



The importance of sampling and dispersion for dry powder applications

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When choosing a house, the most important attribute is Location, Location Location. In particle size analysis, Sampling, Sampling, Sampling is key starting with the process itself. If you do not obtain a representative sample from the process pipe, whether it be from a transition point or the exit from a classifier or cyclone, then any subsequent analysis performed in the laboratory on any particle size analysis instrument is futile. If all processes transported product in an homogenous way to a sampling zone then sampling would be easy. The reality in most processes is at odds with this ideal situation.

There are two basic rules which must be adhered to when sampling from a process. The first is that sampling must be taken from a process location where all the particles are in motion, rather than from a static position, like a silo or hopper. The second condition that must be met is that a full cross section of the process stream or a truly representative aliquot of it must be sampled for a short time rather than a smaller proportion of it for a longer time.

There are situations where the need for perfect sampling is not absolutely necessary. Generally you can say that materials with a small mean size covering a tight distribution can be more forgiving. The broader the distribution, especially with a long, coarse tail and the greater the mean size, the more precise you need to be with respect to sampling. Sampling location in these situations is key, taking care to avoid sampling from a process stream where roping is occurring or where stratification is known to occur.

Once you have adopted an appropriate sampling method and location, the product needs to be transported to the laboratory and once there, redispersed by tumbling (not shaking) the sample 4 or 5 times. The sample container must be less than 60% full for this redispersal to be effective.

Over the past 30 years, dry particle size analysis has become more prevalent especially in the pharmaceutical industry where there is more need to disperse the product with exotic solvents which will disperse but not dissolve the formulation. In addition there are always potential problems with choosing the appropriate surfactant and admixture, type, intensity and duration of ultrasonic treatment. With all these factors to take into account, it is not surprising that so many SOPs used either produce an erroneous result or differing results from different operators using the same standard operating procedure.

Why was dry particle size analysis not adopted earlier by this industry compared to minerals, cement and powdered coatings, all of which were originally measured wet but are now exclusively measuring their products dry? To understand this, we have to look into how dry particle size analysis used to be performed and in some cases how some particle size analysis equipment providers are still making dry particle size analysis feeders/dispersers with inherent flaws in their design.

In the early days, the sample was typically transported to the dispersing zone (an area where a jet of air impacted with the sample and hopefully dispersed it all well enough to deliver to the laser beam for measurement). One of the primary flaws was that sample was delivered to the dispersion zone via a vibratory chute. There are 2 issues with this method. The first is that material, particularly cohesive powders, would vibrate down the chute in varying concentrations so you might either have too much material delivered to the disperser or not enough. If too much arrives then the air disperser is not efficient enough to disperse all of the sample, all of the time, resulting in a fine, dispersed distribution and a coarse, undispersed population. In addition, after taking care in providing a representative sample from the process, not all of the material was measured, with material being left on the vibratory tray and dry sample flow path.

To solve these problems, hardware changes were made to provide a more consistent flow of sample to the dispersing zone. Rotating brushes were used to predisperse the powder but this technique resulted in cross contamination of samples from run to run and from a maintenance perspective, had shorter mean time before failure periods. This was followed by using wire meshes with ballbearings to predisperse the sample, but yet again, not all the sample was measured with cohesive and static samples building up on the chute, balls and sieve mesh rendering the result unrepresentative.

In addition to these dispersing issues, several dry dispersion units would disperse the sample and shoot the material out of a nozzle into the ambient air to be collected by a vacuum. There are 2 drawbacks with this technique, the first being that for pharmaceutical active ingredients, there is a serious health and safety issue. Secondly from an analysis point of view, the smaller particles would travel across the laser measurement zone at a much slower speed than the larger particles, as they lost their momentum in the ambient air. This ultimately produced heavily biased results towards the fines and the higher the dispersal air pressure was set, still finer results could be produced. Dialling a result does not produce much confidence in the result and in many cases leads to the user believing he has a much better product than reality.

In the eighties a piece of hardware was offered, which would drop the sample from a chute onto a rotating v notch sampling zone. From a feed control perspective this was good but only a small proportion of the material actually stayed on the v-notch, the rest falling over the side into a bin and not analysed at all. In the case of broad or coarse distributions, this would produce a size result which was biased towards the fine side with much of the larger particles being missed. In addition, this device had a scraper which skimmed off all the larger material at the top of the v-notch weir thus biasing the result still further towards a finer measurement than reality. Immediately following the scraper was a roller to tamp (crush) the material down into the v-notch, which in the case of crystalline products such as many pharmaceuticals or materials whose particles had a high aspect ratio, could result in them being milled, biasing the result still further towards the fines.

So what is the answer?

Well, what have we learned over the last 35 years?

1. The ultimate dry powder feeder, capable of dispersing all materials typically produced in industry, especially, cohesive and static samples, must not use vibratory feeders as they attract sample to their surface and can send the sample to the dispersion zone in a feast and famine scenario.
2. There must be nothing in the sample flow path such as sieve meshes or ball bearings.
3. As close to 100% as possible of the material added to the sampling tray or holder, must be analysed to provide the user with a result he can feel confident in.
4. The sample must be dispersed and transported to the particle measurement zone, in a closed flow path not open to the environment with all particles, large and small travelling at the same speed.
5. An adjustable air pressure or air flow rate must be incorporated into the feeder such that epoxy powder coatings which need next to no dispersion can be transported at low pressure or flowrate and higher pressures can be used for the more cohesive powders.
6. An appropriate eductor must be used which disperses but does not mill the sample.
7. A post dispersion device is incorporated into the feeder, dispersing system, which allows fine-tuning (if necessary) of the sample dispersion.

All the positive attributes and none of the negative outlined above are incorporated into the Microtrac TurboTrac dry powder feeder. One of the acid tests for a dry powder feeder is the highly fatty (upto15%) turkish coffee. The TurboTrac can disperse and transport this and other supposedly difficult samples in its stride.

The key feature is that rather than putting energy into sticky or cohesive materials by vibration in order to deliver them to a catchment zone, the sampling probe moves towards the sample in a slow consistent manner, snorting up the material as it moves along the holding tray ultimately collecting in most cases at least 99% of the sample. This device has been proving its worth, satisfying users for the past 10 years and is currently the de facto standard for serious dry particle size analysis adopters in most industries.