MIXING MECHANICS

James L. Davis

Keys to mixer testing

In the April column, I discussed how achieving a good mix depends on achieving an effective "marriage" of the particle characteristics, process conditions, and mixer type. I also discussed the various mixer types and gave examples of good unions of particle characteristics, process conditions, and mixer type. So, let's assume that we need to buy a new mixer and, after doing some research, believe that we have chosen a good mixer for our blend. How do we know? What criteria do we use to determine if the mixer we've chosen will do a good job?

There's variation with every mixer and every blend. As I noted in the April column, an ideal or perfectly ordered mix can't be achieved. Instead, a good random mix is the desired goal. Every blend has a key ingredient or two that must be distributed appropriately throughout the blend. For example, let's say our end product is acetaminophen pain-relieving tablets. We want our blend to be such that every tablet will contain the perfect amount of the key ingredient, acetaminophen, but this won't always happen. Each tablet is supposed to contain 325 milligrams of acetaminophen. Yet with standard variation, each tablet's acetaminophen content might be anywhere between 300 and 350 milligrams. This is within acceptable FDA guidelines.

To be certain a new mixer will achieve a blend within our product's acceptable variation, we need to perform testing on a pilot or lab-scale mixer. This entails grabbing samples from the test mixer that have a size equal to the *scale of scrutiny* (the size of the desired product, whether it's a tablet or another shape; find more information in the September 2010 column). Once we're satisfied that the test mixer does the job we need, we'll have to ensure that the same results can be achieved with the production mixer we purchase. Again, testing will be required.

Mixing time

Mixing time is a key to achieving the right blend. If a mixer isn't operated long enough, we'll have an incomplete mix. If it runs too long, the correct blend might be achieved and then deblend. Figure 1 illustrates mixing progress over time. The x axis represents mixing time, and the y axis represents the *mixing index*. The mixing index is a dimensionless value on a 0 to 1 scale; the closer the value is to 1, the better the quality of the mix. The mixing index is represented by the following equation:

$$M = (S_o^2 - S_{ex}^2) / S_o^2 - S_r^2)$$

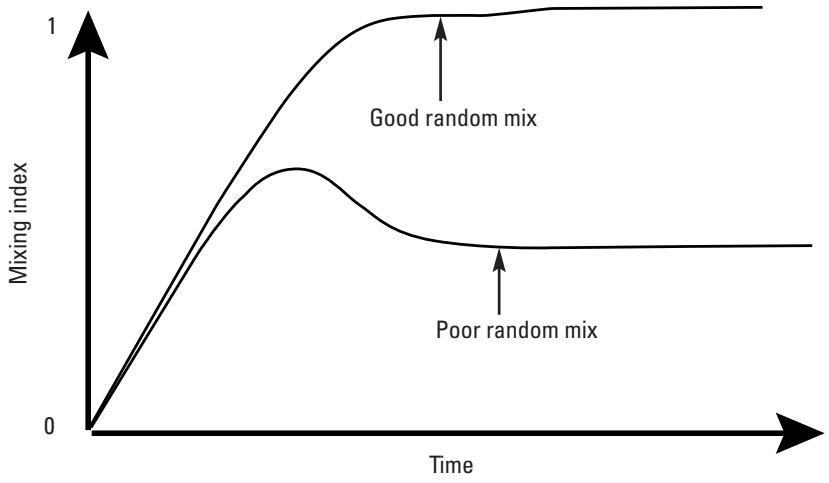
where M is the mixing index, S_o^2 represents the initial unmixed *material variance* (degree of blending) or completely segregated state, S_{ex}^2 represents the experimental variance at a given mixing time (that is, the blend's variance at the time a particular sample is taken), and S_r^2 represents the best variance that can be achieved in a truly randomized mix.

As the experimental mixing variance reaches the best-case randomized variance, the mixing index reaches 1. Figure 1 illustrates two examples: The top curve shows that a good random mix has been achieved with a combination of the correct mixer and the appropriate mixing time. The bottom curve shows that a truly random state wasn't achieved at any blending time. This shows that the mixer was a poor choice for this product.

The recommended method for doing this testing is to mix a batch for a set time period — for example, 5 minutes — and then take samples while the material discharges from the mixer. Then prepare a second batch and mix it for another time period — for example, 10 minutes. A third and fourth mixing time should probably also be chosen, and appropriate samples should be taken from all of the

Figure 1

Mixing progress over time



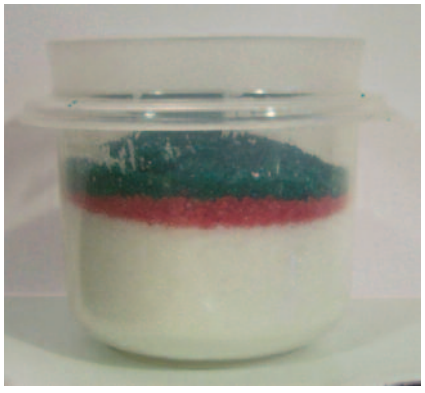
batches. This method of evaluating mixing times is preferred over running just one batch and taking samples from that batch at various times using a sample thief.

A note about sample thieves: A sample thief is essentially a hollow tube within a hollow tube; it's lowered into a material bed, then twisted to expose an opening in the tubes. Material enters the hole, the tubes are twisted to close the opening, and the thief is removed from the material bed. Figure 2 shows how a sample thief can provide false data: In Figure 2a, sugar crystals are layered (segregated) in three colors, and in Figure 2b, a pencil representing a sample thief penetrates the sugar, dragging down the top layers. If the pencil were an actual sample thief, the resulting sample would misrepresent the material as being mixed when in reality there was no mixing at all. This is why sampling multiple batches *after* the blending has taken place is the preferred sampling method when evaluating a mixer.

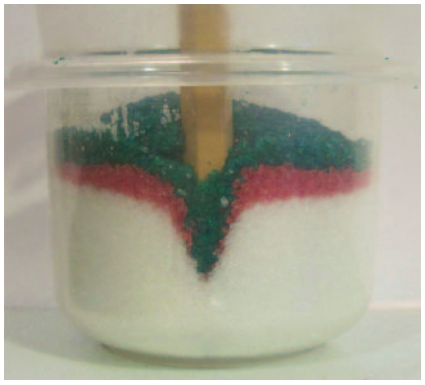
Figure 2

Effect of using a sample thief with sugar crystals

a. Layered sugar crystals



b. Top layers dragged into bottom layer



When, where, and how to sample

Now that we know we're going to take multiple samples, the next questions are when, where, how much, and how often to sample. The ideal method of sampling powders is to sample from a flowing stream. With a mixer, the flowing stream occurs during its discharge. This takes a lot of upfront planning: We'll need to have sample cups prepared and staged before the mixing test is even started so that we can take samples quickly. Each sample cup will be used to dip into the flowing stream and grab a portion of it. Having the cups ready beforehand allows us to quickly take samples when mixer discharge starts.

From a statistical perspective, we'll need to take 20 samples or more to have a 95 percent confidence level in the final data's accuracy. In some cases, we may want more than 20 samples. With more samples there's less sampling variation in the final data.

The correct sample size for the acetaminophen tablet is the amount of powder needed to make one tablet, which is about 0.5 gram. Grabbing an individual sample that small is difficult, so a larger sample can be taken, say 5 grams, then a small 0.5-gram sample can be scooped from the bulk sample. (Usually scooping isn't a preferred sampling method because it may not be representative of the whole blend. But in this case, with such a small scale of scrutiny, scooping from the sample is satisfactory.)

Sample timing should be random. If the mixer takes 2 minutes to discharge and we want 20 samples, then we would expect to take a sample every 6 seconds. Yet we don't want to grab the samples in such an ordered way. If segregation is going to occur during discharge, it will happen at the beginning and end of the discharge period. So, it would be better to take 5 samples in the first 10 seconds of discharge and 5 samples very near the end of the discharge sequence. The remaining 10 samples can be taken somewhat more evenly throughout the remaining 70 seconds of discharge.

Even with those middle samples, some randomness is preferred. They shouldn't be taken exactly every 7 seconds. Mixers can sometimes cause a slight *segregation periodicity* (slightly segregating at regular intervals) during discharge. If the tested mixer has a 7-second segregation interval and samples are taken every 7 seconds, the samples would mask this. So over the 70 seconds, one sample might be taken 4 seconds after the previous sample, the following sample might be at 10 seconds later, and so on. The randomness will help uncover any segregation intervals that might exist.

Now that we've got the samples for one blend time, we have to do it again for additional blend times. Ultimately, all the samples will be gathered and sent off for lab analysis.

The lab technicians, too, must take care to not create segregation. For example, let's say that we sent 0.5-gram samples. If the lab analysis requires only 50 milligrams of powder, then the technicians have to cut the samples properly. They can't simply scoop the powder off the top of each sample, because the sample may have some segregation. They have to use a tool such as a chute riffler or spinning riffler[1] to separate the powder and cut it down to a representative amount for testing.

Evaluating the data

When all the results are in, we need to evaluate the data. Using the equations in a spreadsheet model as described in the September 2010 column, we can calculate the mixture's random variance. The data gathered during the testing will give us the experimental variance. With these figures, we can calculate the mixing index. With luck, at least one of our batches will have a

mixing index approaching 1. But even if that's the case, we may have to consider other factors before selecting this mixer.

For example, a product like acetaminophen has to meet complex and specific FDA guidelines. One common guideline is that all samples have to be within ±10 percent of the claimed active ingredient content. That's for the end product. Since some segregation may occur in the handling steps after the mixer, the target mixer variation should be closer to ±5 percent. This equates to the very high mixing index of 0.95. It means that every sample of powder should have between 309 and 341 milligrams of acetaminophen.

So if our first batch has excessive variations, more mixing time may be needed. If the excessive variations primarily show up in the samples taken at the beginning and end of the mixer discharge, it may mean that the mixing discharge causes segregation. If none of the batches that were run meet the required 0.95 mixing index, or if excessive variation at the beginning or end of discharge occurs, it means that the mixer chosen is unsuitable for this blended product. We'll have to evaluate a different mixer. **PBE**

Reference

1. For information about these tools, see articles listed under "Sampling" in *Powder and Bulk Engineering*'s comprehensive article index (in the December 2010 issue and at *PBE*'s website, www.powderbulk.com).

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 seminars at PBE's Southeast Conference & Exhibition in Atlanta, March 13-15, 2012; see www.powdershow2012.com for information.*

The author will answer your questions in future issues. Direct questions to him at jimdavispe@gmail.com or via the Editor, *Powder and Bulk Engineering,* 1155 Northland Drive, St. Paul, MN 55120; fax 651-287-5650 (toneill@cscpub.com).