Precision Separation and Size Analysis – the Importance of Sieve Calibration

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Precision separation on a kilogram scale is very important in many industries, particularly the pharmaceutical industry where 10 – 20 kg of a product can cost thousands of dollars. 450mm diameter sieves are a very effective way of size separation on such a scale because they give very sharp cuts at the specified sieve size. Unless the sieves are accurately calibrated, however, a repeatable cut point cannot be guaranteed. Even though the sieves used may be nominally the same size, some large differences in absolute mesh size have been observed in the past.

One advantage of 450mm diameter sieves is that once they have been calibrated, they can be used both as an accurate method of particle size analysis with traceability to NIST, and as a precision separation method. For the purposes of this report the performance of large diameter sieves was investigated using 700 - 1400 micron glass beads [1] on a new 450mm diameter Powermatic sieve shaker from Endecotts [2] (Figure 1).

Figure 1: The Powermatic 450mm diameter sieve shaker

A factor that is often underestimated in particle size analysis is that of obtaining a representative sub-sample from a larger weight of material. For best results, a spinning riffler is recommended, although a riffle chute, riffle box or the even more statistically unreliable method of coning and quartering can be used [3]. As obtaining a representative sample adds an extra step to the analytical process it is unfortunately sometimes overlooked, resulting in very poor reproducibility of results.

Where sample weights from 200 to 2000g are used for analysis with large diameter sieves, the subdivision process may not be required, as the entire laboratory sample could be put on the sieve stack. The question of subdivision would not then arise. Furthermore the same machine could also be used for large-scale preparative work.

Almost any diameter sieve may be clamped on to the vibrating table of the Powermatic, but for the 450mm diameter sieves a maximum of five may be loaded at any one time. There is also the possibility of continuous sieving using a special adapter, but this attachment was not evaluated here.

The vibrating energy is provided by two motor-driven eccentric shaker units in the base of the sieve shaker and is controlled by an electronic timer which has pre-set times from 5 to 60 minutes. Particle size analysis of the glass beads in this study required the following sieves:

710μm, 850μm, 1.0mm, 1.18mm and 1.4mm

Sieve calibration

Whitehouse sieve calibration standards were used to calibrate the 450mm diameter sieves [4]. These standards are in the form of accurately measured glass beads whose diameter peaks at the nominal sieve aperture. The width of the distribution is one sieve size either side of the sieve in question, based on the ISO

3310/1, R40/3 supplementary sieve sizes, so for the 850µm standard, the size range is 750µm to 1mm.

To calibrate a 450mm diameter sieve, the complete contents of the reference standard bottle were sprinkled evenly over the surface of the sieve and were shaken by hand for 1 minute (the weights of the reference standards varied from 25g to 50g depending on the sieve being calibrated). Care was taken during the shaking procedure to ensure that the swirling action employed produced an even coverage of the sieve surface by the calibrating beads (Figure 2).

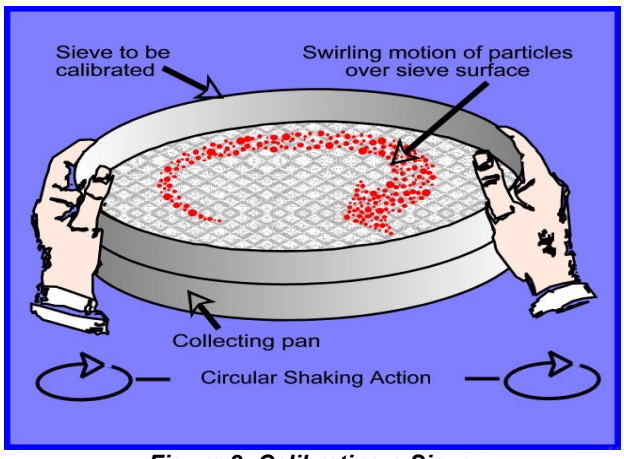

Figure 2: Calibrating a Sieve

After the shaking period was complete, the sieve frame was tapped a few times to dislodge near mesh beads and the weight of material passing through each sieve was measured to an accuracy of 0.01g. Knowing the percentage passing, a calibration graph supplied with the Test Certificate was used to find the mean sieve aperture size. This was then traceable to the International Standard of length, for example, NIST.

The results of the sieve calibration are shown in Table 1.

Table 1. 450mm diameter sieve calibration using microsphere standards

Nominal sieve size µm	710	850	1000	1180	1400
Measured sieve size µm	705	857	986	1163	1377

Sample subdivision

In any particle size analysis investigation, it is essential that every sub-sample presented for analysis is representative of the bulk sample. This is especially important for glass beads in the millimetre size range, where particle mobility can easily cause segregation during transit or handling.

Whitehouse Scientific has designed and built a unique range of spinning rifflers, which have been independently evaluated by Loughborough University in the UK for use by the European Commission BCR programme in the production of particle size reference standards [5]. These rifflers were used to prepare the sub-samples for analysis (Figure 3).

Figure 3: Sample Sub-division by Spinning Riffler

The complete 25kg bag of glass Ballotini beads [1] was put on a 100 stage spinning riffler to produce 100 x 250g sub-samples. Individual samples, or multiples thereof, were then used for testing on the 450mm diameter sieve shaker. Any variation in the results could be attributed

to the experimental conditions and not to errors in sampling.

The 250g sub-samples were further subdivided in a small-scale riffler to produce 7.5g samples for accurate size analysis using electroformed sieves.

As accurate as Electroformed sieve analysis

As particle size analysis by sieving on wire woven sieves has often been criticised as being inaccurate, the next series of experiments was designed to investigate the performance of 450mm diameter sieve analysis in comparison with the most accurate sieving method - Electroformed sieve analysis [6]. 75mm diameter electroformed sieves from Gilson (USA) [7], which were traceable to NIST, were used to produce a benchmark for the analysis.

By shaking the calibrated 450mm diameter sieves for 5 minutes on the Powermatic, five repeat analyses were performed on the 250g sub-samples. The variation in the results was less than 1% and could not be separated by plotting on a cumulative graph. Similarly, and just as important, the results were virtually identical to those produced using the highly accurate electroformed sieve method. Differences in the two methods could be seen only when the data were plotted as a deviation against the mean (Figure 4).

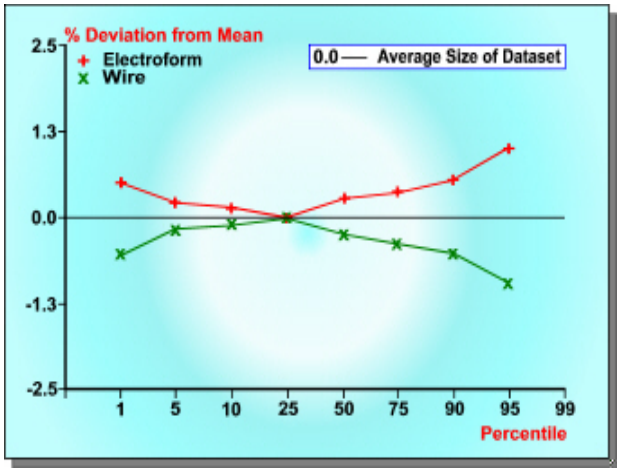

Figure 4: Calibrated Wire Sieves give Similar Results to Precision Electroformed Sieves

Precision separation

The results so far have shown how accurate a 450mm diameter sieve shaker can be for size analysis, provided that the woven wire sieves are first calibrated using reference standards.

In the final series of experiments, the load on the sieve stack was gradually increased from the initial 250g up to 2kg in order to determine the maximum capacity when the machine was used as a batch separator. The shaking time was 5 minutes.

Again the results were extremely close (Figure 5), and within the usual band spread of 1%. Even at the maximum loading there was no evidence of sieve blinding on any of the sieves. The only reason the experiment was stopped was because the loading on one of the sieves had reached 1kg and there was a danger of distorting the sieve mesh.

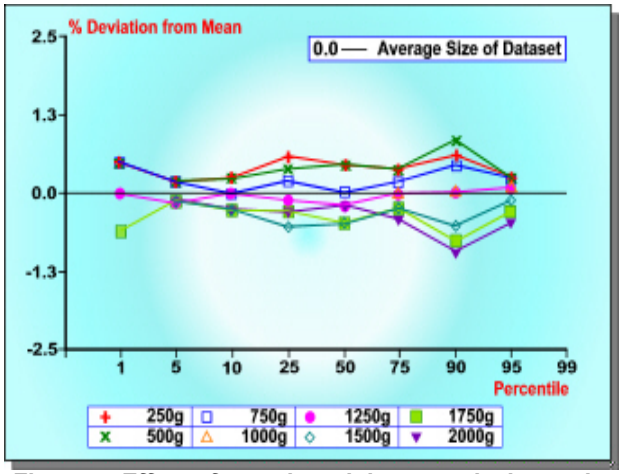

Figure 5: Effect of sample weight on analysis results

The efficiency of the separation process in a batch operation has led to the development of a continuous sifting system for the Powermatic, although this system has not been evaluated in this report.

Conclusion

Particle size analysis using large diameter sieves (450mm) has had the reputation in the past of being irreproducible and inaccurate in comparison with modern methods. Now that sieve calibration standards are available to measure mean

sieve apertures, it is at last possible to accurately quantify sieving performance.

This research using a new 450mm sieve shaker showed that, provided the sieves are calibrated, and representative samples are presented for analysis, the results are indistinguishable from the high precision Electroformed sieve method. Furthermore, the performance is maintained for sample loadings up to 2kg.

An advantage of such a high flexibility with regard to charge is that in many cases there may be no need for a riffling process to reduce a bulk sample down to the gram quantities required for most other particle sizing techniques.

The advantage of using calibrated sieves for particle size analysis also applies to the Powermatic when used as a separator. Because the 450mm sieves can be quickly and accurately calibrated, they can be individually selected before use to ensure there has been no deterioration or that, when replaced, the new sieve can be perfectly matched. Thus quality assurance can be guaranteed.

Finally, because batch weights of several kilograms can be processed on the 450mm diameter sieve shaker, it has the advantage of being able to be used for preparative work of expensive products such as pharmaceuticals and speciality chemicals.

Bibliography/Literature

1. Potters Ballotini, Barnsley, UK and Valley Forge. Pennsylvania, USA
2. Endecotts Ltd, London, UK
3. T Allen and A A Khan, Critical Evaluation of Powder Sampling Procedures, The Chemical Engineer, May 1970
4. G R Rideal, J Storey and T R Morris, Sieve Calibration – A New Simple but High Precision Approach, Part. Part. Syst. Charact. 17 (2000) 1 – 7
5. S Dutton and P J Lloyd, Analysis of Riffler Performance, Loughborough Consultants, July 1994
6. G R Rideal, Absolute Precision in Particle Size Analysis, American Laboratory, Nov.1996
7. Gilson Co. Inc, Lewis Center, Ohio, USA