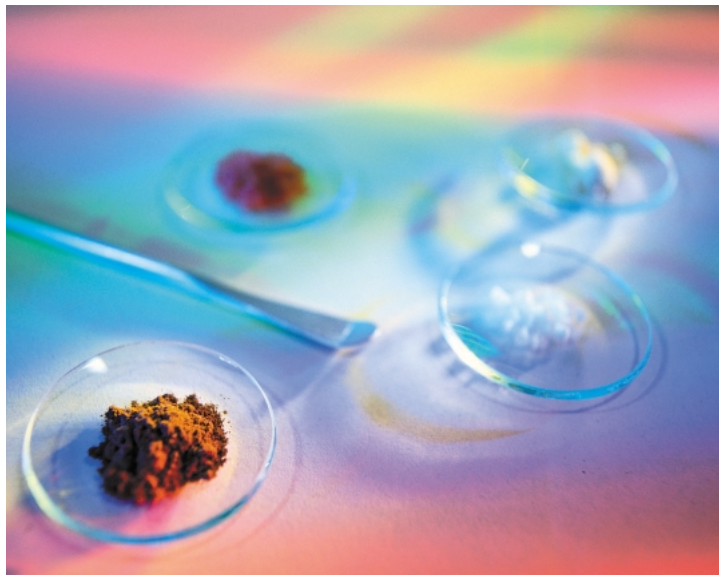


Successful Particle Size Analysis of Dry Powders

A correctly set up dry powder measurement using a laser diffraction particle size analyser is much easier to perform than measurement of wet suspensions. However, incorrect measurement set-up can lead to errors and inconsistencies. This article examines the key steps achieving successful dry to powder measurement. Issues examined include powder flowability and sampling, air pressure and sample dispersion, feed rate and the importance of Standard Operating Procedures.



Rod Jones

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A significant difference between wet and dry measurements concerns sample conditioning. With a wet suspension, there is ample opportunity to condition the sample. Particle agglomeration is clearly signalled by the software, allowing the addition of appropriate additives or stabilisers, and the use of ultrasonics. Repeat measurements can be performed until a "satisfactory" result is obtained, with further measurements confirming the stability of the suspension.

In contrast, there is no opportunity to recycle dry powders to continue treatment, as the sample passes through the laser beam just once. It is therefore important to ensure that the dispersion process is

correct, so that the size analysis accurately reflects the particle size distribution.

Sample flowability and sampling

A sample's flowability has a major impact on how it should be dispersed. Free-flowing materials are prone to segregation when subjected to vibration, either in bulk transit or during transport by mechanisms such as vibrating hoppers.

The implications are twofold. Bulk material should be thoroughly mixed to counter any segregation before samples are withdrawn, and samples should be split by a reliable technique, such as spin riffling. The importance of this cannot be overstated: the standard deviation (SD) between riffle split poly-disperse samples can be as low as 0.146%, compared with 5.14% for scoop sampling [1]. If no riffler is available, bulk samples should be mixed by half filling a screw-top container and tumbling it end-over-end at least 20 times before withdrawing a sample. The design of the spoon or spatula used to take sample from the pot is also important. Coarse particles

fall off the edges of flat spatulas and this can give rise to a significant bias. A suitable small spoon is preferable.

The second important consideration is to ensure that the measurement consumes the entire sample: if significant segregation takes place within the sample during transport in a feeder, then a bias may result. If there is any doubt about the occurrence of segregation, this can be verified by a simple series of repeat, short duration measurements from a single tray full of material. Where there is no difference between the first and final measurements in a series, segregation is clearly not a problem. If there is a significant difference, the whole series can be integrated and averaged to obtain an unbiased result for the sample. Subsequent measurements of such material should

be of sufficient duration to ensure that the whole sample has been seen. This provides automatic integration, and eliminates any bias. Observation of significant differences between short duration measurements gives valuable information about the behaviour of a material and can make a significant contribution to understanding its bulk flow properties

Air pressure and dispersion

Cohesive materials are less prone to segregation and sampling errors, and usually require more dispersive energy to produce a cloud of primary particles. This brings its own challenges. The smaller the particles, the greater the surface attraction (Van der Waals forces). It has been shown [2] that inter-particle attraction due to Van

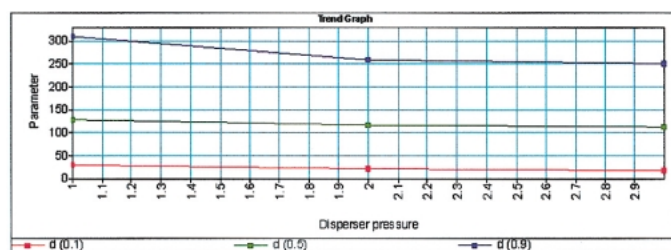


Fig. 1: Pressure titration: as the dispersive pressure increases, the d10, d50 and d90 plateau, indicating that 3 bar is the optimum pressure for correct dispersion

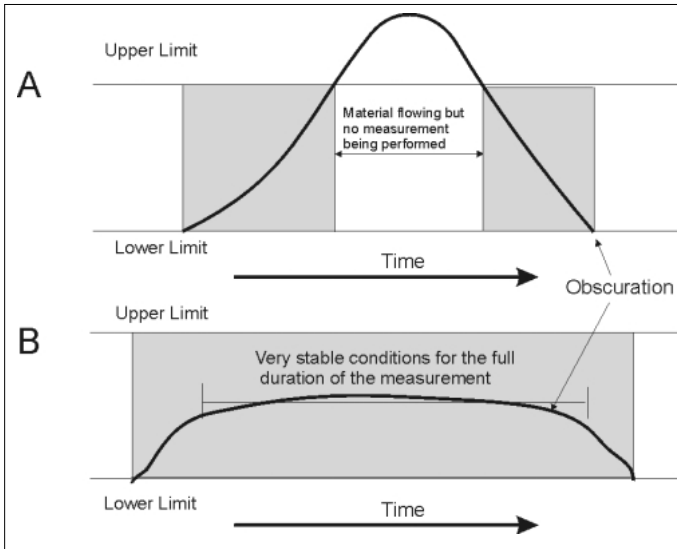


Fig. 2: Setting the feed rate so that the obscuration lies within the limits for the duration of the measurement (using obscuration triggering) will ensure consistency of results. However, too much reliance on obscuration triggering in preference to regulating the feed rate will give less reliable results overall.

der Waals forces is around 100g (g being force of gravity). This increases to 1000g for 1 micron particles and to between 10^4 and 10^5 g for particles of 0.1 μm .

A dry powder feeder is similar in design to an air jet

mill. Tapering the particle transport tube within the feeder accelerates airborne material close to the speed of sound, creating shear forces that separate loose agglomerates. Particle collisions with the walls of the feeder further

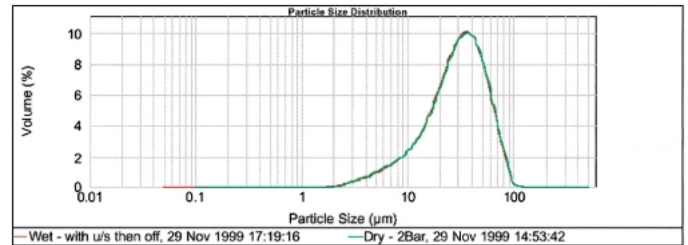


Fig. 3: Overplot of wet dispersion and dry measurement on a dry powder coating - perfect agreement at a dispersive pressure of 2 bar

disperse the more cohesive agglomerates.

For delicate materials, dispersing at too high a pressure will result in overly severe collisions, causing fracture of the material and milling of the product. Conversely, incomplete dispersion may occur at too low a pressure, producing a larger than expected size. To find the optimum between these two effects, the performance of a "pressure titration" (recommended in Section 6.2.3.2 of ISO13320) is advised. This is a series of repeat measurements at different dispersive pressures to determine the most suitable. Creation of a

trend graph of $D(v,0.1)$, $D(v,0.5)$ and $D(v,0.9)$ versus pressure helps to visualise the results. Fig. 1 shows a typical result, where a size plateau is reached with a pressure of 3 bar and is maintained at higher pressures, indicating that 3 bar is sufficient for successful dispersion. Sometimes with very friable material, a plateau is never reached - increasing the pressure simply mills the material to finer sizes. For low density, freeze dried materials, good results will usually be obtained at dispersive pressures as low as 0.25 bar.

Feed rate

Feed rate is important in determining measurement success. If particle transport is the only consideration, then free-flowing powders require a lower feed rate than cohesive powders, which need more energy to transport them into the venturi of the feeder. The ideal obscuration range (Malvern Mastersizer 2000) is between 0.5 to 5% for a dry measurement, differing markedly from the ideal for wet measurements (between 5% and 20%). The finer the material, the lower the ideal obscuration can be since the determining factor in measurement success is the number of light scattering nuclei passing through the laser beam during measurement. If particles are large, there will be fewer nuclei per gram, so sample loading can be increased. If micronised materials are being measured, flow rates should be carefully regulated to achieve obscurations closer to 0.5%. For very cohesive materials, such as magnesium stearate, placing the sample nearer to the front of the sample feed tray will remove the need to increase feed rates to transport the sample to the venturi. Aiming for lower obscurations when cohesive dry powders are being measured has the added benefit of maximising the dispersive effect of the dry powder feeder venturi, since the impact of the

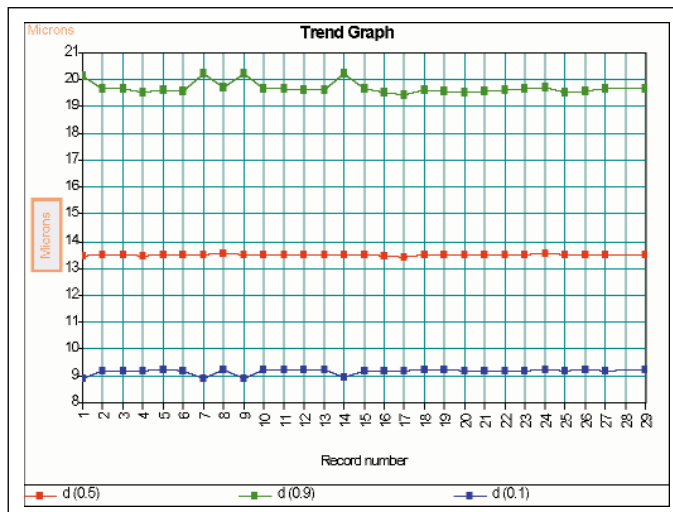


Fig. 4: Three of the measurements were performed by particle sizing experts – the rest were performed by people with no prior experience of particle size analysis

particles on the walls is better controlled at lower feed rates.

On the Mastersizer 2000, Standard Operating Procedures (SOPs) in the software can be set up to govern the ideal feed rate and air pressure automatically. Sample is measured only within an ideal obscuration range, stabilising measurements considerably. Further improvements in measurement quality are achieved by adjusting the feed rate to ensure that the obscuration remains within the limits for the duration of the measurement (as shown in Fig. 2A and 2B).

Comparison with wet measurements – the ultimate measure of success

When an SOP is set up for the anticipated lifetime of a prod-

uct run, and is expected to be replicated globally, determining the ideal settings by running a pressure titration is a good investment of time. Confirmation of the correctness of these settings is possible in many cases by comparing the dry results with a wet measurement, as recommended in Section 6.2.3 of ISO 13320. When in close agreement, these provide a useful defence of the method if it must be audited for regulatory purposes. A perfect example of the confirmation of the correctness of a dry powder measurement is shown in Fig. 3.

Conclusion

Achieving optimum results for dry powder measurements requires some simple precautions. Once the measuring system is set up for the optimum flow of the powder, and a representative sample can be provided to the instrument, the feed rate should be adjusted to give an acceptable obscuration. After this, a pressure titration can be used to determine the most appropriate pressure. Wherever possible, this should be confirmed by comparison with a wet measurement. The duration of the dry measurement should be adjusted to ensure that the whole of the sample is consumed.

If these steps are taken, the result will be an SOP that al-

lows people with little or no experience of particle sizing to perform good measurements every time. Fig. 4 shows a trend plot of 29 measurements of cornflour performed by 3 experts and 26 novices. The novices were shown where to place the samples and were told to follow the instructions on the computer screen. There is no significant variation in the results obtained, so regardless of the fact that 3 users had extensive experience of performing particle size measurements, the results they and the novices produced are all in perfect agreement.

Literature

- [1] T. Allen "Particle Size measurement" 5th Edition, Volume 1. Chapman and Hall 1997. ISBN 0412 729504. Table 1.5, Page 38
- [2] Aerosol Science, Ed. C.N Davies, Academic Press, London and New York, 1966

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