Five Tips for More Efficient Powder Dispersion

A White Paper Prepared By Charles Ross & Son Company

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Abstract

This white paper explores five tips for dispersing powders into liquid more efficiently. The ideas discussed in this paper are recommendations based on Ross' collective experience as a provider of specialty mixing equipment to the process industries for over 170 years. Mixer testing and simulation trials are recommended to confirm the suitability of a specific mixing strategy.

Introduction

The inability to uniformly disperse powders into a liquid batch within a practical amount of time is an all-too-common mixing problem that impacts productivity, labor costs and power consumption.

Issues associated with inefficient powder dispersion include:

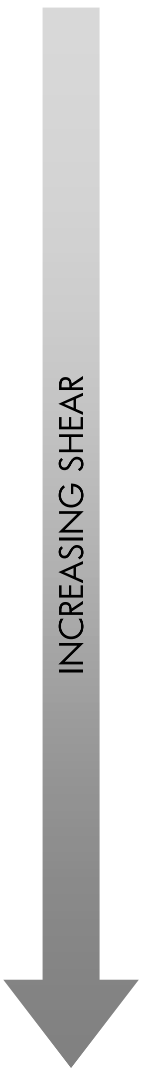
- Stubborn agglomerates or "fish eyes"
- Floating powders
- Dusting
- Poor product quality
- Inconsistencies from batch to batch
- Waste of raw materials
- Need for extra processing steps (sifting powders, filtering out remaining lumps, milling, etc.)
- Frequent rework
- Low throughput / overly long cycle times
- Clogging of downstream equipment

Needless to say, the degree of mixing difficulty varies from one particular formulation to another. The following five tips are meant to serve as a general guide and starting point for process improvement.

1. Shear up!

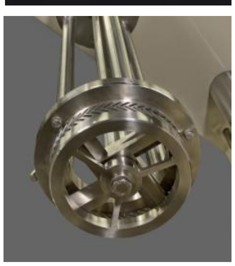
Mixing kettles and batching tanks equipped with the traditional low-speed impeller, propeller or turbine-type agitator are generally effective for straightforward liquid-liquid blending. However, yield and cycle time are not always optimized when the process involves large amounts of powders that need to be uniformly dispersed or quickly dissolved.

High speed, high shear agitation is commonly required when incorporating solids into liquid especially when the dry ingredients are difficult to wet out. Below is an easy guide for some mixer designs that are well proven for powder dispersions.



Running at tip speeds around 5,000 ft/min, the sawtooth disc-style **High Speed Disperser** creates vigorous flow and imparts greater shear compared to other open-blade mixing devices such as propellers or turbines. It produces a vortex on the liquid surface into which solids can be added for quick incorporation and wet-out. High Speed Dispersers are normally used in applications up to around 50,000 centipoise (cP) in viscosity.

In comparison, a classic **High Shear Mixer** features a four-blade rotor running at tip speeds in the range of 3,000-4,000 ft/min within a fixed stator. This type of device generates mechanical and hydraulic shear by continuously drawing product components into the rotor and expelling them radially through the openings in the stator. Because of the restriction provided by the stator, this mixer offers higher shear but less pumping capacity than an open-disc sawtooth disperser blade. For the same reason, its viscosity limit is lower – around 10,000 cP.



Top-entering Ultra-High Shear Mixers like the **Ross PreMax with Delta Rotor/Stator** (US Patent No. 6,000,840) combine high pumping capacity and shear intensity. Turning at tip speeds as high as 5,000 ft/min within a close tolerance stator, the Delta Rotor draws product from above and below the mixing head. Compared to other batch style mixers including sawtooth dispersers, traditional rotor/stator mixers and immersion mills, the PreMax delivers more superior particle size reduction.

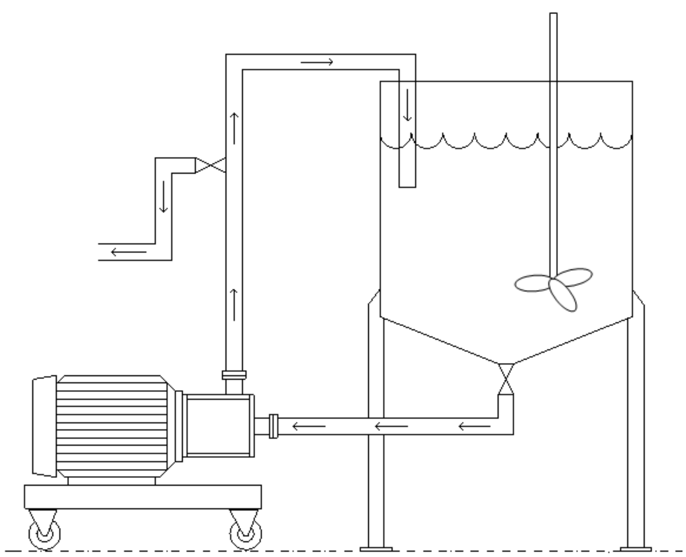
For processes requiring a very fine distribution, inline Ultra-High Shear Mixers offer a viable alternative to more expensive colloid mills and high pressure homogenizers. Designs like the Ross **X-Series** (US Patent No. 5,632,596), **MegaShear** (US Patent No. 6,241,472) and **QuadSlot** feature unique rotor/stator geometries and are engineered to run at tip speeds over 11,000 ft/min. These Ultra-High Shear Mixers impart extremely high levels of hydraulic and mechanical shear without sacrificing throughput.

2. Consider the benefits of recirculation.

As a follow-up to the first tip, keep in mind that increasing shear input does not necessarily require a complete overhaul or replacement of your existing mixer set-up. One of the simplest ways to accomplish this is to install a recirculation line so an inline mixer can apply the supplemental shear and agitation. This is often a practical option because the inline device does not compete for space at the top of the vessel nor disturb pre-existing equipment and connections.

In a typical set-up, liquids and solid raw materials are first combined in the tank then recirculated through an inline High Shear Mixer. When the desired level of dispersion and homogenization is reached, the recirculation line is closed and the mixer serves as a transfer pump, moving the finished mixture to a nearby collection tank or downstream process.

In addition to boosting production by reducing cycle time, an inline High Shear Mixer offers the advantage of servicing multiple batch tanks virtually regardless of size. It can handle a 2,500-gal stirred tank as easily as it can turnover a 25-gal batch of the same viscosity, i.e. the mixer is capable of a certain flow rate which is not dependent on tank capacity. The larger batch will simply take longer to turn over. Fortunately, recirculation enables precise control over the mixing process in the sense that the number of passes through the high-shear zone can be monitored with confidence. With appropriate piping, simple valves can be used to divert finished product downstream and switch instantly from one source vessel to another.



Inline high shear mixer piped to a batch tank for recirculation and pumping downstream.

3. Tap the power of vacuum.

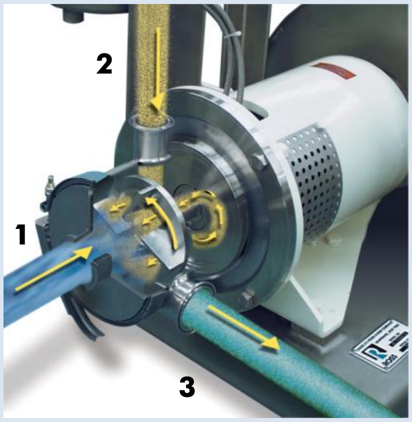
Pulling vacuum to introduce powders into liquid is not a new concept but the technique has changed significantly over the years. The traditional approach is to use an external vacuum pump to establish negative pressure in the head space of the vessel. Solids are typically introduced through a bottom port and the point of entry is in close proximity to a high speed agitator. This helps to wet-out the powders before they reach the liquid surface. This method requires a skilled operator to carefully monitor the vacuum level and make frequent adjustments based on the rate of powder injection. Too strong a vacuum can cause powders to shoot out of the liquid and dust, float or form lumps. On the other hand, a weak vacuum will not motivate the solids to enter the batch. In reality, it is a very difficult balancing act which can lead to variable cycle times and product quality.

A more efficient way of utilizing vacuum for powder injection does not require a pump at all. Technologies like the Ross Solids/Liquid Injection Manifold (SLIM) combine high speed and high shear mixing with the power of vacuum. But instead of relying on an external vacuum pump, the SLIM utilizes a specially engineered rotor/stator assembly that homogenizes like a High Shear Mixer while also generating a vacuum for drawing solids into liquid. The system allows the operator to inject powders at a fast but controlled rate for highly repeatable and superior dispersion results. Powders encounter the liquid phase not only sub-surface but also where shear conditions are most intense. By preventing the formation agglomerates or "fish eyes" in the first place, the SLIM ensures maximum yield of challenging powders like gums, alginates, carbomers, cellulose, fumed silica, pectin, etc. Revealing full functionality through efficient mixing lowers raw material costs and eliminates downstream filtering or rework operations.

To minimize transfer steps, powders may be suctioned right out of their original container using a flexible wand. This technique is particularly useful in reducing dusting in the mixing area and suitable for powders that are relatively lightweight and free-flowing. Another method is to transfer powders into an asymmetrical hopper directly above the mix chamber for faster powder induction aided by gravity.

Ross SLIM installations at a pharmaceutical laboratory (top), a cosmetics plant (middle) and a chemical factory (bottom).

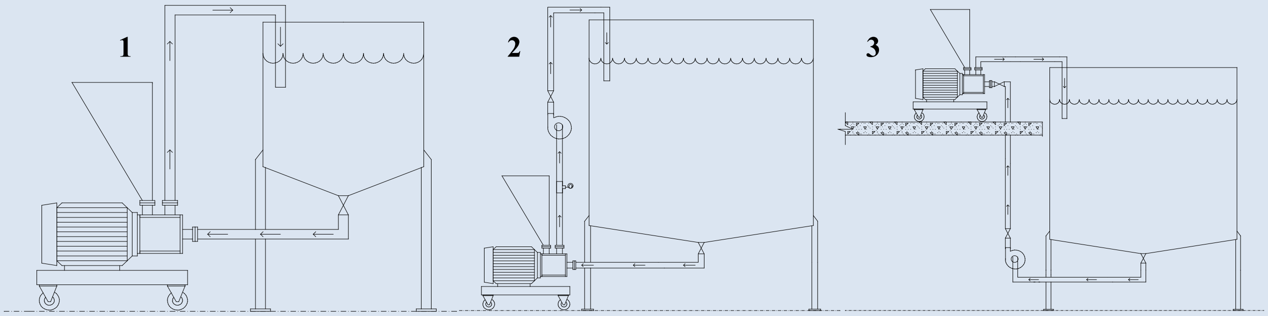
ANATOMY OF A POWDER INDUCTION MIXER



The liquid stream (1) enters the inline SLIM Mixer and immediately encounters the powder injection (2) at the high shear zone of the rotor/stator assembly. The resulting dispersion (3) is expelled centrifugally through the stator openings at high velocity.

After injecting the solid components of a recipe, the SLIM may also be used to deliver liquid additions.

TYPICAL INLINE SLIM INSTALLATIONS



Scenario #1 describes the inline SLIM Mixer being used with a small to medium size (50 – 1000 gallon) recirculation vessel. The mixer is located on ground level and is as close to the outlet of the tank as possible. Discharge tubing is kept to the minimum possible length.

Scenario #2 illustrates a typical SLIM set-up for large (1000+ gallon) recirculation vessels. Since the discharge tubing is long, a centrifugal pump is used to minimize backpressure on the discharge side.

In Scenario #3, the mixer is located on an elevated mezzanine or upper level floor. The SLIM Mixer is not self-priming so a centrifugal pump is used to help feed the inlet side.

ORDER OF ADDITION: SOME RULES OF THUMB

✓ The liquid vehicle, mixed with any dispersing agent (if applicable), is first recirculated through the SLIM. As the rotor reaches operating speed, the SLIM's solids inlet valve is opened and pre-weighed powders loaded into hopper are quickly drawn into the liquid stream. To modulate the rate of powder injection, the operator can partially close the inlet valve. Once the first type of powder is all in, the SLIM valve is closed while other powders are loaded into the hopper.

✓ Very minor components may be charged first, so the bulk powders can chase down any particles remaining on the hopper.

✓ The SLIM works best at lower viscosities, so if a thickening agent in the formulation causes a quick and considerable rise in viscosity, that ingredient would normally be added last. In some cases, gums are introduced first to prevent settling of dense powders.

✓ Liquid (soapy) surfactants that render the mixture bubbly must be added last to minimize foaming. These can be added directly into the open tank as the product continues to recirculate.

4. Viscosity, not percent solids, dictates mixer selection.

While the term “high solids” is commonly used within different markets including adhesives, paints and coatings, there are no standards defining minimum or average solids content. (Note: “Solids” can refer to the non-volatile materials or the inorganic components of a mixture.) The tricky thing is that high solids compositions take on a wide range of viscosities. For example, sprayable high solids coatings obtain their low viscosity with the use of liquids with good solvency and low-molecular weight polymers. Hot-melt adhesives that are “100% solids” are, in fact, almost water-like in viscosity during processing at the appropriate temperatures. Meanwhile, a gel containing only 3% solids can be very viscous and unpourable.

Mixer selection is generally dictated by viscosity rather than percent solids. The starting viscosity of the solvent(s), the maximum viscosity reached by the mixture during processing and the final viscosity of the end product (if different from maximum viscosity) are all important considerations.

To accelerate dispersion or dissolution of powders in moderate to high viscosity formulations, elevating shear input is not enough. Dispersers and rotor/stator mixers are often used in combination with an anchor agitator designed for laminar bulk flow. The anchor features “wings” that extend to the tank’s periphery and normally includes scrapers which actually contact the vessel surfaces. Scrapers significantly increase heat transfer efficiency especially in cooling operations. By motivating the exchange of product from different parts of the vessel, the low-speed anchor agitator essentially “feeds” the high speed devices with materials that would otherwise not flow readily.

As viscosity rises to the million centipoise range, even a multi-shaft mixer arrangement will eventually fail to produce sufficient flow. This is because the disperser, rotor/stator and anchor agitator all turn from a fixed axis of rotation. The logical solution is to utilize agitators that move through the batch *regardless of product flow*. This is the forte of planetary mixers where two or more blades rotate on their own axes as they orbit on a common axis. The agitators continually advance into the batch and contact fresh product all the time.

A Triple-Shaft Mixer can handle viscosity peaks up to several hundred thousand cP.

Planetary Dispersers and classic Double Planetary Mixers with rectangular blades are ideal for highly-filled applications up to around 2 million cP.

Helical-style blades extend the Double Planetary Mixer’s viscosity range to as high as 6 million cP.

5. Don’t hesitate to test the waters.

Testing is an essential part of mixer selection, not only in establishing new processes but also in improving a particular existing operation like powder dispersion. Simulation trials can be performed in-house using trial/rental equipment. Running these tests on your production floor or R&D lab is advantageous in that you can perform virtually unlimited mixing trials under normalized process conditions. Immediately after mixing, the finished product can be analyzed according to your standard quality check procedures. Most rental programs allow you to keep the mixer as long as needed and, if justified by the results, convert it to a purchase.

Another option is to test at the manufacturer’s laboratory which lets you to evaluate multiple mixer designs and methods of powder dispersion. You also benefit from the expertise of mixing engineers who can make valuable on-the-fly suggestions. Reputable mixer manufacturers offer free-of-charge testing services. If this is a practical option for you, below are some important items you can do or research in advance:

- ✓ Plan on attending the mixing trial. Some testing laboratories allow unwitnessed simulations but your expertise on the characteristics and properties of your specific product will boost the trial’s chances for success. Plus, you will be able to observe how the mixer is operated, learn useful techniques and identify any potential issues well before the equipment is delivered to your facility.

- ✓ Indicate the batch size or flow rate that you will require on the actual process. To obtain reliable data for scale-up, a good rule of thumb is to test on a mixer no smaller than 10% of the capacity you are looking to eventually purchase.

- ✓ Prepare a control sample for comparison with the trial batches. Check if the manufacturer’s laboratory has analytical equipment that you can use for checking end product quality (particle size analyzer, microscope, Hegman gauge, picnometer, etc.). Specialized tests may have to be done back at your facility so be ready with sample containers or boxes/pails for the finished product, MSDS’s and return shipping information.

- ✓ If possible, pre-weigh all raw materials for each batch. This will save a lot of time and leave more room for testing and discussion, as needed. Send enough raw materials for an extra run or two, just in case.