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 North East of England. A female 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 DLS (Dynamic Light Scattering). Here are my findings so far, take a look and let me know what you think - 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 had only ever shown her one peak at around 4 microns, with all her analysis being based how this single peak shifted. "Certainly," I said, "if you took an average of the two peaks that the CPS has highlighted, this would be around 4 microns. The CPS's superior resolution power has enabled the peaks to be separated."

Something else that the CPS Disc Centrifuged 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 that when I sent her the data, that this "extra" peak be removed. 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)".

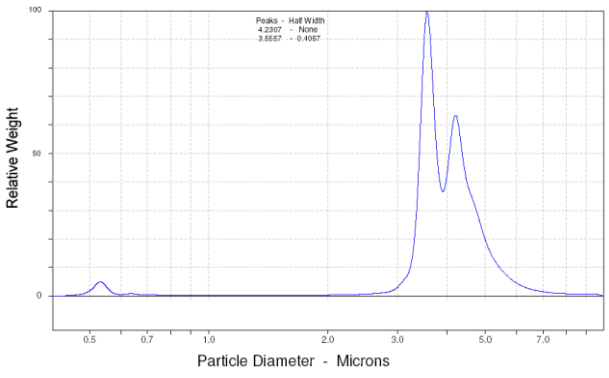

Fig. 1

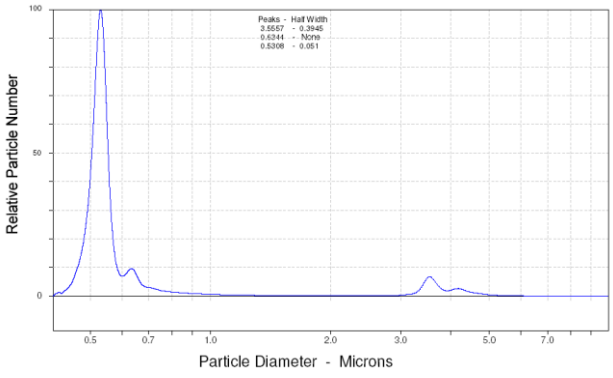

Fig. 2

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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, 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 go and perform SEM (Scanning Electron Microscopy) on her sample. Whatever you do, you should always obtain an optical image to confirm your results before you draw 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, your big ones from the two main peaks either side of 4 micron and loads and loads of smaller ones at around 500nm. At this point the student was finally convinced and realised that she had based her entire thesis on misleading results!

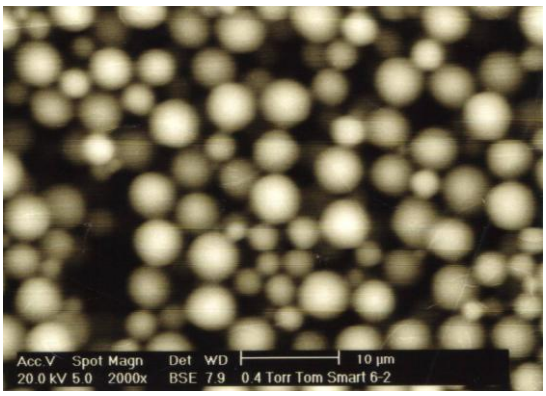

Fig. 3

I spoke to the student two weeks later to check how she was getting on. She had gone back to the manufacturer of the DLS 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? DLS (Dynamic Light Scattering) 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 actually measures the particles as they pass a light source detector, providing a distribution in real time. When characterising your particles you cannot rely on just one technique, otherwise you will never see the full picture!"

The End

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

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