Practical powder blending: Sampling

Bulk solids manufacturers often need to sample a powder blend to ensure that the material meets product specifications. In previous “Mixing Mechanics” columns I’ve discussed common blending failure modes, including improper run time, segregation, and ingredient agglomeration. I’ve also mentioned sampling as another potential blending failure mode, which may seem odd, since the reason you sample is to ensure your blend’s adequacy. When improperly done, however, sampling can introduce or give the appearance of the very conditions the sampling is meant to identify or fail to identify those conditions when they actually exist.

In this column, I’ll discuss how to ensure that your blend-sampling regime is effective and appropriate for your application. I’ll discuss physical sampling and inline sensing in parallel because many of the issues are the same for each and because this will allow me to easily compare the two methods.

Physical sampling involves manually collecting a material sample from a stationary powder bed or moving powder stream and analyzing the sample in a lab. Inline sensing evaluates the blend quality midstream and in real time, typically using near-infrared spectroscopy or fiber-optic sensors. When sampling a powder blend using either method, it’s important to ensure that you’ve chosen the appropriate scale of scrutiny (or sample size), that the samples collected are representative of the blend as a whole, that the samples collected are accurate, and that the sampling results are statistically significant.

Determine the appropriate sample size

The crucial starting point when sampling is to determine the appropriate sample size to be extracted from the blend (or the size of the region to be analyzed by a sensor). The appropriate sample size depends on the reason you’re sampling the material in the first place. Where multiple samples are extracted and analyzed to determine the blend’s homogeneity, for example, as the sample size becomes smaller, the variability between samples increases. Very small samples composed of only a few particles can have a very large degree of variation between them, leading to analytical inaccuracies. On the other hand, large samples may have less variation between them, but they increase the amount of material collected and the effort required to analyze it.

For most processes, the appropriate sample size is determined by how the powder is processed downstream from the blender, how the powder is subdivided into product units, and how the powder’s properties affect the function of those product units.

When sampling for blend homogeneity, the sample size should be equal to the size of the packaged final product. In pharmaceutical manufacturing, for example, the powder ends up compressed into a tablet or capsule. If you’re trying to determine that each tablet or capsule has the same chemical composition, the appropriate sample size is equal to the amount of powder used to fill a single capsule or make a single tablet. Using a larger sample size could mask product variability and potentially be detrimental to a patient taking the product.

If the final product is a spray-dried tea sold in 10-kilogram bags to be used all at once, the sample size should again be equal to the final product’s size. A smaller sample size might indicate variability that isn’t relevant to the product’s functionality. In other words, if the 10-kilogram bag will be used all at once, it doesn’t matter if the blend is consistent within the bag, only that the bag is consistent with all the other 10-kilogram bags.

If you’re not sampling for blend homogeneity, then a different scale of scrutiny might be appropriate. If you’re interested in the blend’s microstructure, for example, the appropriate sample size will typically be slightly larger than the particle size; but if you’re trying to diagnose a blend segregation problem, a sample larger than the product unit can help isolate the segregation from other causes of variability.

Sample size is easy to visualize for physical sampling but less so for inline sensing. Typically, the sensors used for this purpose only test a very small amount of powder at a time, leading to the detection of a large amount of variability that’s not always relevant.

If you’re taking multiple measurements and averaging the results, the situation becomes more complicated. If the powder is in continuous motion relative to the sensor, averaging multiple measurements increases the amount of powder analyzed but doesn’t necessarily increase the sample size as a percentage of the blend. Ensuring that your sample size is large enough depends as much on how frequently you sample as it does on the number of samples you take.

Ensure that the samples are representative

For a sample to be representative of the blend as a whole, the sampling process must ensure that all portions of the blend are sampled with the same probability. For a truly repre-
sentative physical sample, you would need to either select the sample locations randomly or sample every location in the blender; neither of which is typically practical. As an alternative, you might try to develop a sampling plan that tests places in the process that might be problematic. While this is a well-established practice, it can lead to frustration because the sampling locations you choose might not actually be the most critical.

When using inline sensing, the easiest way to ensure a representative sample is to test the powder as it’s being discharged from the blender. To the extent possible, you should locate the sensor (or sensors) to capture the entire material stream. This will generally yield better results than the established practice of using a single sensor inside the blender to monitor the evolution of the blend’s variability. The single sensor location you choose often won’t be representative of the blend as a whole.

Ensure that the samples are accurate

An unfortunate fact of physical sampling is that the tool most commonly used, the sample thief, often introduces significant error to the sampling results. A sample thief is a metal cylinder with one or more recessed cavities that can be opened and closed by twisting a handle. To take a material sample with a sample thief, an operator inserts the thief into a stationary material bed and opens the cavities, allowing material to flow in. The operator then closes the cavities and removes the thief with the sample material inside.

The sample thief can introduce bias and variability to the sampling results in several ways, including:

- Certain blend ingredients can stick preferentially to the sample thief.
- The blend can segregate along the sample thief’s insertion path.
- Certain blend ingredients can flow preferentially into the sample thief.

Inline sensing methods can also lead to sampling inaccuracy. For example:
- Material can stick to the sensors, leading to measurement errors.
- Sensors using near-infrared spectroscopy may not be sensitive enough to accurately analyze the material.
- Incorrect sensor placement can lead to sample bias.

The best way to determine whether your sampling method is accurate is to compare the sampling results to the final product. If the sampling method is accurate, the final product composition and variability should be the same as your sampling results.

Ensure that the sampling results are statistically significant

Once you’ve chosen the correct sample size and determined that the sampling results are representative and accurate, you still need to determine how many samples or measurements will enable you to confidently reach a conclusion about your blend’s composition. Again, the answer depends on why you’re sampling. It’s common to collect and analyze as few as 10 samples and rarely practical to collect more than 30. If you’re evaluating the blend’s average composition, and neither segregation nor agglomeration are significant concerns, then 20 to 30 samples is often sufficient because the samples will typically have a Gaussian (or normal) distribution and reliably estimate the overall blend composition.

However, if your purpose is to estimate the standard deviation of the sampling results (to determine the blend’s degree of homogeneity), then 20 to 30 samples will be insufficient, even for accurate samples with a normal distribution of compositions. Unfortunately, reliably estimating a population’s standard deviation often requires a larger number of samples than is practical.

If you’re sampling to diagnose inefficient blending, segregation, or agglomeration, the situation can be considerably more difficult because these conditions will typically yield samples that won’t have normal distributions, and accurate material characterization of the sampling results may require hundreds of samples or more. In such a situation, inline sensing with rapid sample acquisition may be the only practical approach.

Consult the literature

While I haven’t included a single equation in this discussion, sampling methodology can be greatly enhanced by using statistics and math. Robust statistical sampling methods exist, and a significant amount of published literature is available on this and other blending topics. In a future column, I’ll provide a guided tour of helpful powder-blending literature.

Fernando J. Muzzio is director of the National Science Foundation’s Engineering Research Center on Structured Organic Particulate Systems (http://ercforsops.org/) and Distinguished Professor, chemical and biochemical engineering, Rutgers University, Piscataway, N.J. He can be reached at 848-445-3357 (fjmuzzio@yahoo.com). He earned his BS in chemical engineering at the University of Mar del Plata, Buenos Aires, and his PhD in chemical engineering at the University of Massachusetts, Amherst. He has published more than 200 peer-reviewed papers on mixing and blending, presented at numerous conferences, and holds several patents.