

Answers to 10 common questions about batch mixing

Basil J. Michel The JSJ Group, Ltd.

Proper mixing is one key to controlling quality in many processing operations. In this first of a question-and-answer series on common powder and bulk solids handling problems, a consultant answers 10 of the batch mixing questions he's commonly asked.

Choosing a batch mixer

Q: I'm confused by the variety of mixers available. How do I know which type to choose for my batch process?

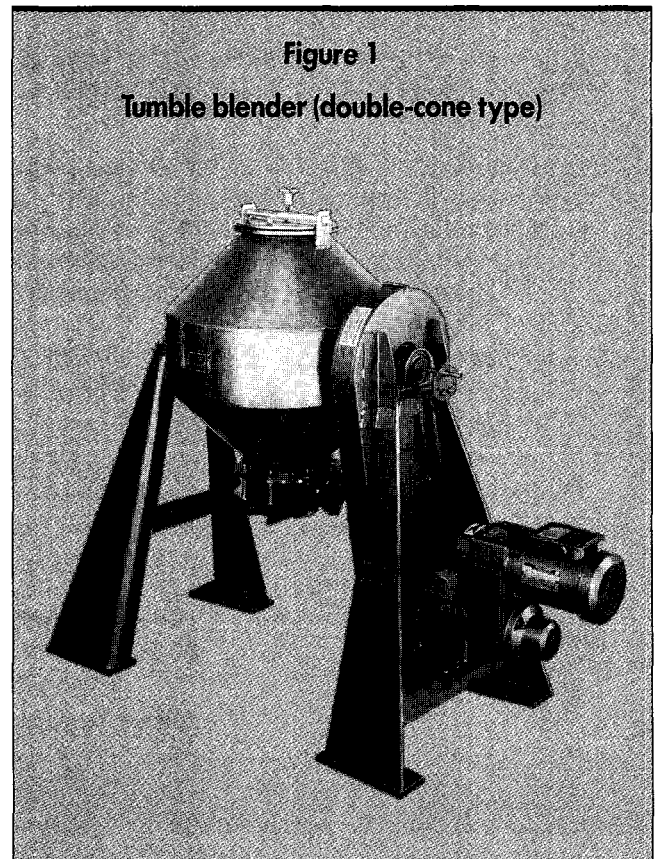
A: Three basic types of mixers are available for batch operation: a tumble blender, an impeller-driven mixer, and a multiple-motion mixer. All three can work well for mixing, but each is best suited for a slightly different application.

A tumble blender (Figure 1) is an enclosed vessel mounted on trunnions or legs. In operation, the vessel revolves end over end, gently mixing ingredients while causing only limited particle size reduction. The unit is commonly used for friable materials and pharmaceuticals. A tumble blender can be equipped with a high-speed processing bar, which is a rotating bar mounted across the vessel's center. The bar creates some shear during mixing and is often used when some particle size reduction is desired. More commonly, the bar is used for wet granulation, in which a liquid binder is added to the vessel to attach small particles to larger ones to prevent segregation during later processing steps. During wet granulation, the bar helps fluidize the particles before they contact the liquid binder.

An impeller-driven mixer (Figure 2) typically has a U-shaped horizontal trough with an impeller that consists of a shaft mounted with plow blades or pitched paddles. Two common impeller-driven units are a ribbon mixer and a plow mixer. In operation, the impeller rotates, aerating the particles and creating a fluidized bed that produces excellent mixing results. The

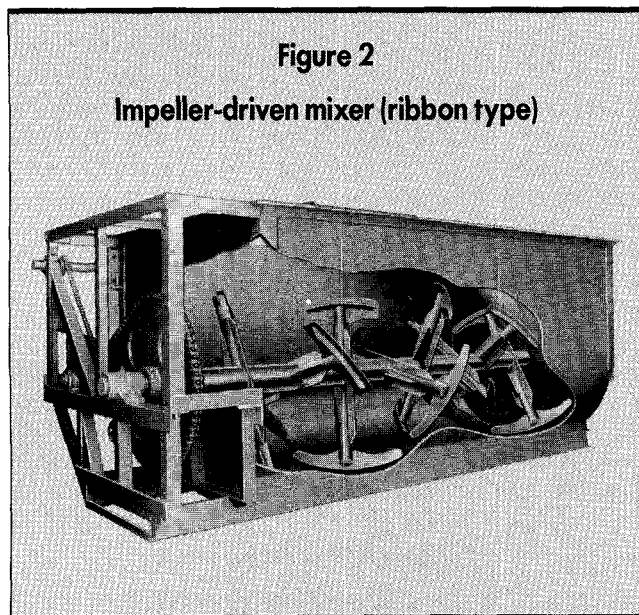
impeller's action creates more shear on the particles than a tumble blender's action, but usually causes only slight particle size reduction. The mixer is sometimes used for mixing pastes.

A multiple-motion mixer can be one of two types: a double-shafted mixer with paddles or plow blades or a kneading mixer. A double-shafted mixer (Figure 3) typically has two horizontal impellers consisting of shafts mounted with paddles or plow blades; the paddles or blades on one shaft overlap those on the other shaft. In operation, the impellers counter-rotate, fluidizing and mixing the material. The mixer is used for powders and some moist materials. A kneading mixer (also called a double-arm

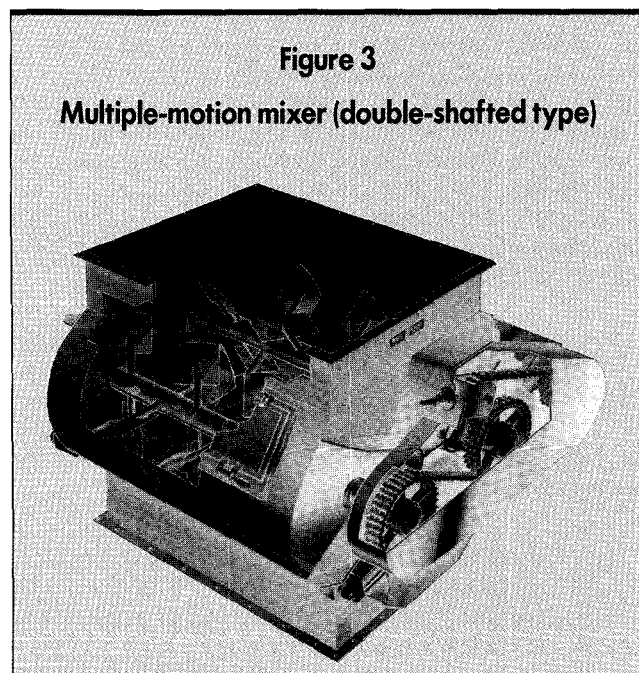


Courtesy Paul O. Abbe Inc., Little Falls, N.J.

kneading mixer) typically consists of either a horizontal or vertical cylindrical housing enclosing two impellers. The impellers sometimes can rotate around each other, creating planetary rotation, and can also overlap or intermesh. In operation, the impellers counter-rotate, fluidizing and kneading the mixture. The mixer is frequently used for mixing pastes, because its kneading action allows reactions to go from dry to paste phases when liquid is added. The kneading action also makes the mixer suitable for drying mixtures to produce solids or powders. When used for drying, the mixer uses a heated air stream, a heat jacket, or even an open flame to dry the materials and, typically, a small purge stream to remove moisture vapor.



Courtesy H. C. Davis Sons Manufacturing Co., Inc., Bonner Springs, Kans.



Courtesy Paul O. Abbe Inc., Little Falls, N.J.

Determining mixing quality

Q: How can I tell when my material has been adequately mixed?

A: Unlike liquids, which mix to a molecular level, dry materials comprise discrete particles and, in most operations, depending on the sample size, the particles are never truly mixed. For instance, if a sample consists of one particle, the sample isn't mixed. Thus, determining whether a mixture is adequately mixed really depends on how the material will be used in the next process step or what the end user needs. These factors also affect the sample size you need to consider.

For instance, to determine whether a mixture fed to a reactor is adequately mixed, the sample size you need to consider is that of the container used to feed the reactor. If the mixture in the container consists of correct proportions of all ingredients, even though the ingredient concentration differs throughout the container, the correct ingredient proportions will reach the reactor, so mixing is adequate. On the other hand, if the mixture will be fed to a tablet press in a pharmaceutical operation, you need to consider whether a mixture sample equal to the tablet's size is adequately mixed.

To determine whether a mixture is adequately mixed, you can use techniques ranging from observing the mixture's gross characteristics, such as color, to very sophisticated laboratory methods for determining the active ingredient concentration in relatively small samples. Follow these general rules:

1. If possible, observe the mixture or take the sample in the mixer's vessel so the mixture isn't transferred before potential mixing problems can be corrected.
2. If you can't observe the mixture or take the sample *in* the mixer's vessel, do so as close as possible to the mixer. This way, you can catch an off-spec mixture before it's further processed or packaged, helping to eliminate rejected batches and to hold costs down.
3. When taking a sample, select a sample size equal to that required by the next step in the process (for instance, in a tableting operation, equal to the tablet's size).

Though it can be difficult, make sure you take representative samples. If you use a probe or *thief-type* sampling device, insert it into the mixer vessel's center and avoid areas near the axis of rotation or internal seals. Be aware, however, that taking samples inside the vessel can't account for later variations caused by segregation when the material is discharged or transferred. To detect such segregation problems, sample your mixture twice: once in the mixer vessel and once just before the mixture is packaged.

The number of samples you take depends on your required mixing accuracy. In general, the smaller the sample and the more critical the ingredient concentration, the more samples you'll need to take.

Once you collect the samples, you can use chemical testing, tests based on weight variation (for instance, testing differences in the weights of food packages), and colorimetric techniques (which use electronic sensing combined with visual observation) to determine mixing quality.

Segregation problems

Q We use a tumble blender. I've heard that if you mix too long, you can undo the mixing results. What is this "demixing" and what causes it?

A Unlike most liquid blends, in which mutually soluble liquids form a stable blend that doesn't degrade over time, dry mixtures have a tendency to segregate. This is because particles have different sizes, shapes, and densities. When the particles are in motion (as in mixing), the differences cause the particles to segregate.

In a tumble blender, the ingredients are loaded in batches, often forming stratified layers in the vessel. Then the unit begins tumbling, and its symmetrical rotation creates a nonrandom mixing pattern. This pattern, when combined with the various particles' different trajectories, can demix or segregate the particles in a pattern different from that formed by the layers during loading. To avoid this segregation in the later mixing stages, it's best to use a tumble blender for relatively short mixing times. A tumble blender can also be designed to provide asymmetrical rotation to minimize demixing caused by trajectory segregation. For instance, a V-blender can have one extended leg and a double-cone mixer can have offset cones; by using such designs, you can shorten mixing times, improve mixing quality, and limit further segregation.

Q Can demixing occur in an impeller-driven or multiple-motion mixer?

A While demixing is less likely in an impeller-driven or multiple-motion mixer than in a tumble blender, it can still occur. During mixing, an impeller-driven or multiple-motion mixer fluidizes the materials. If the batch is discharged during operation, mixing — while not totally random — is relatively uniform, without segregation. However, when the mixer stops operating, the fluidized mass settles, and heavier particles drop to the vessel's bottom, creating stratified particle concentrations. You can avoid such demixing by discharging the mixer during operation. If the normal mixing speed creates discharge problems, such as dusting, try using a two-speed motor to decrease the impeller speed during discharge.

Q When I analyze material samples taken from our mixer vessel, the mixing quality is okay, but I find mixing quality and weight variations in the final packages. How can I avoid this?

A A mixture like yours, which segregates after it leaves the mixer, can cause major problems. For instance, with pharmaceuticals, critical concentration variations in a tablet's active ingredients can be harmful to users. With food products, when volumetric feeders are used to fill packages to a specified weight, changes in the mixture's bulk density can cause package weights to vary and generate consumer complaints. (Though overfilling the packages can stop consumer complaints, this solution is costly.)

Segregation can occur every time dry materials are moved from one process or container to another — for instance, from a mixer to a storage hopper or from a storage hopper to a packaging unit's

feed hopper. In fact, many problems attributed to mixing are instead segregation problems that have occurred after mixing.

The two most common types of segregation that affect final packaging are sifting segregation and fluidizing segregation. In *sifting segregation*, fine particles concentrate at the center of a heap of material as the material is discharged into a container, while coarse particles, unable to fit in the heap's center, roll to the outside of the heap. This creates segregation in a radial direction (from the heap's center to its perimeter) in the container. In *fluidizing segregation*, fluidized materials settle in a container, with coarse particles settling on the bottom and fine particles accumulating on the top. This creates a concentration variation in an axial direction (from top to bottom) in the container. Analyzing your process to determine which type of segregation is affecting your material will help you prevent the problem.

To avoid segregation, design your processing line with as few transfer points as possible between the mixer and the final step. Also avoid using free-fall chutes or drops in the processing line. Finally, use controlled-discharge mass-flow patterns to help remix the material. In a sifting segregation case, use a container discharge that has absolutely uniform velocity across the hopper in a mass-flow discharge pattern. The uniform discharge velocity from the outside to the inside allows the segregated fine and coarse particles to reach the discharge simultaneously, remixing them, though not achieving the same mixing quality as the mixer. In a fluidizing segregation case, use a container discharge that has a velocity gradient (with fastest discharge at the center and slowest discharge at the outside), which will blend the stratified layers to a more uniform mixture. This achieves better mixing than a uniform-velocity mass-flow discharge, but still won't achieve the same mixing quality as the mixer.

Other mixing problems

Q What's the quickest way to determine if my mixer is causing particle degradation?

A Particle degradation can be deterioration in quality from a reaction (such as spoilage of a food product) or size change (such as breaking friable particles). Deterioration in quality is usually obvious from simply looking at the mixture. Size change is less obvious, so you'll need to compare a mixture sample with samples of the ingredients and good-quality final product. One way to do this is to fill clear glass jars or vials about half full with samples of the mixture, the ingredients, and good-quality final product. By rotating the jars or vials by hand, you can spot significant differences in the samples' flow characteristics, which are usually caused by particle size changes.

Q My material goes through a wet granulation step in a tumble blender to avoid segregation downstream. But the granulated material contains agglomerates, formed by liquid droplets in the blender, and I have to treat the agglomerates before I can use the material downstream. How can I avoid the agglomerates?

A Wet granulation to prevent later segregation is a tricky operation because it's difficult to disperse the liquid uniformly through the batch to get good liquid-solid contact. The agglomerates you describe are usually formed when the liquid is introduced within an unfluidized or only partially

fluidized portion of the material bed. When improperly fluidized, the material can block the liquid's entrance, forcing the formation of droplets and, hence, agglomerates. To prevent agglomerates in your tumble blender, you can add the liquid through a stationary spray device, which is a hollow pipe located in the mixer's void space (above the material bed). To ensure that the material passing the high-speed processing bar is well fluidized, you can also reduce the material bed's depth. The spray device then adds the liquid to the most aerated portion of the material bed, significantly improving granulation and preventing the formation of liquid droplets and, hence, agglomerates. However, reducing the material bed can slow productivity because you add less material to the mixer.

Choosing a mixer that can handle dry and paste phases

Q Most of the processes in my specialty chemical company are batch and handle only dry materials. However, some materials go through a paste phase. Can one mixer handle both dry materials and pastes?

A Yes, but your company will have to invest more capital in the more sophisticated mixer required, when for most of your processing a less sophisticated mixer (for instance, a tumble blender, which handles only dry solids and is simply constructed and thus less expensive) is all you need. If you need to handle both dry and paste phases, you can select a ribbon mixer, which can handle dry materials and, usually, pastes; however, this impeller-driven mixer is more expensive than a tumble blender. If your paste is very difficult to handle, you can select a multiple-motion mixer with impellers that rotate with planetary motion or intermeshing sigma blades, but this is even more expensive.

To determine which mixer is suited to handling your paste without being overly sophisticated or expensive, have the paste tested. *Paste* — a material partway between a dry material and a liquid — can mean different things to different people and can't be as easily characterized as a dry material. To doublecheck that the equipment will work with your dry material as well as your paste, especially if you select a multiple-motion mixer, have a small sample of the dry material tested. This will determine, for instance, whether the multiple-motion mixer's higher shear rate will degrade the dry material during your simpler mixing processes.

Typically, the mixer manufacturer can test a small sample of your material in various mixers with different impeller arrangements, depending on your material's characteristics. Many manufacturers perform such tests at no charge.

Adding a minor ingredient

Q To properly package our material, we add an active ingredient in less than 5 percent concentration to a carrier that acts almost like a diluent. What kind of mixer should we use, and how should we add the ingredients to get good mixing?

A The kind of intimate mixing you're describing typically requires a high-speed processing bar or impeller. In general, a tumble blender with a high-speed processing bar will do the best job of mixing minor ingredients, but you should test your material in the unit to be sure its shear rate doesn't degrade the particles. You can also use an impeller-driven mixer, particularly if equipped with a plow blade, for mixing minor ingredients.

The way in which you add the materials also affects the mixing quality. For best results, regardless of the mixer type, first add one-half of the bulk materials (your diluent), then add the minor ingredient (the active ingredient) into the batch's center, then add the remaining materials (the rest of the diluent). If you add the minor ingredient first, it can adhere to the vessel walls and deposit itself in a slow-moving portion of the mixer (for instance, near a tumble blender's axis of rotation) or material bed or get caught in a slow-moving, symmetrically rotating area of the material bed where mixing is slower.

Mixing and direct compression

Q Our pharmaceutical process currently requires a granulation step. We'd like to skip this step and the processing and equipment it requires and go to direct compression. What should we consider before making this change?

A With pharmaceuticals, using direct compression (transferring a mixture from a mixer directly to a tablet press) typically involves a mixture with a low concentration of active ingredients. This can cause the same problems as with mixing minor ingredients. For instance, if the size difference between the largest and smallest particles is 5 to 1 or greater, the mixture is likely to segregate. In some situations, direct compression can be dangerous because it makes controlling mixing quality difficult. With certain pharmaceuticals with a particle size difference 200 to 1 or greater (such as 200-micron excipients mixed with 1-micron active ingredients), segregation is so significant that you may have to avoid direct compression and use granulation to maintain the final product's integrity. In some cases, you can avoid segregation problems by reducing your material's particle size difference (for instance, by grinding the larger particles) to less than 5 to 1.

If you can't reduce the particle size difference, reduce segregation problems by using few transfer points and remixing the material in the hopper or other container just prior to loading the tablet press. (The material can be remixed by using equipment designed to rotate and tumble-blend the hopper or other container.) If making these changes doesn't eliminate segregation problems, use granulation — either wet, by adding a liquid binder, or dry, by compressing the mixture between rollers. In either case, you'll need additional equipment, which will raise the system's cost.

PBE

Suggested reading

Clark A. Beebe, "Tumble blending systems: A productivity and cost comparison," *Powder and Bulk Engineering*, January 1988, page 34.

A. Kirk Brennan Jr., "Selecting the right mixer: Batch or continuous," *Powder and Bulk Engineering*, January 1990, page 38.

"Emerging technology: Making higher quality mixtures in less time," *Powder and Bulk Engineering*, January 1989, page 35.

Calvin E. Johnson, "Preconditioning process powders with dry granulation," *Powder and Bulk Engineering*, December 1987, page 14.

Brian H. Kaye, "Using an expert system to monitor mixer performance," *Powder and Bulk Engineering*, January 1991, page 36.

Brian S. Mawson, "Selecting a mixer with help from your mixer manufacturer," *Powder and Bulk Engineering*, January 1991, page 42.

Geoffrey Mound, "An introduction to wet agglomeration," *Powder and Bulk Engineering*, February 1991, page 47.

Basil J. Michel, PE, is president of The JSJ Group, Ltd., PO Box 18235, Rochester, NY 14618; 716/247-8900. He holds BS and MS degrees in chemical engineering from the University of Rochester in Rochester, N.Y. The JSJ Group provides a range of technical consulting services, most of them for mixing applications.