How well blended is your powder mixture? How well is it even possible to mix it? Are you sure that your mixer is blending the powder properly, or is it segregating your mixture while trying to mix it? In this column we’ll talk about a good mixture.

Liquids and gases are mobile. Most liquids and gases have no structure, but powders do. Powders transfer stresses from one particle to the next to give them structure and less mobility than liquids and gases have. Even within powders, differences in structure and mobility can lead to incomplete mixing or segregated mixing. Figures 1a and 1b represent a combination of two ingredients with an equal number of particles (represented as dots). Figure 1a shows that the ingredients are completely segregated before they’ve been mixed. Figure 1b shows a perfectly ordered mix: The square outlines in Figure 1b each represent a 16-particle sample; no matter where the 4-particle by 4-particle square is drawn, the sample will have exactly 8 green and 8 blue particles.

Unfortunately, in actual powder mixing, it’s impossible to achieve this perfectly ordered mix. The best that can be achieved is a random mix. Figures 2a and 2b show two random mixes. Figure 2a shows a good random mix in which a 16-particle sample might have 10 blue particles or 6 blue particles or even exactly 8 blue particles. But some mixers or downstream handling systems can segregate the particles, as shown in Figure 2b. Here, the blue and green particles are separated enough to yield a large majority of blue particles in some samples and a significant minority of blue particles in other samples. The key to powder mixing is to get a good random mix that doesn’t segregate after mixing.

Scale of scrutiny

The figures also illustrate another key point: Sample size is critical to evaluating whether a powder has a random mix. If we change the sample size to 100 particles (the entire number shown in each image), then both Figures 2a and 2b would actually be well mixed. At a 100-particle sample size each system has 50 blue and 50 green particles. It’s not important whether the blue and green particles are mixed within the sample; we only care about the whole sample. Sample size is determined by the scale of scrutiny—that is, the sample size that must be evaluated to determine if a mixture meets its quality goals. Scale of scrutiny is critical to evaluating a good mix.

To better understand scale of scrutiny, let’s say that your company is making a sugar-free iced-tea drink-mix powder. An artificial sweetener constitutes a small portion of the overall blend. Your packaging states that the proper method to make iced tea is to use the large scoop supplied in the package to remove 1 scoop of powder and blend it with 1 gallon of water. This means that when manufacturing the powder, your scale of scrutiny should have been 1 scoop. Presumably, you’ve used a sample size equivalent to the scoop size to evaluate the powder before selling it and have made sure that each scoop-size quantity of powder contains the proper amount of the sweetener.

If the consumer decides to scoop a small teaspoon out of the package to make a glass of iced tea, then the consumer might be disappointed in the taste. You’ve only guaranteed that a large scoop sample will make the right flavor of tea. A 1-teaspoon scoop might not contain enough of the sweetener to provide the proper sweet taste.

Conversely, if you declare on the package that the consumer can scoop 1 teaspoon of powder for 1 glass of iced tea, then your scale of scrutiny should have been 1 teaspoon, and you’d better be certain that you mixed the powder to ensure that every tea...

---

spoon contains the proper amount of the sweetener to make a quality glass of iced tea.

The scale of scrutiny can become critical in products such as pharmaceuticals. For example, a drug manufacturer could guarantee a specific amount of active ingredient in a single tablet. The powder amount in the tablet is the scale of scrutiny. If a consumer chooses to purchase a double-dose tablet and split it, then the consumer may not be getting the right dosage in each half. The drug supplier only guaranteed the right dosage in a tablet, not a half tablet.

In some cases a quality random mix that meets the consumer’s requirements can’t be achieved with the proposed ingredients. The particle size and density of each ingredient affect the ability to create an adequate mix. Mixing in the Process Industries provides equations for calculating a quality mix based on particle size and density. If you’re planning a new product, you can create an Excel spreadsheet to model these equations to yield answers about the statistical probability of your mix. The key factors in the mathematical model are the particle size distribution and the scale of scrutiny. The model will show the best that can be achieved in a perfect random mix.

Let’s explain how these equations work by going back to the instant tea example. Assume the mixture must meet the following requirements:

- New instant tea drink-mix powder = 92.3 percent instant tea, 6.2 percent citric acid, and 1.5 percent artificial sweetener.
- 1 teaspoon (5 grams) of powder makes an 8-ounce glass of sweetened iced tea.
- The sweetener must meet a quality standard: The standard variation per sample must be within a 3-sigma requirement of ±20 percent relative standard deviation (RSD) from the target. The sweetener target is 75 milligrams in every teaspoon. At 3-sigma RSD, the quality requirement then demands that every 1-teaspoon sample contain between 60 and 90 milligrams of artificial sweetener.

Let’s also assume that the ingredient particle sizes are measured in a sieve shaker, and that the median particle sizes are:

- Instant tea = 200-micron $D_{50}$ particle size range
- Citric acid = 800-micron $D_{50}$ particle size range
- Artificial sweetener = 900-micron $D_{50}$ particle size range

Once you run the mathematical model using the spreadsheet, you find that the sweetener’s particle size distribution is too large to adequately mix this ingredient at the required sample size. For the conditions above, in an ideal mix, the sweetener can only achieve 37 percent 3-sigma RSD. This means that any random 1-teaspoon sample taken from the finished drink-mix powder will have between 47 and 103 milligrams of sweetener when the desired range is 60 to 90 milligrams. If you use the sweetener at the supplied particle size distribution, the product will be too sweet in some cases and not sweet enough in others. It’s far out of range from the required ±20 percent RSD.

### Problem-solve

To fix this problem, you can take either of two approaches: First, you can change the scale of scrutiny. If you change it to 1 scoop of powder to make 1 gallon of iced tea, then the RSD for the artificial sweetener drops to a 10 percent 3-sigma RSD, which meets the quality requirement. The target for each scoop would be 1.2 grams of sweetener, and every scoop would have between 1.08 and 1.32 grams of sweetener. Your product instructions to the consumer would have to be modified to require the consumer to make 1 gallon of iced tea with 1 scoop of powder. If the customer makes just 1 glass of tea with 1 teaspoon of powder, the sweetness wouldn’t necessarily be satisfactory, and none of us want dissatisfied customers!

As a second option, to guarantee the quality of a 1-teaspoon scoop, you would have to micronize the artificial sweetener to get its particle size distribution small enough to produce an adequate mix. By grinding the key ingredient, you’ll generate many more particles of the active material per gram of mix. This will help achieve a random mix. If you micronize the sweetener from the 900-micron $D_{50}$ particle size range to 200-micron $D_{50}$ particle size range, then the RSD per teaspoon of sample drops to 4 percent over a 3-sigma quality requirement. For a 1-teaspoon sample that should contain 75 milligrams of sweetener, this means that every teaspoon will have between 72 and 78 milligrams. This is well within the product requirements, and it’s much better than the 47- to 103-milligram range of the previous blend using nonmicronized sweetener.
This simple three-component example exercise shows that particle size and scale of scrutiny significantly affect the statistical capability of a mix. Multicomponent blends with several key ingredients are much more complex, and it’s much harder to achieve a random mix. Smaller particles and larger dosage sizes will give a more statistically achievable mix. The exercise also shows that good random mixes might not be statistically achievable with the ingredients you’ve planned to use. If they’re not, then you have to take measures to make the mixture statistically achievable. In the next column we’ll discuss how to choose the appropriate mixer to properly make the statistically achievable blend.

Reference

*James L. Davis, PE, is president of Powder Processing solutions (www.powderprocessingsolutions.com) and a consulting engineer specializing in solving difficult powder processing problems and optimizing complex powder systems for efficient operation. He was with Procter & Gamble for 26 years, 15 of them in powder processing. He holds a BS in mechanical engineering from the University of Cincinnati. Jim will be presenting two seminars at PBE’s Northeast Conference & Expo in Somerset, N.J. May 23-25, 2011. See www.powdershow2011.com for information.*