Challenge Testing in Liquid Filtration –

New Technology Extends Filter Cut Points into the Nano-size region

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The Challenge Test Method

As the name implies, challenge testing involves challenging a filter with solid particles or oil mists of a specified size. The carrier fluid for the challenging particles can be either a gas or a liquid, the selection of which depends on the final application of the filter.

For example, if the application was in a compressed air system where there is the possibility of oil mist contamination from the compressor, a liquid suspension of particles would not realistically reflect the performance of the filter medium and an air carrier for the particles would be much more appropriate.

Similarly, if the filter is to be used in water purification, it would not be sensible to choose air as the carrier for the challenge particles. The challenge test method must therefore be appropriate to the application of the filter.

Testing filters in the dry stage has benefited in recent years from the advances in particle size measurement by optical and electrostatic methods, which cover a wide range of particle sizes down into the nanometer size range. Aerosols of oil mists or sodium chloride crystals for instance can be generated and drawn through the filter by vacuum to measure filter efficiencies. This is done by comparing the particle sizes before and after the filter element.

The instruments used tend to be centered around specific industries and their associated ISO specifications. In this review, only liquid based challenge tests are considered.

Liquid based challenge testing

Fig.1: Wet Tester. A simple apparatus for challenge testing a filter

To test the cut point of a filter, dilute suspensions of the challenging particles must be transported through the filter medium. If the particle concentration is too high, a secondary filtration through cake build-up takes place, which will undermine the true cut point of the filter.

The apparatus does not need to be complex; a simple filter holder over a Buchner flask is all that is required, figure 1.

The Challenge particles

Particle detection and measurement are critical in determining the cut point of a filter. The broader the particle size distribution of the challenge particles, the more difficult it is to find the largest particles passing, simply because they are present in such low concentrations.

To enhance the resolution of the method, narrower particle size distributions are required. It is then much easier to find the cut point because more particles are available for detection.

The particle shape of the challenging particles is also very important. Elongated particles have three dimensions, which can give ambiguous results dependent on the method of measurement, but spherical particles only have one dimension so measurement of the true pore widths is less uncertain, figure 2.

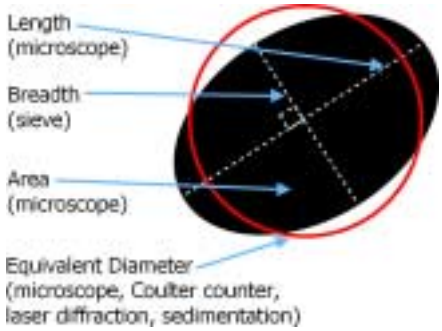

Fig.2: The size of an irregular particle depends on the method of measurement

A typical set of challenge particles based on glass microspheres is shown in table 1.

Table 1. Glass microspheres used in challenge testing filter media

0.1 - 1	0.5 - 2	2 - 6	5 - 8	7 - 10	10 - 14	12 - 19	16 - 25
25 - 38	36 - 59	53 - 75	63 - 86	75 - 103	106 - 147	127 - 175	180 - 248

Despite the uncertainty relating to particle shape and size distribution, irregular test dusts are still produced for specific applications such as contamination in hydraulic systems and are written into several ISO standards. This topic will be the subject of a later review.

Methods of particle sizing

In recent years there have been enormous strides forward in the sizing and counting of fine particles. The Coulter counter was the first instrument to offer an automatic method of particle measurement and is capable of measuring from one to several hundred microns.

Optical or light blocking methods soon followed and covered the same size range as the Coulter Counter but had the advantage of not requiring electrolytes to make the suspensions electrically conductive.

Both methods offer the high precision required to measure the low concentration of large particles that often emerge on the down side of the filter. These particles reflect the maximum pore size or cut point of a filter.

Although optical microscopy is an obvious choice for 'seeing' the actual particles passing a filter, it has hitherto been limited by the laborious data collection that is essential for strong statistical results. There are now, however, many image analysis packages that

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enable the high speed counting of millions of particles and thereby produce very robust statistical information from the microscope.

An additional advantage of the microscope method of particle size analysis is that a shape threshold can be implemented to exclude any elongated particles that may be present in the challenge particles, which would give erroneous results.

The main disadvantage of microscopy is that it has a lower limit of about 2 microns, so cannot be used in challenge testing of the very finest filters now being used in industry.

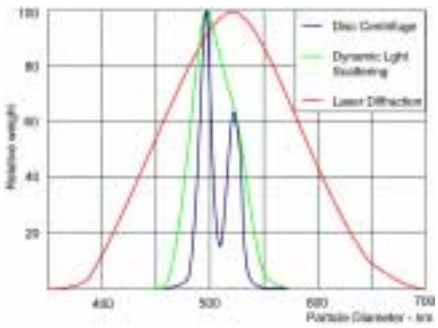
Into the sub-micron region

Accurately measuring sub-micron particles in suspension had been a problem for many years but the advent of laser technology soon brought particle measurement into the sub-micron region. The system enabled the movement of particles by Brownian motion to be tracked and measured. Deconvoluting the resultant laser response produced the first Photon Correlation Spectrometers (PCS), later to be re-named Dynamic Light Scattering.

Although PCS proved very useful in applications such as paint technology, its use in challenge testing is limited because the modelling process required to produce particle size distributions is not sensitive to individual particles at the extremes of the particle size distribution.

Concurrent with the innovation of the laser was the development of a very old technique: centrifugation. Sedimentation from a homogeneous suspension was not a very sensitive method because of a combination of the concentration of particles required in the suspension and the ability to quantitatively measure the sedimentation rate.

However, a new method of differential sedimentation was developed, which finally gave the sort of resolution required for a sub-micron challenge test. In this method, particles are constrained on the surface of a spinning liquid and released individually according to their size, large ones first followed by smaller ones, figures 3 and 4. This powerful technique is accurate down to nanometer sizes and is expected to play a major role in challenge testing in the future.



Above: Fig.3: The Line start disc centrifuge for measurement of nano-sized particles

Left: Fig.4: Disc centrifuge resolution compared to Laser Diffraction and Photon Correlation Spectroscopy

Of course, one of the earliest methods of accurately looking at individual particles was the electron microscope. This 'seeing is believing' method brought with it all the confidence associated with optical microscopy and so was regarded as one of the few, if not the only absolute method.

Fig.5: A new generation of bench top Electron Microscopes will have a major impact on filter testing by the challenge test method.

The limitations of Electron Microscopy include: the time required in taking enough pictures of particles to obtain statistically robust data, the sheer physical size, and consequently the laboratory space needed for the equipment, and the skills required in using it.

It was therefore very exciting to see the latest evolution of the electron microscope developed by Hitachi. The TM-1000 is no bigger than a desktop computer and can record images as easily as a digital camera so no special room of highly trained technicians are required, figure 5. It can even measure suspensions so the laborious task of sputter coating can be eliminated. A further advantage of the system is that it can examine the filter medium as well as the challenge particles passing through.

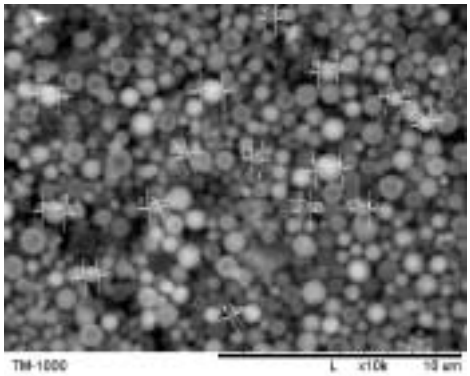


Fig.6: 0.1-1 micron microspheres passing a 0.2 micron filter indicate a problem with the filter. (Hitachi TM-1000).

Figure 6 shows the down stream particle sizes from a nominal 0.2 micron filter. The fact that microspheres up to 1 micron pass the filter indicate that there is a fault in the filter or that the filter medium has been wrongly labelled.

Finally, one of the most innovative methods of submicron particle size analysis is based on a development of the Dynamic Light Scattering method. In this technology, a collimated laser beam is focused on a dilute suspension of the particles where their speed and direction under the influence of Brownian motion can be tracked.

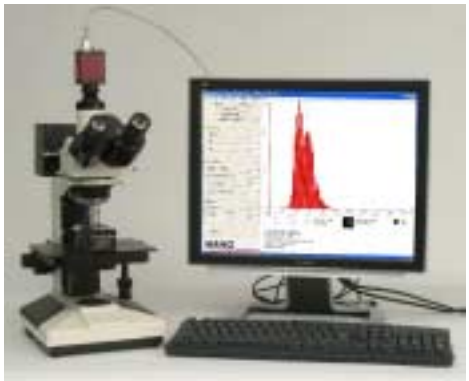


Fig.7: A new sub-micron particle size analyzer from Nansight produces high resolution from Dynamic Light Scattering.

The fluorescing particles from the down side of a filter, for example, are simply viewed through a special cell on a conventional laboratory microscope where they can be, counted and analysed using image analysis software. The system is capable of measuring down to 10 nanometers and, unlike the correlation theory used in Dynamic Light Scattering, has the high resolution capability required by the challenge test method, figure 7.

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Conclusion

While Porometers have been used for some time in determining efficiencies, the results are based on flow rate theory through a filter medium and can sometimes give equivocal results. Challenge testing on the other hand is an absolute method, in that particles that pass a filter are physically measured.

Confidence levels in the results are then related only to the confidence levels in the particle sizing method. An additional advantage is that the particles can be certified according to an international unit of length, for example NIST, so the results are also traceable.

With an increasing interest in nano sized particles, there is a corresponding desire to find high resolution techniques of particle size analysis, which are essential in all challenge testing methods.

Some of the techniques described herein, both developments of established methods such as centrifugation and electron microscopy, as well as totally novel methods such as the Nanosight will undoubtedly find their way into the front line of filter challenge testing in the future.

About the Author

Dr. Rideal graduated from Lancaster University, England. He is the author of several patents describing the construction of inorganic materials such as foams, films and costings from nano mineral particles. He was the founder of Whitehouse Scientific in 1983, a company specializing in particle size standards, which was selected as the top certification laboratory by the European Bureau of Certified Reference. Dr. Rideal was Chairman of the Filtration Society for two years. A year ago, he joined with other prestigious members on Filtration News ' Editorial Advisory Board'.