Particle Size: Are Your Getting the Full Picture?

A True Story

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A number of years ago I was running a CPS Disc Centrifuge seminar at a University in the UK. A student came up to me with a small vial in her hand and told me that she was a final year PhD student and about to write up her thesis. She had been working with Silica particles for the last 3 years, mainly collecting particle size distribution results using Dynamic Light Scattering (DLS) and Low Angle Laser Light Scatter (LALLS) - and she was interested in running her sample on the CPS Disc Centrifuge.

After running the sample, I presented her with the result which showed two distinct peaks – clearly a bi-modal distribution (see Fig. 1). This caught the student by surprise as DLS and LALLS had only ever shown her one peak at around 4 microns, with all her analysis being based on how this single peak shifted. I then told her that if she took an average of the two peaks that the CPS Disc Centrifuge has highlighted, this would be around 4 microns. The CPS Disc Centrifuge's superior resolution power had enabled the peaks to be separated.

The CPS Disc Centrifuge also showed a tiny peak at around 0.5 micron. The student was not expecting to see this and initially suspected it must have been contamination on the disc, or carry-over from another sample. We ran the sample again together, making absolutely sure there was no contamination; the result was exactly the same as before. The student then requested for this "extra" peak be removed before I sent her the data as she only wanted the trace from 700 nm upwards because she would not be able to explain to an examiner where it had came from.

However, I paused and explained that we were looking at weight distribution. Imagine that you have a few big footballs and thousands of tiny peas. In terms of weight, which one is more significant? It would be the footballs; the peas would be insignificant. Whereas, if you convert it to a number distribution, what would happen then? The graph would show thousands of peas and only a few footballs. They key is to identify whether weight or number is more important however whatever the answer, you cannot just ignore the other. I then convert the graph to a number distribution to illustrate the point (see Fig. 2).

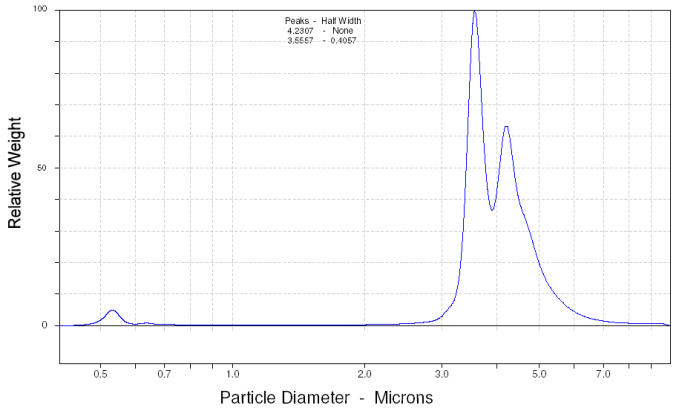

Fig.1

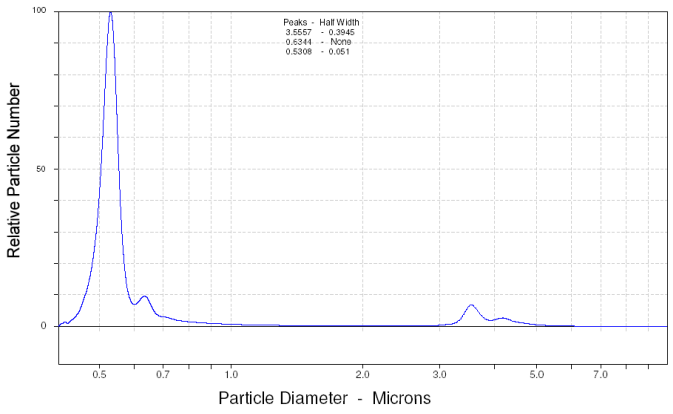

Fig.2

Even after explaining weight vs. number distribution the student was still adamant that the additional peak could not be from her sample, simply because she had never seen it before. I then explained that the reason she had never seen it was because when you have a mixture of big and small particles in your sample and measure by DLS for example, you only need a few large particles to completely skew the results because they hide the smaller ones.

Still not convinced, I persuaded her to perform Scanning Electron Microscopy (SEM) on her sample, as you should always try to obtain an optical image to confirm your results before drawing any hugely significant conclusions. The SEM image that the student came back with was very revealing (see Fig.3). It clearly showed particles of two different sizes - big ones from the two main peaks either side of 4 micron and many smaller ones at around 500 nm. The student was finally convinced at this point, realising she had based her entire thesis on misleading results.

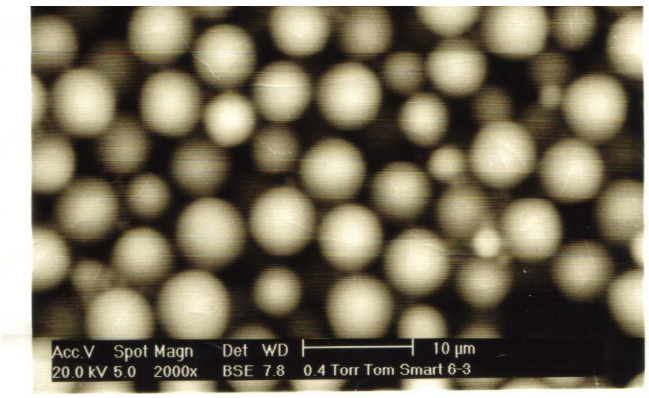

Fig.3

I spoke to the student two weeks later to check how she was getting on. She had managed to get in touch with the manufacturer of the LALLS instrument she had been using to check why the smaller particles were not shown in any of her measurements. The response she received was essentially that she had not told them she had expected smaller particles in the sample and that if she were to change the algorithm parameters when running the sample, she possibly would have seen those. (Unfortunately this was not possible post measurement.)

The Low Angle Laser Light Scatter uses a predictive algorithm; depending on what values you put into the software you can actually receive very different results. The problem with this is that if you do not know what your sample is to begin with, how do you know what parameters to put in? This illustrates the difference between using a predictive algorithm and using a real algorithm as utilised by the CPS Disc Centrifuge.

The CPS Disc Centrifuge actually separates the particles; it is in fact a separation technique that can be considered somewhat analogous to chromatography. It can separate particles down to around 2nm depending on particle density; then measures the particles as they pass a light source detector, providing a distribution in real time.

When characterising the size of your nano particles you cannot rely on just one or even two techniques, otherwise you may never see the full picture!

Intrigued about your own samples and wondering whether you are seeing the full picture?

To arrange a test analysis using the CPS Disc Centrifuge UHR, email **info@analytik.co.uk** or call us on **+44(0)954 232 776**.

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