Filter Challenge Testing with Latex 'Multistandard'

Determination of pore size distribution using the CPS DC

A précis of 'What's new in Filter Testing' by Dr G Rideal, Science Correspondent of the Filtration Society

There are two main methods of measuring the pore sizes in a filter: challenge testing and Porometry. The former is a simplistic and conceptually easy to understand process of challenging a filter with a liquid suspension or dust cloud of particles. Measuring the relative concentration of particles before and after the filter determines the filter efficiency while measuring the largest particles passing determines the cut point of the filter. Challenge testing has been perceived as being limited and inaccurate in that it could not measure pore size distribution, only the cut point or maximum pore size. The inaccuracy stems from the fact that test dusts had been used in the past. The test dusts were wide in particle size distribution and irregular in shape, which led to large variations from lab to lab.

The uncertainties of the test dust challenge test have been largely overcome by a new set of narrow distribution glass microspheres introduced by Whitehouse Scientific. By recovering the beads trapped in the pores of the filters it is now also possible to measure the pore size distributions. Below about 20microns however, the interactive forces between particles increase exponentially and particle agglomeration in air is difficult to prevent. The only method of bead transportation through a filter is by means of a liquid carrier.

The latest series of glass filter calibration standards go down to a few microns and it is not too difficult to measure the particle sizes in a suspension passing a filter using a number of techniques including microscopy.

Below 1micron however, it is difficult to make narrow distribution glass microspheres and polymer latex microspheres have to be used; but the main problem in challenge testing in the nanometre size range is finding instrumentation with sufficient resolution. A recent development addressing the resolution issue is the CPS Disc Centrifuge, see Figure 1. Strictly speaking the CPS DC is not new in that centrifuge sizing has been around for many years, however significant advances in high speed control (24,000 RPM) and data handling have revolutionised the method. Figure 2 shows the CPS disc in rotation during an analysis, the separated bands of differently sized particles can clearly be seen as they approach the detector.

Fig.1

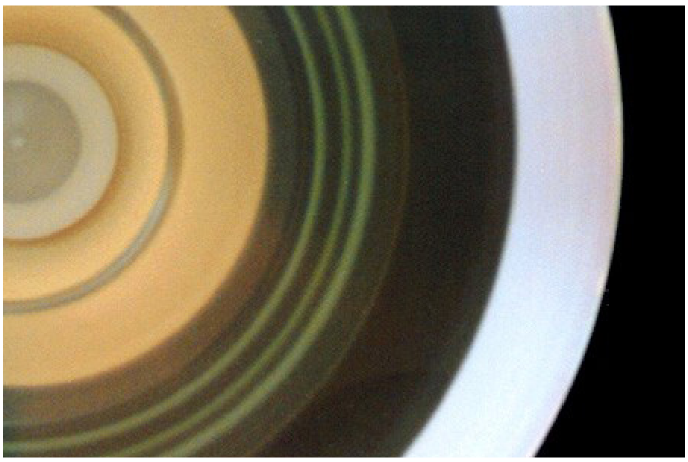

Fig.2

To measure filter cut point and pore size distribution, a multi-modal latex standard comprising of ten individual peaks from 10 nm to 1.5 microns has been prepared (BS-Partikel6). When a dilute suspension of this 'Multistandard' is drawn through a filter, the cut point can be readily detected from the sizes that have failed to pass the filter, while the pore size distribution can be determined from the degree by which the individual peaks have been suppressed, see Figure 3 (CPS DC Data).

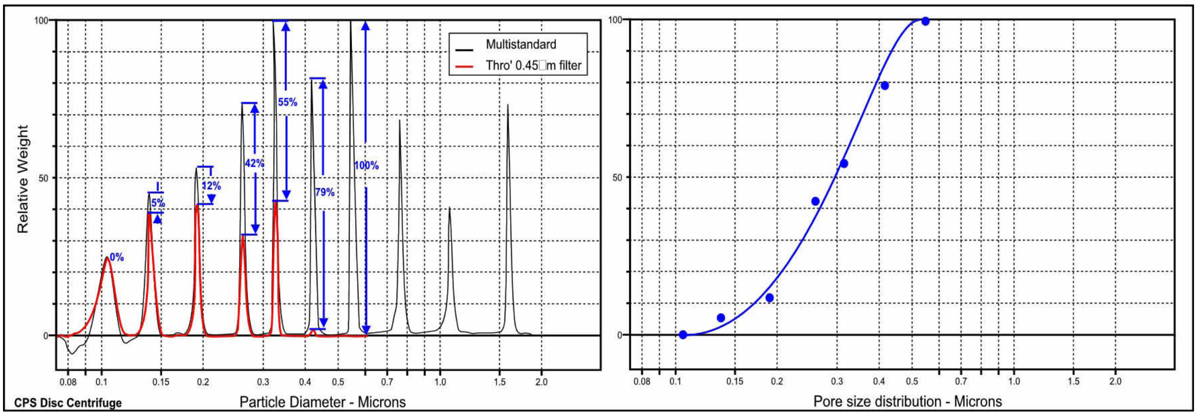

Fig.3

The CPS Disc Centrifuge can measure down to about 3 nm and is expected to play a major part in filter analysis in the next few years, especially in the pharmaceutical industry where the performance of filters can be a matter of life or death.

An Introduction to Differential Centrifugal Sedimentation

Differential Centrifugal Sedimentation (DCS) is an innovative, yet simple technique that has been 'reborn' in recent years. Previous limitations and difficulties with the technique of sedimentation have been overcome with advances in technology and some smart thinking regarding instrumentation and disc design. DCS is now a powerful tool in measuring nano particle size distributions down to around 3nm.

With the unique ability to resolve very close multi-modal particle distributions, even within 2%, and to distinguish extremely small shifts and changes in particle size, DCS is once more becoming a valuable particle characterisation tool. The term 'high resolution' is a commonly used expression in the world of particle sizing, however DCS really does achieve unparalleled resolution as can be seen when used in characterisation of nano particle coatings covered earlier in this article. Practical range of the technique is from around 3nm right up to 80micron (exact range will be dependent on density), however the real benefits over and above more traditional so-called nano particle sizing techniques are generally noticed below around 300nm.

These days, DCS has become fast, very simple to use, is highly accurate and reproducible. It can measure up to 40 samples on the same 'run', does 'speed ramping' for measurement of broad distributions in a single sample, and can even measure 'buoyant' or 'neutral density' particles (i.e. particles having a lower density to the medium in which they are dispersed). Due to the high resolution achievable DCS is ideal for resolving aggregates and agglomerates and to observe tiny relative shifts in peaks and tails of particle size distributions. It may also be used to measure absolute particle size too; however density of particle material must be known. It can even be used for quantitative measurements if optical property (refractive index) of the particulate is known. Number or weight distributions can also be easily calculated and displayed.

Want to find out more?

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