Measuring Filter Performance By Challenge Testing

Part 2: Pore Size Information

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Introduction

Challenge testing is the technique of presenting a range or particles to a filter that are wider than the expected cut point. The particles that pass the filter are then measured. The cut point is described as the pore size in a filter required to clarify a fluid suspension (in air or liquid) and must not be confused with the 'maximum' pore size. Part I of this article (Filtration News, Vol. 23, No 4, July/August 2005) reviewed pore size definition, types of apparatus that can be used and the examined the importance of particle shape and size distribution in obtaining accurate and reproducible results. By way of introduction, the equipment and standards are summarised below.

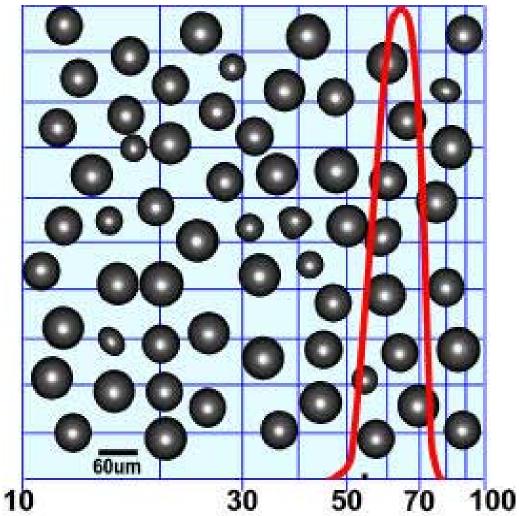

Figure 1. 53 – 75µm filter calibration

Challenge test particles

For optimum results, challenge particles should be spherical. This means that the derived pore size is expressed in terms of its equivalent spherical opening – an unambiguous parameter. Secondly, the challenge particles should have a narrow (but not monosize) distribution. This enhances the resolution of the challenge test. Thirdly the challenge test particles should be accurately sub-divided for individual tests using a spinning riffler, which minimises sampling errors and operator bias. A typical size distribution of a challenge test reference standard is shown in figure 1, while a typical range of standards is shown in table 1.

Table 1: Band widths of sonic filter standards (µm)

2-6	5 - 9	8-12	9-16	12 - 22
16-25	20-34	26-36	31-46	36-55
45-62	53-73	63-86	75-103	80-123
106-147	127-175	151-209	180-248	214-295
252-346	304-417	360-498	383-591	484-700

A very important aspect of testing filters with real particles is that the dimensions can be related to internationally recognised standards such as the National Institute of Standards and Technology (NIST). The challenge test particles should be measured either by sieving, although the precision electroformed sieves must be used, or directly my microscopy.

Particle shape

The advantage of using NIST traceable electroformed sieves for calibrating the filter standards is that the beads are measured by width, the parameter that determines whether or not they will pass the filter,

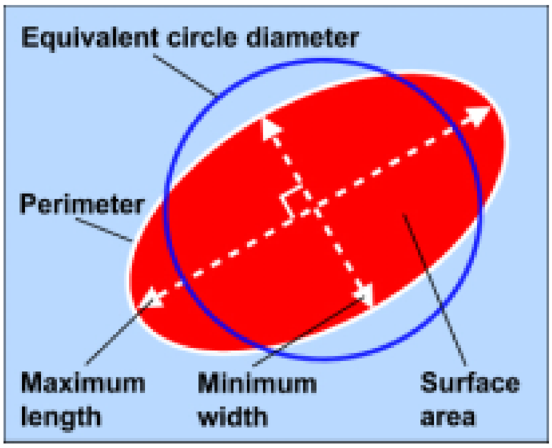

Figure 2. Particle dimensions

Most particle sizing techniques measure the equivalent spherical diameter, which is bigger than the width for non-spherical particles, figure 2. In the case of spherical particles, there is only one dimension so all methods should give the same result. Nevertheless, if microscopy and image analysis are used as the measuring technique, a threshold should be employed to exclude measurement of the few non-

Continued on Page 10

spherical particles that may be present. For example, beads with a maximum to minimum ratio of greater than 1.3 should be eliminated from the analysis.

Certifying challenge microspheres

In its simplest form, the challenge test microspheres do not need to be certified. The operator simply measures the particle size distribution of the beads passing. However this method requires expensive equipment; a microscope and image analysis software.

An alternative method is to measure the weights of the microspheres at different diameters and construct a calibration graph, the only equipment required for a cut point being a balance. The percentage of the beads passing the filter is used to determine the cut point, figure 3.

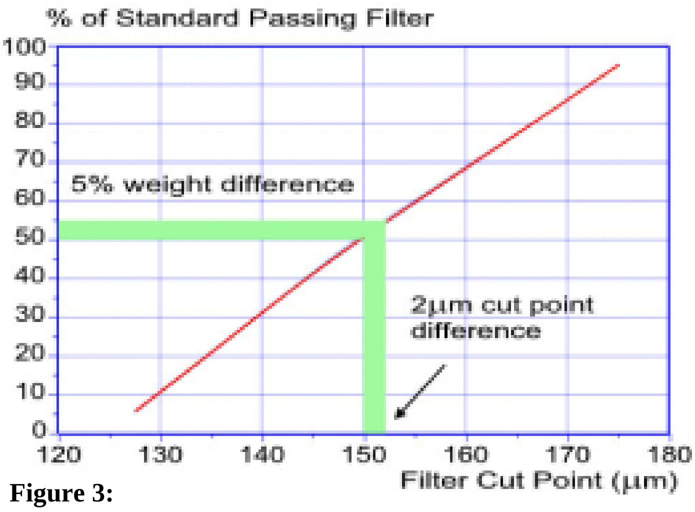

Figure 3:
A calibration graph to determine filter cut point Challenge test

The advantage of using narrow particle size distribution can clearly be seen in figure 3, a 5% weight difference of the beads passing only corresponds to a 2µm difference in the cut point.

Challenge test methods

Once an accurately defined set of filter standards has been prepared, the next stage is to 'challenge' the filter surface with the particles. The methods used are largely dictated by the pore size in the filter medium. For bead sizes over about 50µm a simple shaking device such as a sieve shaker may suffice because the microspheres behave independently, however as size decreases, increasing particle interaction comes into play and a more vigorous challenging method is required.

In the Sonic method, energy is applied to the particles rather than shaking the mesh, so inter-particle attractive forces are overcome. This enables pore sizes down to about 5µm to be measured for certain open structure fine filters, figure 4. However, for more complex filter media with low air permeabilities, Sonic challenge testing may be limited to a minimum pore size of about 15µm. The great advantage of the Sonic challenge test method is that it takes as little as 1 minute to perform.

Fig. 4: A dry filter challenge tester (Gilson)

It is important in challenge testing to be sure that a realistic end point has been reached; any sieving or filtering process is governed by the largest pore present. It may take 5 minutes for most of the beads to pass a given filter but if there is one pore in a million that is grossly oversize, it could take 5 days for all the challenge test beads to find the opening as pass through. The filter cut point is not therefore the 'absolute' maximum pore size (more later).

Fig. 5: A suspension based filter testing apparatus

For pore sizes below about 20µm or where there is a depth filter mechanism taking place, for example in felts, a wet transportation method must be used for the challenging microspheres. In this very inexpensive method, all that is required is a split filter holder, a Buchner flask and a small vacuum pump, figure 5.

Care must be taken in this method to ensure that the concentration of particles in the suspension is not too high otherwise the particles themselves may 'cake' so reducing the effective pore size and flattering the performance of the filter. To overcome caking a back-flush system with clean water is recommended.

Defining the filter cut point from the Sonic Challenge Test

To understand the cut point more precisely, one must analyse the particle size distribution of the standard before and after it has passed the filter and compare size of the beads passing to the cut point, figure 6. In

Continued on Page 12

Measuring Filter Performance . . . Continued from Page 10

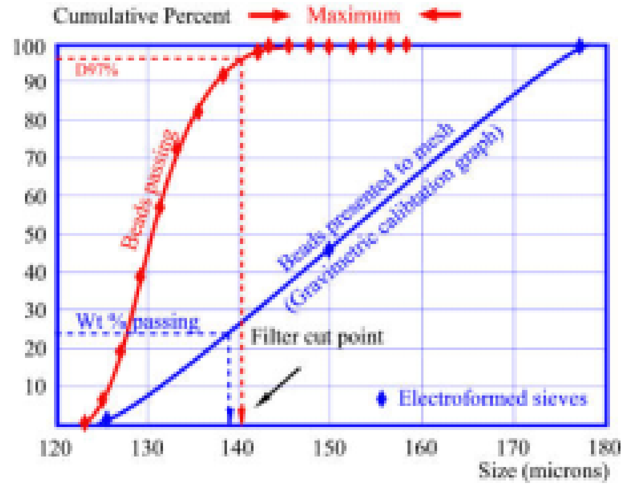

Fig 6. Microscope analysis of the beads passing compared to the sonic test result

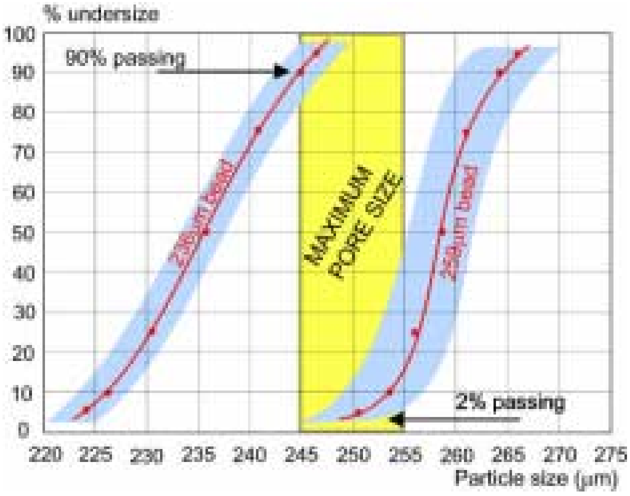

Figure 7. Challenging a 243µm cut point filter with ultra narrow particle size analysis

this instance for a complex woven stainless steel mesh, 29% of the standard passed the filter, which equates to a cut point of 140 microns. When the microscope analysis of the standard passing the filter is superimposed on the calibration graph, the cumulative percent undersize at 140 microns corresponds to a percentage of approximately 97%. This suggests that the filter would be 97% efficient in trapping particles above 140 microns. In other words, the cut point corresponds to a pore size close to the maximum pore size in a filter.

To confirm the relationship between the cut point and D97 of the beads passing, a range of woven meshes were analysed, table 2.

Table 2. Comparison of filter cut points with analysis of the beads passing a woven mesh

Nominal Size (µm)	85	115	175	200	250
Filter standard used	75-103	106-147	180-248	214-295	252-346
Filter cut point	90	137	202	243	296
Beads passing (D97%-number)	98	129	209	247	299

It can be seen that, in most cases, the cut point corresponds closely to the D97 of the beads passing. It is worth pointing out at this point that, if the particle size distribution of the challenging beads was broad, then the number averaging of the data would drag down the D97 value giving a false reading of the filter pore size.

The big question remaining however is how much larger are the maximum beads in the final 3% of the beads that pass the mesh. This can be determined by challenging a filter with ultra-narrow size distribution glass microspheres. The 200µm nominally rated filter from table 1, having a cut point of 243µm was challenged with 2 'Monospheres: 236 and 259µm, figure 7.

While 90% of the smaller standard passed, only 2% of the larger monodisperse standard passed, but within those beads, the largest size detected was 264µm so the ' absolute maximum' pore size is about 264µm. This is 9% above the cut point. The projected 'maximum' pore size for woven meshes is therefore approximately 10% above the cut point.

Note. The concept of 'absolute' maximum in filter testing should be avoided unless the application is highly sensitive, for example, removing biological contaminants in medical applications where just one virus in 1000's of litres could be fatal. For most processing applications, engineers are looking for good product recovery and a recovery in excess of 97% is good enough for most applications.

Measuring filter cut points below 20µm

As indicated above, below about 20µm a wet suspension system is recommended in order to eliminate inter-particle attractive forces that occur in the dry state. In this method, a challenge test reference standard is presented to the filter and the particle size of the penetrating particles analysed, figure 8.

In the particle size analysis of the beads passing it is essential that only the spherical particles in the filter standard are measured and not any elongated beads, which could distort the results (see figure 2).

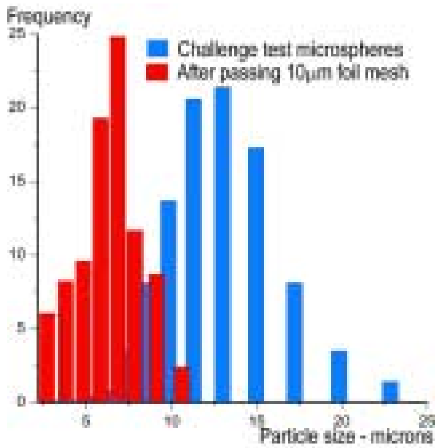

Figure 8. Challenge testing a 10µm metal foil filter

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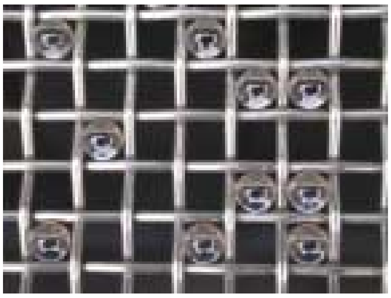

Fig 9. Near mesh beads lodged in a test sieve

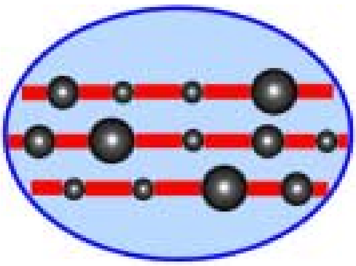

Figure 10. 'Near mesh' capture of calibration microspheres by a filter

Metal foil filter media, as shown in figure 7, are often used in ink jet printers. With today's high resolution printers, it is essential that the filter screens out any large particles, which cause nozzle blockage and expensive down time. Many filter media manufactures are now using the challenge test method to certify their products.

Pore size distribution from challenge testing

It has been known for some time that a test sieve can be calibrated using a near mesh method. This process involves challenging the test sieve with a range of particles, which cover the anticipated range of apertures. Microspheres are the ideal challenging material because there is no shape implication. After shaking the microspheres over the surface of the sieve, oversize particles are removed by inverting the sieve and gently tapping.

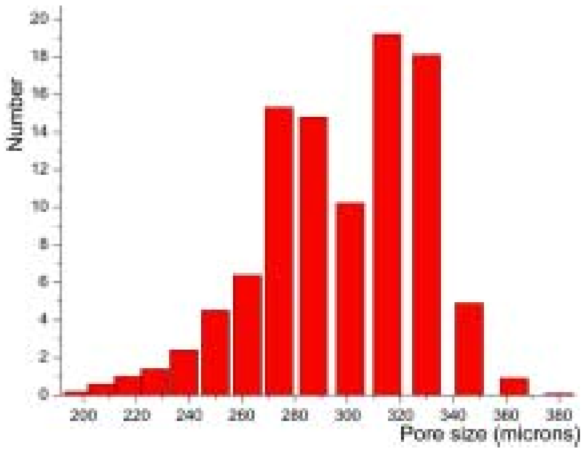

Figure 11. Challenge testing reveals fine pore structure

The tightly wedged microspheres between the wires are then released by gentle brushing and analysed by microscopy, figure 8. The theory is that these 'near mesh' microspheres replicate the range of apertures in the sieve (for more details see G Rideal, Particles and Particle Characterization Systems, 17 (2000) 1- 7 Wiley).

This technique has been developed by Whitehouse Scientific to measure the pore size distribution of more complex 3-dimensional woven filters such as Dutch and twilled weaves whose internal pore sizes cannot be accurately determined by any other means.

The pore structure is penetrated by the microsphere standard, figure 10 and, after inverting and brushing the filter surface to remove oversize beads, the internal near mesh beads are recovered and measured. This process has been able to reveal fine detail within the filter medium.

Firstly, the accuracy of the method is able to detect bi-modal pore size distribution in a filter mesh, figure 11. In quantifying the pore size distribution, the beads at 3% of the cumulative undersize distribution correspond to the minimum pore size, while the beads at 50% and 97% correspond to the average and maximum pore size respectively. For reasons stated in part 1 of this paper, the sizes at 0% and 100% are too unreliable to be used as the 'absolute' minimum and maximum pore size.

Comparison of projected and near mesh maximum pore sizes

It was seen above that a projected maximum pore size could be calculated from the cut point in certain cases (projected maximum pore size = cut point + 10%). Another method of checking the validity of the projected maximum pore size is to compare it with the 'near mesh' method, table 3.

Table 3. Maximum pore size from Sonic and 'near mesh' methods

Sonic challenge test		Near mesh challenge test		
Cut point (μm)	Projected Max pore D_{97} (μm)	Minimum pore size P_3 (μm)	Average pore size P_{50} (μm)	Maximum pore size P_{97} (μm)
212	233	182	209	233

It can be seen from table 3 that firstly, the pore size distribution of a Dutch woven mesh is quite narrow: 182 – 233μm. Secondly, the cut point is close to the average pore size and, finally, these is good agreement between the projected maximum from the cut point and the maximum measured from the beads penetrating the filter mesh.

Identifying 'open' and 'closed' pores

There is not always good agreement between the two methods of measuring the maximum pore size. That is because the projected maximum pore size is based on the beads *passing* the mesh, while the maximum pore size measured from the 'near mesh' method is based on the pores *within* the mesh.

Continued on Page 15

Measuring Filter Performance . . . Continued from Page 14

If the pores within the mesh do not exit at the same diameter they are known as ‘closed’ pores and will only allow a bead to penetrate so far down before it wedges between the wires. If the diameter of the pore is constant all the way through the mesh, it is an ‘open’ pore.

Comparing the two methods of measuring maximum pore size therefore reveals whether there are ‘closed’ pores in the filter, figure 12.

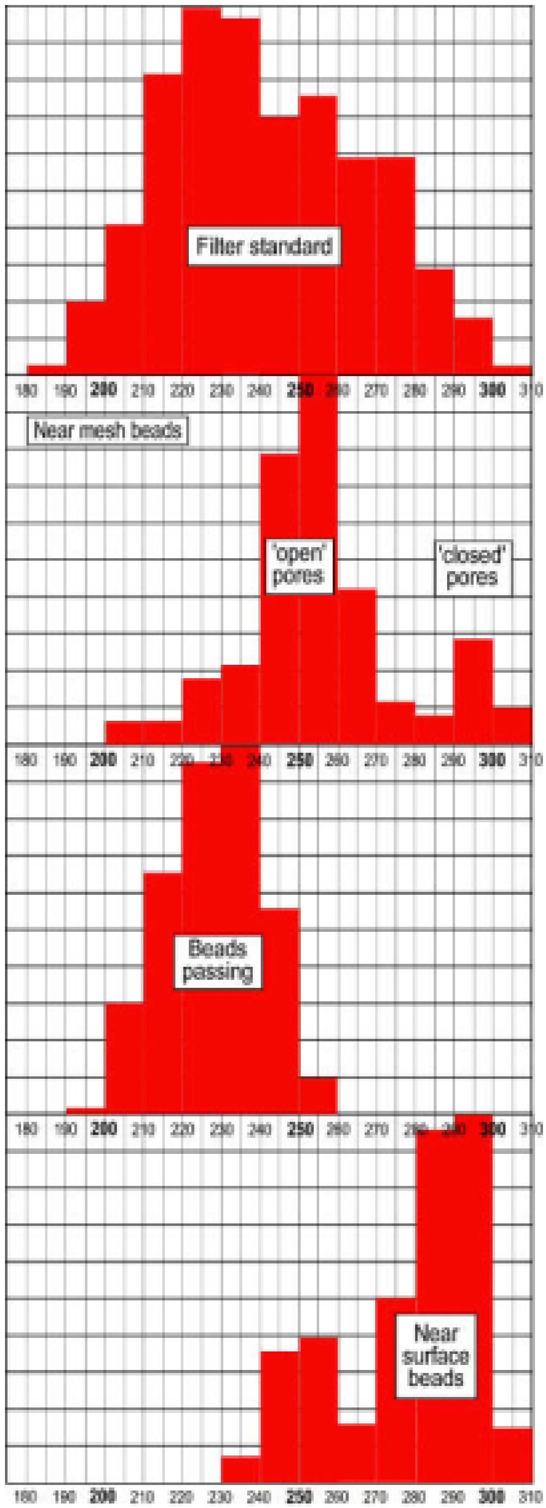

Figure 12. ‘Open’ and ‘closed’ pores in a woven filter

Conclusion

This review has majored on glass microspheres as the challenge particles in pore size measurements because the unambiguous shape of the particles give unambiguous results. However, there are other filter challenge techniques that generate in-situ particles to give high precision cut points down into the sub micron region, eg Palas, TSI etc. These methods may be the subject of a later review.

The main advantage of the challenge test described herein is that valuable information can be obtained on the fine detail of the pore structure. For example, are the pores monomodal, multimodal, open or closed? Furthermore, the technology developed has the ability to extend the measuring range up to 1000 microns without sacrificing accuracy; a region where most other technologies break down.

One of the biggest advantages of the challenge test is that the results are traceable to international units of length such a NIST, a criterion being increasingly specified by many users of filter media. The simplicity, unambiguity, traceability and speed of the method has meant that many industries operating in critical areas now insist on the filter pore size measurement by the Sonic challenge test.

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 coatings 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. The election of Dr Rideal to the position of Chairman of The Filtration Society recognizes his unique contribution to developments in the field of pore size measurement of filter media. Dr Rideal has recently joined with other prestigious members on Filtration News’ Editorial Advisory Board.