Particle Size; Are you getting the full picture?

A true story by Dr Hiran Vegad, Analytik Ltd

I was running a CPS Disc Centrifuge seminar last year at a University in the UK. A student came up to me with a small vial in her hand and said, "I'm in the final year of my PhD and about to write up my thesis; I've been working with Silica particles for the last 3 years now, mainly collecting particle size distribution results using Dynamic Light Scattering (DLS) and Low Angle Laser Light Scatter (LALLS) - I'd be interested for you to run my 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. "Certainly," I said, "if you 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 has enabled the peaks to be separated."

Something else that the CPS Disc Centrifuge showed was a tiny peak at around 0.5 micron. The student said, "I did not expect to see that. It can't be part of my sample; surely it must be some contamination in your disc, or carryover from someone else's 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. 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.

I had to stop her there and say, "Hang on; what we're looking at here is a weight distribution. Imagine you had a few big footballs, but then you had thousands of tiny peas. In terms of weight, which one is more significant? It's those few footballs, the peas would be insignificant. However, if I convert this to a number distribution, what would happen then? The graph would show thousands of peas and only a few footballs. Which is more important to you - weight or number? Whatever the answer, you cannot just ignore the other." I clicked on the graph to convert it to a number distribution just to illustrate the point (See Fig. 2).

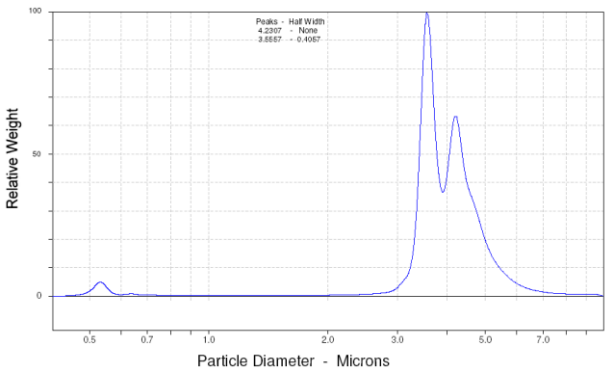

Fig. 1

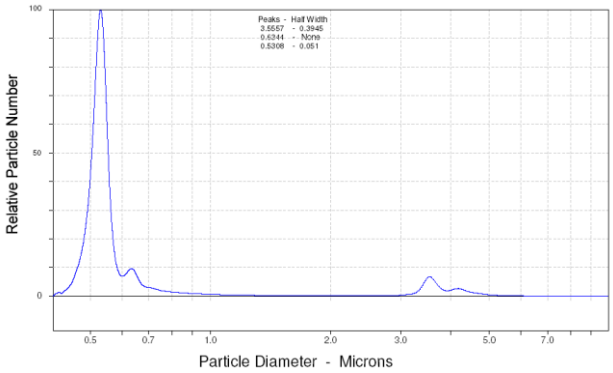

Fig. 2

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The student, even after this was still adamant that this additional peak could not be from her sample because she had never seen it before. I explained, “The reason you’ve never seen it is 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.”

The student still didn’t believe me. So I convinced 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.

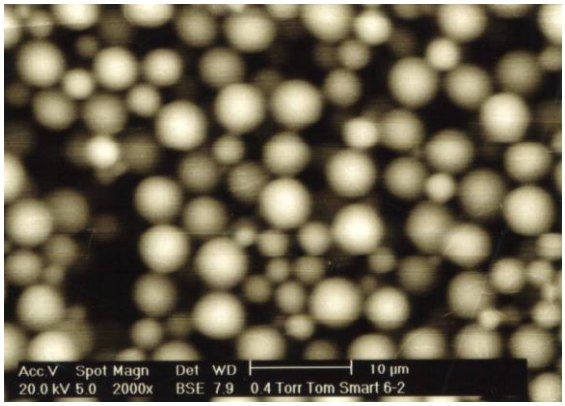

Fig. 3

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, “Oh, you didn’t tell me you expected smaller particles as well in the sample, if you were to change the algorithm parameters when you run your sample, maybe you would have seen those.” (Unfortunately this was not possible post measurement.)

I went on to explain; “So what does that tell you? 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. Now if you really don’t 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; it 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 will never see the full picture!

Intrigued about your own samples and wondering if you're seeing the full picture?
Email info@analytik.co.uk or call +44 870 991 4044 and we can easily arrange to run a test analysis using the new CPS Disc Centrifuge UHR.

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

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