Particle Size and Shape Analysis – A Challenging Test

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The trouble with particle size analysis is that size is often quoted as a single number, such as, a 500 micron powder. The only particles that fulfil this criterion are those that are perfectly spherical in shape and exactly the same size. A simple analogy would be taking a ball bearing from a store bin. One could safely assume that, within the specified tolerance, all the ball bearings would be spherical in shape and of the same size.

In particle technology however, such a situation is rarely seen. Most powders are irregular in shape and have a wide distribution in sizes. This leads to a number of problems, not just in describing the size and shape distribution of a collection of particles, but in their use and downstream application.

The Challenge Test

A good case in point is the Challenge Test method of measuring the cut point of a filter [1]. Here the filter is challenged by particles having an average size close to the expected maximum pore size of the filter. The particle carrier may be air or liquid. The particle size is measured before and after passing the filter, from which the filter cut point is determined.

The first question is, out of the myriad of particle sizing techniques, which is the most appropriate for the job?

The most ubiquitous method in particle size analysis, the laser diffraction method, is not suitable in this case because, although it is highly accurate and reproducible, the resolution at the top end of the distribution is not sufficiently high enough to detect the comparatively small number of particles that pass the largest pores in the filter. Furthermore, internal modelling algorithms can often smooth out fine structural detail.

The other really important issue when particle sizing is used in Challenge Testing is how the concentration of particles at the different sizes is measured. Many techniques from sieving to laser diffraction measurement give results based on a weight or volume basis. The averaging process can give significantly different results *(Figure 1)*.

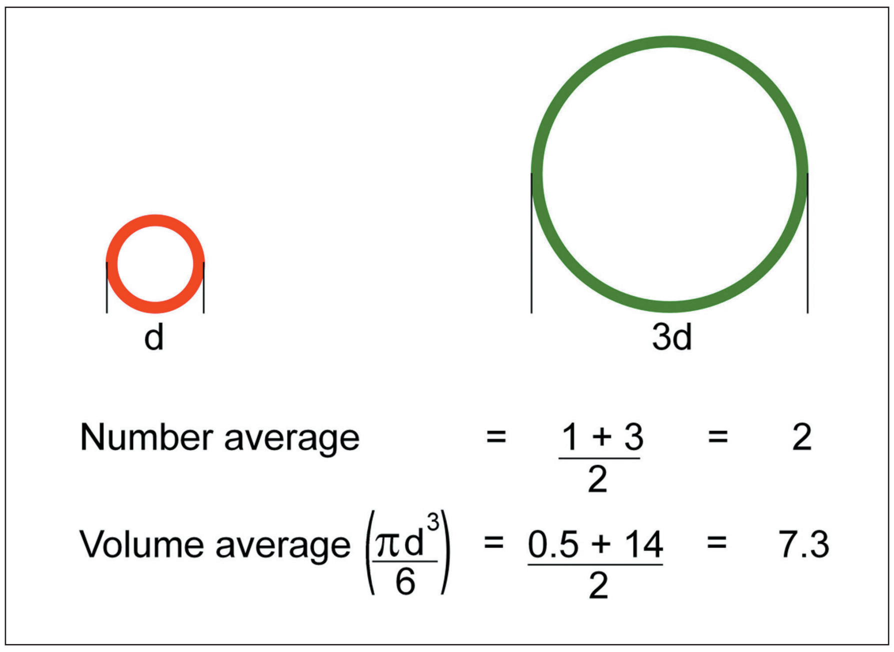

Figure 1. Averaging particle concentration by number or volume gives significantly different results

As the performance of a filter is based on the number of pores available, a number averaging of the particles is the most appropriate for the Challenge Test method. Particle counting methods include Electrical Zone Sensing, such as the Coulter [2] and Elzone [3] counters and Optical Zone Sensing, which use white light or laser light blocking techniques.

Microscopy is perhaps the most obvious, and certainly the oldest, method of particle counting and has a distinct advantage over all the other methods in that it can measure the shape of a particle as well as its size. Hitherto, microscopy has been more of a qualitative rather than a quantitative tool in that counting sufficient particles has been too labour intensive to provide sufficiently robust statistical data. However, in today's technology of ultrahigh speed cameras and computers, it is possible to count hundreds of thousands or even millions of particles very rapidly.

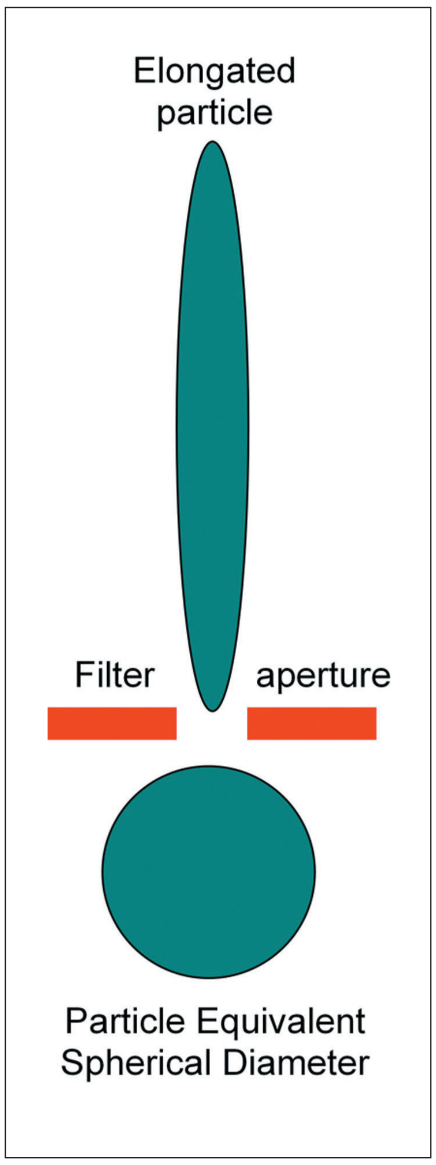

Figure 2. Using ESD rather than particle width seriously overestimates a filter aperture size

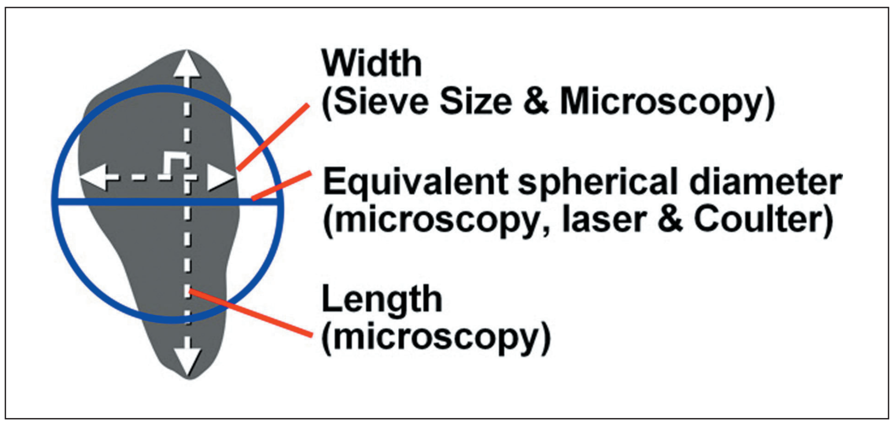

Figure 3. Particle size of irregular particles depends on the method used

The influence of particle shape

Particle shape becomes important in Challenge Testing for two reasons: Firstly, a particle will pass a filter depending on its width, not its equivalent spherical diameter *(Figure 2)* and secondly, the size of the particles passing the filter is strongly dependent on the method of measurement *(Figure 3)*.

In the Challenge Test method therefore, the particle sizing technique must be able to discriminate between particle width and equivalent spherical diameter in order to provide accurate data. Alternatively, the challenge test particles should be perfectly spherical in shape, which is not always possible to achieve.

Size Distribution and Resolution

Some of the earliest challenge test particles were Arizona Test Dusts [4]. These are still popular today and have both irregular shapes and broad particle size distributions. They therefore suffer from a number of disadvantages including ambiguous sizing, orientation dependency and, because of their broad size distribution, poor resolution *(Figure 4)*.

In order to further increase the accuracy of the challenge test, narrow distribution microspheres are to be preferred. However, very narrow particle size distributions also have their limitations:

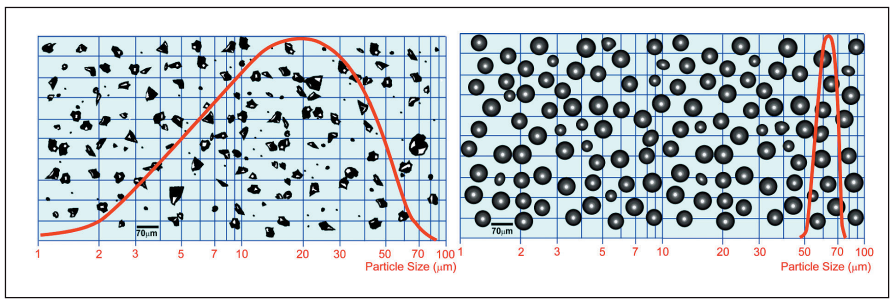

Figure 4. Narrow distribution microspheres give higher resolution results than broad distribution irregular particles

1. Several tests must be performed until the smallest size failing to pass the filter is found.
2. Single size monospheres have a limited range of sizes.
3. Filter pores are very rarely all the same size, so there will always be some 'bleeding' through the larger pores.
4. Single size monospheres are very expensive.

The optimum challenge test particles are therefore spherical in shape with a distribution somewhat broader than monodisperse, but narrower than the Arizona Test dust particles.

The only disadvantage of narrow size distribution challenge particles is that a wide range of standards must be prepared to cover all pore sizes *(Table 1)*.

When the filter standards in *Table 1* are used in the Challenge Test, it is possible to test with measurement uncertainties of less than 1 micron [6].

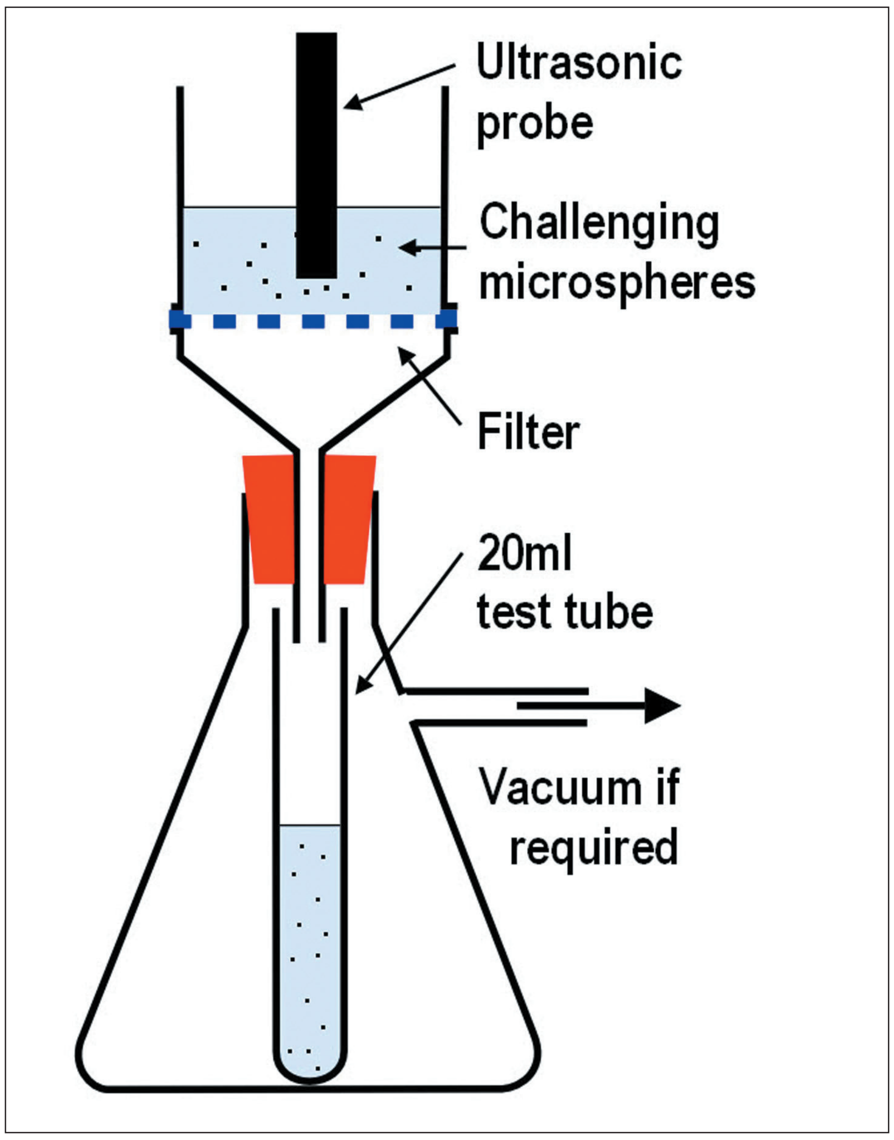

Figure 5. Challenge Test apparatus for filter calibration

Accounting for Misshapes

In the preparation of the calibration microspheres, crushed glass is spherulised in a melt process. There is therefore always the possibility of some particles not melting into spheres and others colliding and fusing together. Although most misshapes are removed during the refining process to produce the filter standards, the few that remain must be accounted for in the particle size analysis of the particles passing the filter.

The Challenge Test apparatus consists of a simple split filter holder for the filter under test, gravity or a vacuum to draw the suspension of challenge particles through the filter and an ultrasonic probe to prevent particle build-up on the surface of the filter, *(Figure 5)*.

After the Challenge Test, microscopy and image analysis is used to measure the particles passing the filter (Whitehouse ShapeSizer [5]). The raw data averaged on a number basis is shown in *Figure 6*.

It can be seen that the largest particles present have a shape factor (maximum/minimum) greater than 1, showing they are non-spherical, which would lead to an overestimation of the filter aperture size *(as illustrated in Figure 2)*.

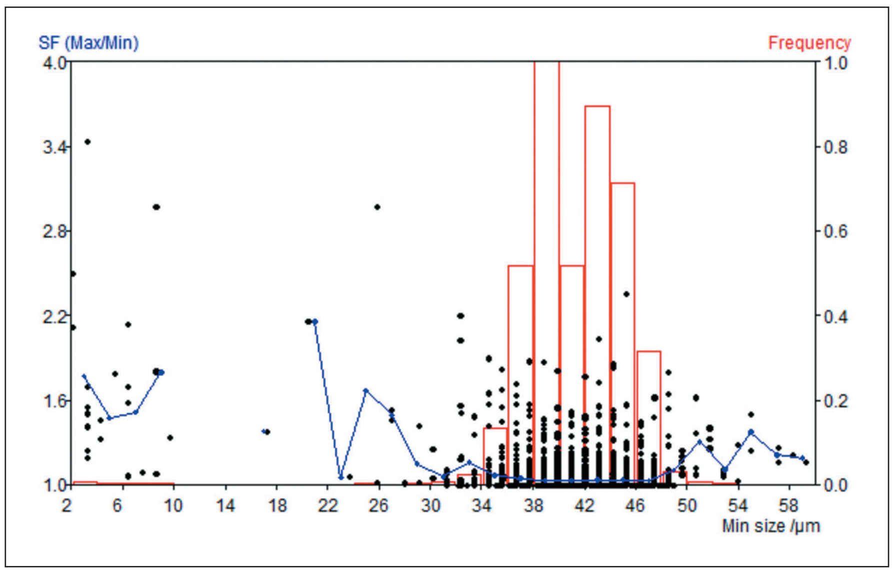

Figure 6. Raw data of particles passing a filter indicates a maximum aperture size of approximately 59 microns (Number average from the Whitehouse Image Analyser)

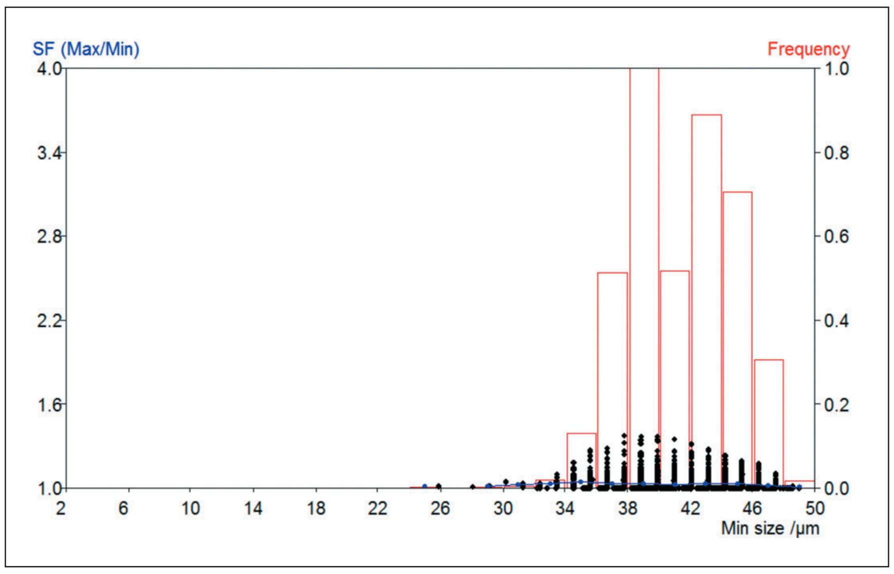

Figure 7. Removing non-spherical particles from a Challenge Test (Figure 6) reduces the maximum aperture size of a filter from 59 to 49 microns

When the non-spherical particles are electronically removed from the analysis, the maximum aperture size of the filter is reduced from 59 microns to 49 microns *(Figure 7)*.

Table 1. Range of NIST traceable glass microsphere filter standards (microns)

2 - 6	5 - 9	9 - 11	10 - 14	12 - 18
16 – 25	20 - 34	26 - 36	31 - 46	36 - 55
45 - 62	53 - 73	63 - 86	75 - 103	80 - 103
106 - 147	127 - 175	151 - 209	180 - 248	214 - 295
252 - 346	304 - 417	360 - 498	383 - 591	484 - 700

Conclusion

An understanding of the basics of particle size analysis is essential when it comes to interpreting the results, whether it be in the analysis of a product or the use of particles in a calibration application.

Large errors can occur if for example, number averaged data is compared with volume or weight averaged data. It is essential to understand the parameters produced from the various particle sizing methods. For example, sieve analysis measures the width of particles, whilst most other methods generate the equivalent spherical diameters of particles.

In the filter Challenge Test method, failure to recognise the influence of particle shape can cause significant errors in the results. Microscopy and Image Analysis is unique in being able to measure particle shape and eliminate non-spherical particles from the analysis, producing highly accurate results.

It is hardly surprising therefore, that one of the biggest growth areas in particle size instrumentation is in the Image Analysis sector.

References

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6. 'Challenge Testing Filters Using Certified Microspheres', Graham Rideal, FILTRATION, 11 (3), 2011, p 172