What's New in Filter Testing
a review of a Filtration Society seminar

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There are few areas in our lives that are not touched by filtration. We are surrounded by filters in the home, from tea bags and coffee makers to dish washers, washing machines and tumble driers, not to mention food products in our cupboards from mustard and flour to sugar and cereal, all of which involve some form of filtration.

The drive to work in your car requires oil, petrol and air filters for the engine, particulate removal for the exhaust and filters for the heating or air conditioning system. Indeed, every fabric in the construction of the car involves a filtration process somewhere down the line.

On the global scale, filters control the condition of the environment. Not just in the atmosphere with soot from diesel engines or smoke from coal fired power stations and incinerators, but in our waterways and seas with pollution from water treatment systems or industrial processes.

A matter of life or death

The appropriate use of filters and, more importantly, their measurement or certification, may make life more comfortable or convenient or may even have the laudable objective or extending the life of the planet, but the most serious short term impact of filtration is in the pharmaceutical industry where their use could affect our very lives in the next ten minutes not the next ten years.

The performance of a filter in a pharmaceutical application is therefore a matter of life and death.

There is therefore always a considerable interest in the pore size measurement of filters by the pharmaceutical industry. This was reflected last week in the popularity of the Filtration Society's Testing and Standards seminar and exhibition where record numbers of delegates and exhibitors attended.

This brief review highlights some of the very latest technologies in measuring pore sizes of filter media.

Challenge testing or Porometry?

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.

Porometry is not such a fundamental process in that it relies on the interpretation of gas flow through a 'wetted' filter. Wetting the pores effectively 'blind' the filter making it impervious to the passage of gas. However, as the gas pressure is raised, it overcomes the surface tension of the liquid and blows through the pores starting with the largest and finishing with the smallest. The gas pressure/flow rate curve can then be interpolated to determine the pore size distribution in the filter.

Historically, Porometry has had an advantage over the challenge test method in that it can measure a pore size distribution, not just the maximum pore size. However, one of the disadvantages is in the theory used to convert flow rate through pores to pore sizes.

The assumption made is that all pores are cylindrical, which is clearly not always the case. Furthermore, the 'black box' nature of the instrument makes it difficult to homologate instruments from different manufacturers.

Consequently, there are sometimes uncertainties in the pore sizes determined by Porometry and comparisons, not just between different manufacturers, but even for the same model of instrument can be poor. In the critical application in the pharmaceutical industry, uncertainty in pore size measurement of filters can be fatal.

Addressing uncertainties

This year has seen significant advances in the design of Porometers. The latest instrument from Benelux Scientific[1] introduced at the Filtration Society's seminar incorporates state of the art flow meters and pressure transducers enabling the pore size range to be extended down to 14 nanometres and up to 300 μm, figure 1.

Figure 1. A Porolux porometer from Benelux Scientific

The question of interlaboratory comparisons has been addressed by collaborative research between Tecan[2] and Whitehouse Scientific[3] who have produced and certified precision pores in metal foils. The first three Porometer calibration discs have diameters of 25 mm and pores of 3, 5 and 10 microns with deviations of less than 0.5 microns, figure 2. Initial comparisons with Electron Microscopy have been very encouraging. This new standard will at last enable direct comparisons to be made between all models and manufacturers of Porometers.

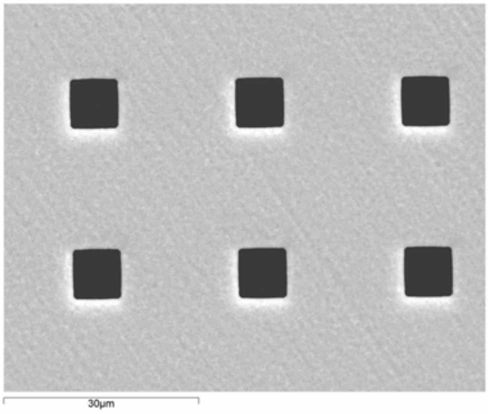

Figure 2. Electroformed pores in a Porometer calibration disc (Tecan)

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. In conjunction with a dry supersonic fluidisation device the cut points of filters down to 15 microns can be measured in just 2 minutes. By recovering the beads trapped in the pores in the filters it now also possible to measure the pore size distributions.

Below about 20 microns however, the interactive forces between particles increase exponentially and particle agglomeration in air is difficult to prevent, even with the high energy imparted by the sonic forces. 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, Coulter counting and light blocking methods.

High resolution sub-micron analysis

Below 1 micron 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.

Two recent developments addressing the resolution issue are a Disc Centrifuge (CPS)[4], figure 3, and a Brownian Motion tracking device from NanoSight[5], figure 4. Strictly speaking the former 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.

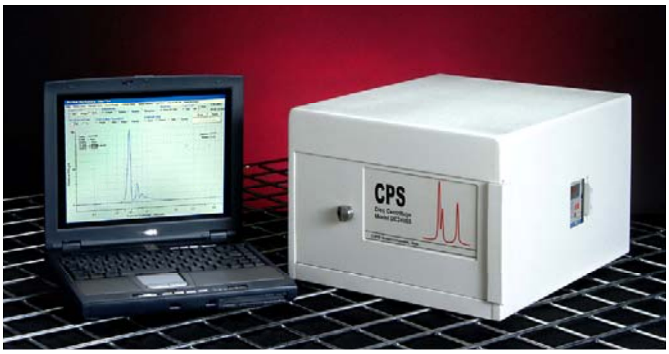

Figure 3. A CPS disc centrifuge for measuring particle sizes down to 5nm

Figure 4. The NanoSight submicron particle size analyser

To measure filter cut point and pore size distribution, a multi-modal latex standard comprising of ten individual peaks from 0.1 to 1.5 microns has been prepared (BS-Partikel[6]). 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, figure 5.

Interpreting random trajectories

The NanoSight technology is one of the most exciting developments in nanoparticle measurement. The system involves illuminating the particles with a high power laser light and tracking their random trajectories resulting from collisions with the molecules of the suspending liquid (the so-called Brownian effect). From the speed and distance travelled, a high resolution analysis of particle size distributions can be determined, figure 6.

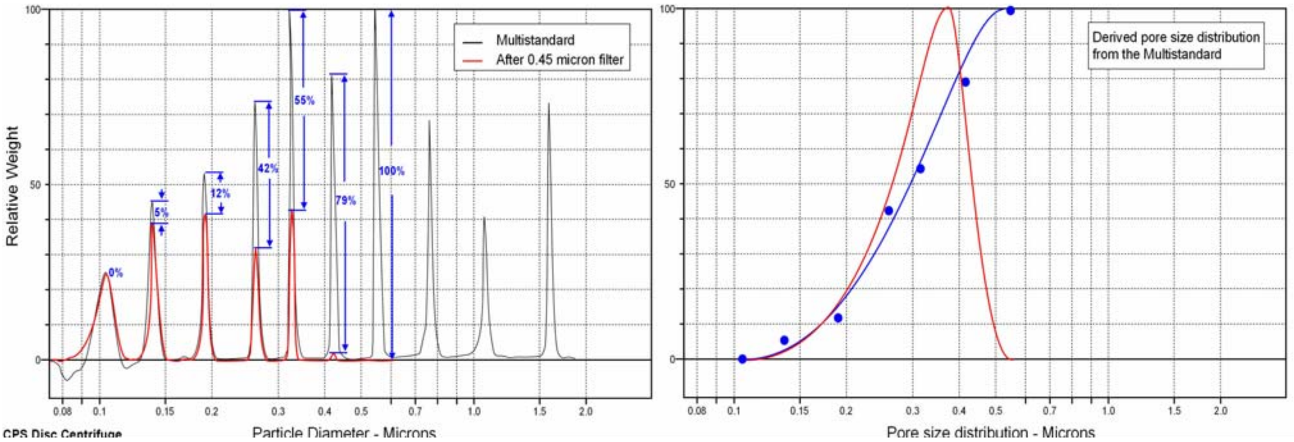

Figure 5. Determination of pore size distribution from a 'Multistandard' latex mixture

Both the CPS and the NanoSight technologies can measure down to about 5 nanometres and are 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.

Key to success

Whatever the pore size though, a key factor to ensure good interlaboratory comparisons of all the techniques is the availability of reference standard pore sizes for Porometry and precision particle size standards for challenge testing.

The mission of The Filtration Society to 'advance knowledge in the design and use of filtration' was certainly brought sharply into focus at the recent seminar on Testing and, who knows, may even save a few lives in the future!

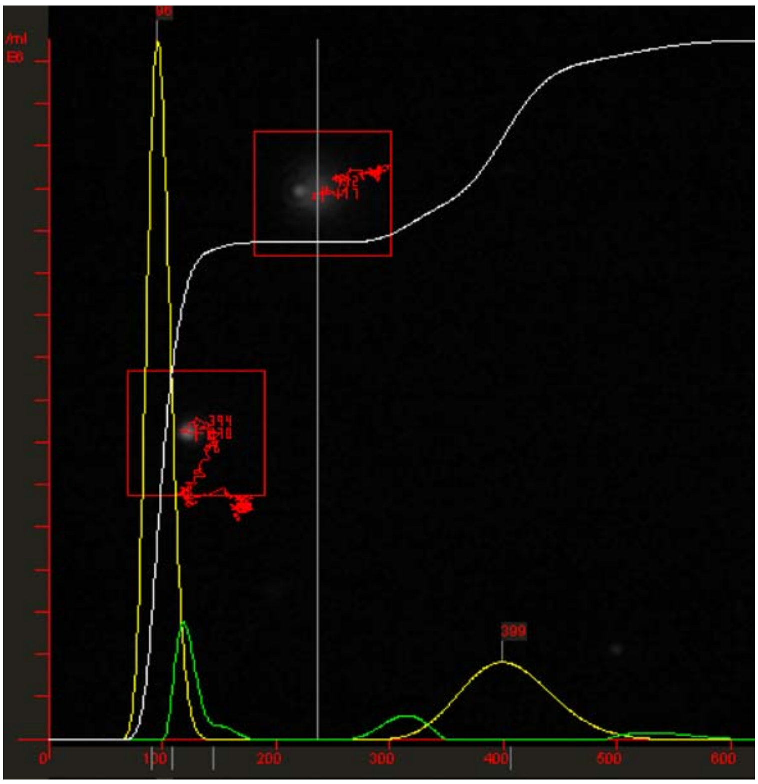

Figure 6. Molecular collision on particles can be tracked and converted to size using NanoSight technology

References

1. www.porometer.com
2. www.tecan.co.uk
3. www.WhitehouseScientific.com
4. www.cpsinstruments.eu
5. www.NanoSight.com
6. www.BS-Partikel.de

Filter Testing and Standards seminar – Chester 28th October 2008

7 main lectures and 6 poster lectures were presented. Bound copies of the proceedings can be purchased, see www.filtsoc.com

The FILTRATION Society

The Filtration Society

The Filtration Society is a 'not for profit' organisation founded over 40 years ago to advance knowledge in the design and use of filtration and separation processes. With extensive membership in over 30 countries it is recognised internationally as the premiere resource in its field. The journal FILTRATION and quarterly technical seminars ensure that the most up to date scientific and technical developments are brought to the attention of potential users.