PHYSICAL MEASUREMENT OF PORES BY GLASS BEAD CHALLENGE TESTING

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Pore size measurement by the indirect method of Porometry can produce varying results, especially for larger pores in excess of about 100 microns. Consequently, 'bubble point' rated filters often fail to stop the expected particles in real situations. Historical labelling of filters, and even nominal ISO standard specifications can therefore be very misleading. In this work, narrow particle size distribution glass microspheres from a few microns to several hundred microns were used as challenge particles to assess a number of filter media. A new Sonic Filter Tester could perform the test in 1 minute in the dry state. The challenge test results were unaffected by the porosity of the samples, unlike the Porometry results were large variations were seen. In other examples, a sintered polymer air filter rated at 5 microns had a filter cut point of 140 microns while a non-woven sand screen used in petroleum extraction rated at 125 microns had a cut point of 395 microns. Recovering and analysing trapped microspheres within the pore structure can be used to measure pore size distribution and also reveal subtle internal details in woven filter media.

INTRODUCTION

Challenge testing of filter media using test dusts has been employed for many years but the results depend both on the shape of the particles and the shape of the pores through which they must travel. Using spherical challenge particles eliminates one of the variables but historically their wide particle size distribution has caused large uncertainties in the results. Single size latex beads improve the resolution, but the method is limited by the sizes of standards available, as the test is based on a 'go, no go' principle. Latex standards are also very expensive.

A new set of narrow particle size distribution filter standards based on glass microspheres has been prepared, which can resolve pore sizes with a sensitivity down to 1 micron.

In conjunction with the new standards, a highly efficient dry sonic method of fluidization has been developed, which rapidly challenges the filter medium with the microspheres. The method is effective down to about 20µm, below which particle interactive forces or poor air permeability can impede their passage through the filter medium.

Below about 20µm, an aqueous transportation system must be used to overcome the particle attractive forces. It is then possible to test filters down into the sub-micron region.

This paper begins by clarifying the definition of the micron rating of a filter, describes the range and particle size distribution of the new filter standards, illustrates the equipment used and concludes with a number of test results for filters nominally rated from 5µm to several hundred microns.

DEFINING THE MAXIMUM PORE SIZE

The important criterion in specifying a filter medium is usually it's cut point – will it remove or trap the particles in question? This is determined by the 'maximum' pore size in the filter. The reason for putting quotes around the word 'maximum' is that it is probably the most abused parameter in filtration.

'Absolute' maximum pore size is often specified without having an understanding of the uncertainty surrounding the term. The 'absolute' maximum pore size can only be found in a 100% examination of all the pores in the filter medium, which is clearly impractical for most applications. To try to estimate the absolute maximum pore size pore from a small part of a larger filter cloth will inevitably lead to uncertainties so large that the measurement is too unreliable to be of any

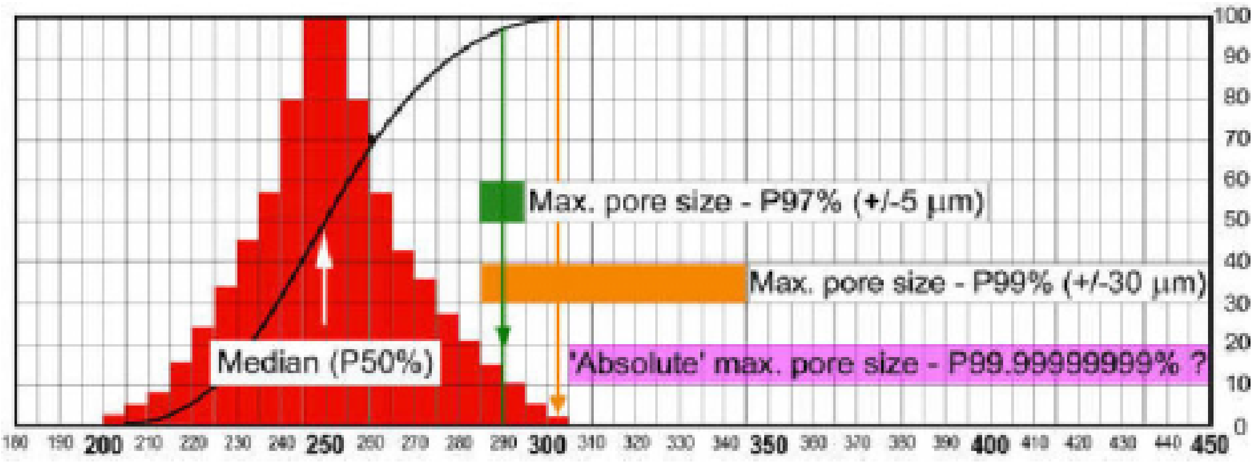

Figure 1: Uncertainties of measuring different 'maximum' pore size.

use. The reliability of the maximum pore size is therefore a function the homogeneity of the filter media as a whole and the ability to take a representative sub-sample for analysis.

Assuming that a representative sample can be taken, the confidence in the maximum pore size is dependent on the number of pores examined. There is less uncertainty in finding and measuring 1 in a 100 pores (P99%) than in finding and measuring 1 in 10 million (P99.99999999%). Measuring a maximum pore size of P97% where there are 3 in 100 or 30 in 1000 pores is far more certain and repeatable, figure 1.

Pore size defined as P97% is therefore considered the most statistically robust method of determining the maximum pore size.

MICROSPHERE FILTER STANDARDS

A selection from the filter standard range is shown in table 1. By keeping the particle size distribution narrow, a high concentration of particles close to the filter

0.2 – 2	2 – 6	6 – 8	8 – 10	10 – 14	12 – 19	16 – 25	25 – 38
36 – 59	53 – 73	63 – 86	75 – 103	106 – 147	127 – 175	180 – 248	252 – 356

Table 1: A range of narrow size distribution filter calibration standards

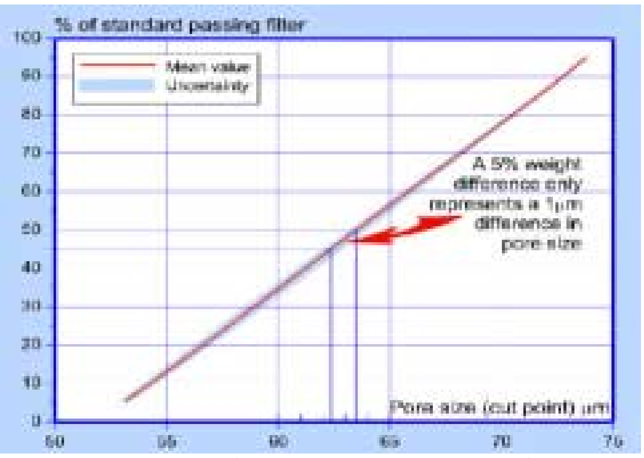

Figure 2: The precision of narrow particle size distribution filter standards.

Figure 3:Sonic filter tester and holders.

cut point can be collected resulting in a highly accurate and repeatable analysis.

Filter standards for dry testing

For optimum precision and NIST traceability, filter standards over about 15µm are certified by Electroformed sieves. A calibration graph of the percentage of the microspheres passing the square apertures is constructed. When used on an unknown filter, the percentage of the beads passing can then be used to determine the aperture size or cut point of the filter. As can be seen from figure 2, the results are very accurate because of the narrowness of the distribution.

Filter standards for wet testing

Below about 20µm, certifying the microspheres by electroformed sieving, and passing them through filter media in the dry state is much more difficult. A simple weighing process of calibration as above is therefore not possible and the particle size of the standard must be measured before and after passing the filter. Optical microscopy can be used down to about 3µm but submicron analysis must be performed by electron microscopy or centrifugal sedimentation. Once again, the narrow particle size distributions give highly accurate results.

CHALLENGE TEST APPARATUS

Dry sonic challenge testing (>20mm)

Unlike a simple 2-dimensional sieve mesh, filter media often also have a significant thickness, which restricts the passage of the calibrating microspheres. Simple shaking of a filter, as in the case of a test sieve, does not produce sufficient energy to allow the microspheres to pass the filter.

To overcome the problem, intense sonic energy can be applied to the microspheres themselves rather than the filter and holder. The fluidised microspheres are energised at 50Hz by high velocity oscillating air currents and then rapidly pass filter (typically in about 1 minute), figure 3 (Based on the Gilsonic Autosiever[1]).

Challenge testing sub-20µm filters

A simple split filter holder and small vacuum pump is all that is necessary to draw a dilute suspension of calibrating microspheres through the filter to be calibrated. figure 4.

Approximately 100mg of the filter standard in 25ml of water is used and the particle sizes compared before and after the test.

RESULTS

Definition of a filter cut point

In order to define a filter cut point, the pore size determined from the calibration graph after a sonic test was compared with the particle size of the microspheres passing the filter, in this case a 3-dimensional wire woven mesh, figure 5.

It can be seen that, from the weight passing the mesh, a cut point of 139µm was determined. From microscopy, the 97th percentile size of the beads passing was found to be 140µm.

The cut point is therefore the 97th percentile of the absolute maximum pore size. The repeatability of the method is usually better than 2%.

Comparison of Sonic cut points with Porometry

Bubble Point (B.Pt) pore sizes from different Porometer manufacturers do not always agree, as shown in table 1. It can be seen that, not only is there a

Figure 4:A simple wet filter tester.

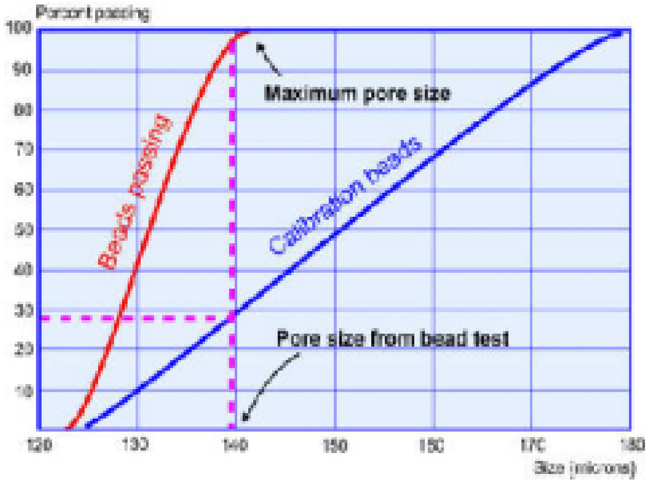

Figure 5: Defining filter pore size at the cut point.

Filter code	PX-20	PX-40	PX-50	PX-70	PX-100	PX-140	PX-170	PX-200
Cut point	21	36	48	75	95	140	175	203
Coulter B.Pt	36	56	90	180	160	230	300	300
PMI B.Pt	57	102	125	238	173	240	443	362
PMI air permeability (cfm/ft^2)	1.3	8	13	97	29	65	285	180

Table 2: Pore size comparisons for Madison DualTex™ woven filters[2]

Target cut point (µm)	Measured range, 25 tests (µm)	Uncertainty 2 x SD* (µm)	Final size (µm)
270	263 – 283	12.4	272 +/-12.4 (4.6%)
230	225 – 240	6.8	231+/-6.8 (2.9%)
150	143 – 154	6.0	147+/-6.0 (4.1%)
*SD = standard deviation			

Table 3: Cut point variation during sand screen production.

significant difference between the two instruments, but in some cases (highlighted) the Bubble Point pore size is over twice the filter cut point. When the air permeability is also measured, it can be seen that the two specimens with disproportionately high air permeabilities (PX-70 and PX-170) distort the Bubble Point measurements even further.

The Porometry uncertainties are partly due to the fact that the Bubble Point is sensitive to the single maximum pore size and so is subject to the variability in the absolute maximum pore size illustrated in figures 1 and 5. A tortuosity factor[1] can be used to convert the absolute maximum pore size to a 98% efficiency and theoretically produce data analogous to the challenge test cut point results, but unfortunately, the high measurement uncertainty is retained.

Repeatability of the Sonic cut point

Woven filter media

Because the Sonic cut point measures the 97th percentile of the maximum pore size, the repeatability is excellent. Table 3 shows that, from the analysis of 3 different grades of woven sand screens during production, the measurement uncertainty is less that 5%.

Non-woven filter media

One of the biggest problems in measuring cut points of non-woven filters, particularly at large pore sizes, is obtaining a representative sample. The meshes can be very uneven, so results vary according to the sample selected, figure 6. In order to detect significant changes in performance therefore, many tests must be undertaken. Until the Sonic cut point challenge test was available, some such filters were assigned a nominal rating of 125µm when the actual cut point was as high as 395µm, figure 7.

Pore size distributions from the challenge test

After the cut point has been measured on woven filters by the dry sonic method, the beads trapped within the pores when released and analysed reflect the pore size distribution. This method can reveal subtle internal pore structures, figure 8.

Measuring the particle sizes at 3%, 50% and 97% of the cumulative size distribution correspond to the minimum, average and maximum pore size.

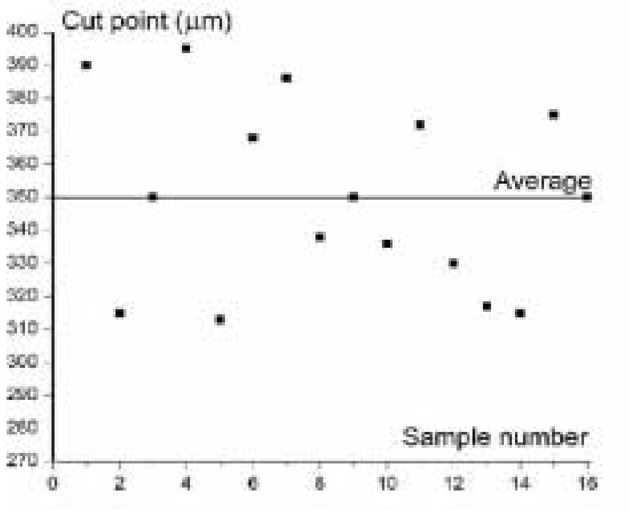

Figure 6: Nonwoven cut point variation test.

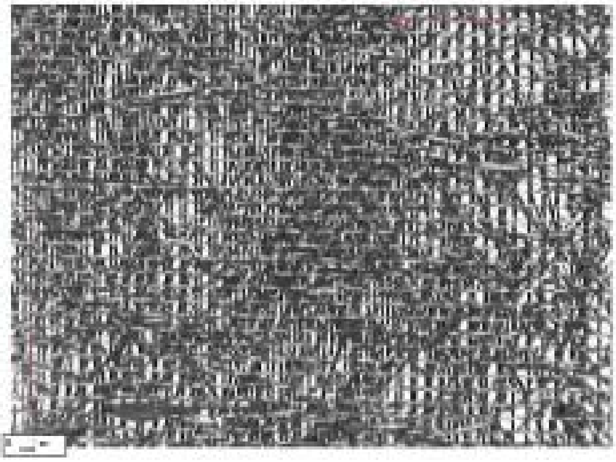

Figure 7: Inhomogeneity in a nonwoven filter.

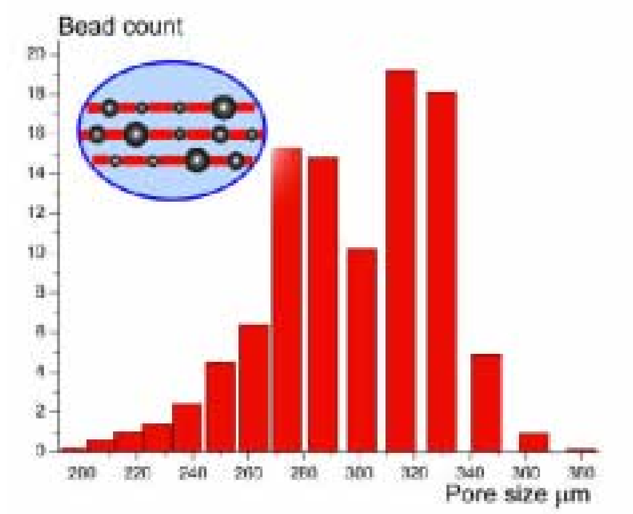

Figure 8:Pore size distribution from trapped beads after the challenge test..

Relationship between cut point and maximum pore size

A microscope analysis of the beads both passing and trapped in a woven filter has shown that the maximum pore size can be estimated from the cut point by simply adding 10%, table 4.

Measuring filter cut points below 20µm

The apparatus described in figure 4 has been used in conjunction with the narrow size distribution filter standards to measure the cut points of a wide range of filters. Dilute suspensions are used to prevent ‘cake’ build up on the surface of the filter, which would act as a secondary filter. The results for a 10µm foil ink jet filter are shown in figure 9.

Unlike the secondary method of Porometry, the glass microsphere challenge test method gives unambiguous and conclusive evidence of the performance of the filter medium.

The problems of incorrect filter specification

There have been many examples in the past where filters have been optimistically labelled. In one such example, an air filter constructed of sintered polypropylene beads was assigned a size of 5µm.

In order to test the filter, a special housing had to be constructed to hold the filter element, figure 10. By evacuating the centre of the apparatus and introducing the dilute suspension into an outer compartment, the challenging microspheres were drawn through the filter and collected in the conical reservoir below for analysis.

Assuming that the pore size was 5µm as specified,

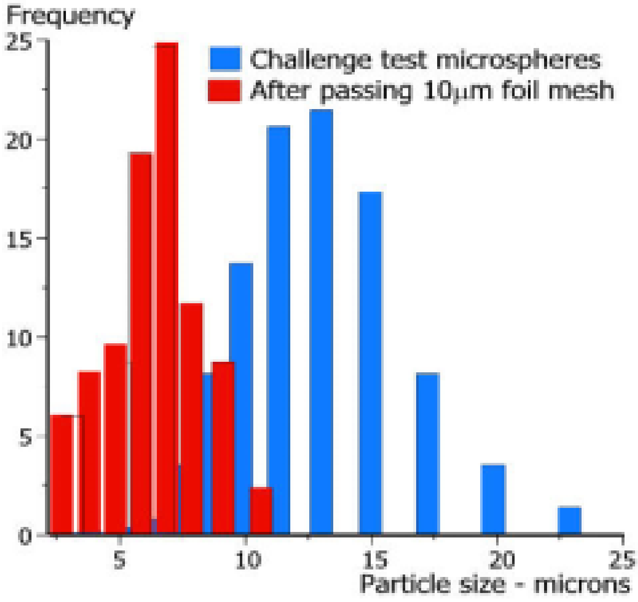

Figure 9: Performance of a filter using an aqueous based challenge test method.

Figure 10: Challenge test apparatus for an air filter.

an 8 – 10µm was initially introduced. As none of the microspheres were retained by the filter, the size of the calibrating microspheres were gradually increased until the cloudy suspension started to clarify. The eventual cut point was found to be 140µm!

Microscope analysis of beads: trapped			:passing	Sonic challenge test	
Min	Average	Max	D97%	Cut Point	Projected Max
225	292	338	308	305	336

Table 4: A projected maximum pore size calculated from the filter cut point.

CONCLUSION

The cut point is a conceptually easy to understand parameter in specifying a filter because it relates to the ability of a filter to clarify a suspension. This new unambiguous challenge test method is rapidly gaining popularity in many critical filter applications such a pharmaceutical, fine chemicals and oil extraction. This is not only because the results are highly reproducible, but they can also be traced to international standards of length such a NIST.

REFERENCES

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3. www.WhitehouseScientific.com - see ‘Library’