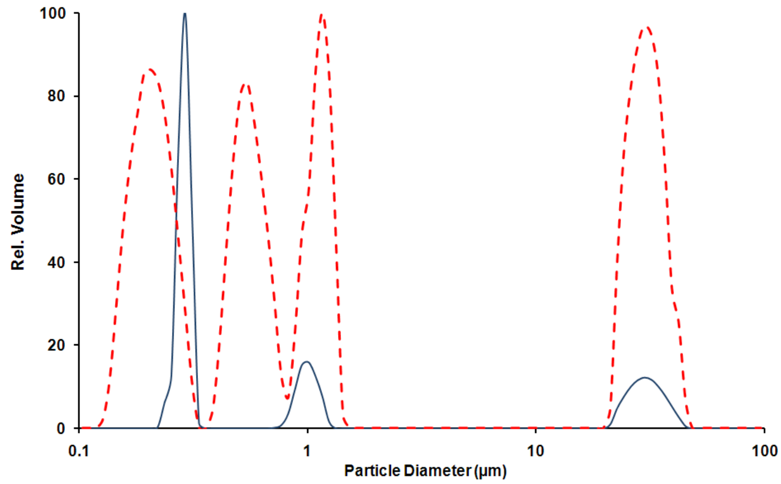

Fig.3 - *Differential particle size distribution of the same mixture (as figure 2) as measured by standard LD detection (——), and by LD with extended size range (- - -).*

This limited capability of most LD instruments to resolve the scattering information of mixtures of submicron particles is well known, and relates to the tendency of smaller particles to generate a less differentiated light scattering pattern, regardless of the angle of scattering. Since DCS calculates the size of the particles from the measured sedimentation time, size resolution is not an issue.

In referring back to Figure 2 there are other important features for DCS to be considered, namely (a) the relatively high upper size limit of > 30 µm, (b) the dynamic size range of more than 2 decades, and (c) the stable baseline over the entire dynamic size range. Therefore, in selecting a particle sizing technique DCS should be considered for high resolution applications and those requiring extended dynamic range.

Introducing the UHR CPS Disc Centrifuge

The Ultra High Resolution CPS Disc Centrifuge (Figure 4) nanoparticle size analyser utilises the technique of Differential Centrifugal Sedimentation (DCS). A sample is injected into a hollow, optically clear disc (Figure 5) containing a compatible density gradient, driven by a variable speed motor. The sample strikes the back face of the disc and forms a thin film which spreads as it accelerates radially toward the gradient liquid. On reaching the fluid surface, sedimentation of individual particles begins, and as particles approach the outside edge of the rotating disc, they block the detector light beam that passes through the disc. The change in light intensity is continuously recorded, and converted into a particle size distribution.

Fig.4 - *The UHR CPS Disc Centrifuge*

While DCS has been used to characterise particles for many years, recent technological advancements in the UHR CPS Disc Centrifuge now allow for routine analysis of nanoparticle distributions down to 2nm and varying in diameter by as little as 2%. Typical analysis times are in the order of 3-15 mins, with up to 40 samples analysed in a single run.

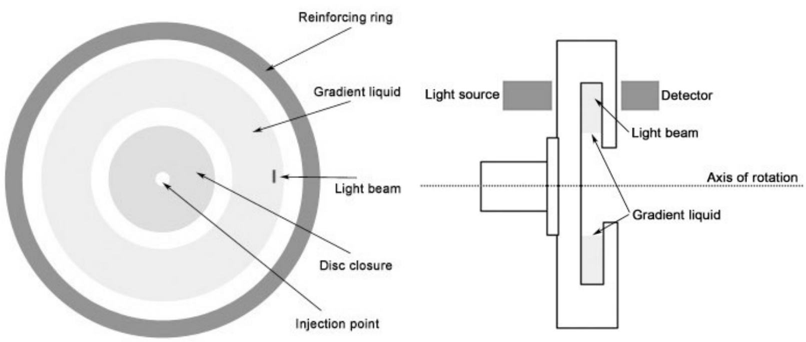

Fig.5 - *Schematic and cross-section of disc*

Want to find out more?

To learn more about high-resolution particle size characterisation using the UHR CPS Disc Centrifuge visit **analytik.co.uk/cps** (UK and Ireland) or visit **cpsinstruments.eu**.