Ways to Optimize Solid-Liquid Mixing

A White Paper Prepared By

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Ways to Optimize Solid-Liquid Mixing

Abstract

This white paper presents a number of mixing technologies and techniques employed in solid dispersion and dissolution.

The aim of this paper is to provide practical information on the efficient use of specialty mixing equipment for the preparation of low, medium and high viscosity solid-liquid mixtures under low and high shear conditions.

Introduction

Whether mixed into liquid in the form of powders, pellets, granules, flakes, crystals or fibers, solids often represent a processing challenge. Manufacturers generally prefer to deal with only fluids in their mixing operation mainly for convenience. Compared to solids, liquids are easier to handle and mix with other liquid ingredients. For example, within the paper industry, chemicals are commonly shipped in roughly 50% concentrated form. Water is simply added to achieve the dilute solution required for process needs or making of the finished product.

This convenience of course comes at a price. In fact, tremendous savings may be obtained if raw materials are ordered in solid form instead of liquid form. Significant amounts can be saved in shipping costs alone, especially with large volume requirements of slurries or solutions that are mainly water in composition. The key is to have an efficient and repeatable in-house mixing process for dispersing solids into liquid.

Then there are those formulations that require solid-liquid mixing no matter what which manufacturers cannot escape from. Depending on the properties of the solid(s) and the liquid vehicle, the mixing operation can be a straightforward batch or continuous process or it can involve several stages and require different types of mixers. From dispersing fillers into a viscous composite or dissolving solid polymers into solvent to wetting out hydrophobic powders or breaking up agglomerates of pigments, the degree of mixing difficulty varies from one specific application to another.

To structure our discussion in this paper, we use viscosity and shear intensity as the categories within which we enumerate a number of ways to optimize solid-liquid mixing.

Low Viscosity and Low Shear Applications

Solids that readily disperse or dissolve into liquid may be mixed using a variety of low speed agitators such as propellers, pitched-blade turbines, impellers and anchor stirrers, all of which provide relatively gentle agitation. Because they easily wet out, such powders require very low energy input per unit volume. These processes are generally very straightforward and inexpensive. Good mixing is simply based on adequate batch turnover within the vessel. Tank and blade geometry, as well as mixer speed, are the main design parameters.

When mixing substantial amounts of powders into a relatively large batch, manufacturers may consider installing a recirculation line equipped with an inline rotor/stator mixer to accelerate the mix cycle. This makes sense especially if obtaining a narrower particle size distribution is also desirable. The supplemental shear and agitation provided by the inline mixer helps to create a more uniform dispersion while boosting throughput by completing the batch faster.

An additional benefit to the inline mixer is its ability to be used as a pump for transferring the finished product downstream.

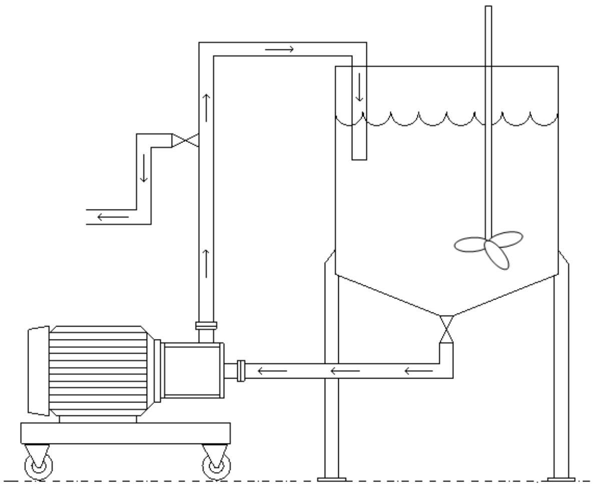


Illustration of an inline rotor/stator mixer connected to the recirculation loop of a gently-stirred tank.

Low Viscosity and High Shear Applications

More challenging solids are mixed into liquid in a variety of ways which could involve a pre-mix stage to combine the raw materials followed by a "polishing" step. If attractive forces between the individual particles are strong enough, wetting them out (i.e., adding the solids into a liquid batch under gentle agitation) only disperses agglomerates of the particles. High shear forces are therefore necessary to break up groups of these agglomerates. How aggressive those shear forces need to be vary from one formulation to another. Some powders, for example, will completely disperse when mixed using a high speed blade such as a saw-tooth disperser or a rotor/stator device. In such cases, the powder wet-out stage and polishing step are accomplished using a single mixer.

Running at tip speeds of around 5,000 ft/min, a saw-tooth disc-style disperser creates vigorous flow within the mix vessel and imparts greater shear compared to other open-blade mixing devices such as propellers or turbines. It generates a vortex into which solids can be added for quick incorporation into the batch.

Saw-tooth disperser

Rotor/stator mixer

A rotor/stator mixer typically features a four-blade rotor running within a close tolerance fixed stator at tip speeds in the range of 3,000-4,000 ft/min. This type of device creates mechanical and hydraulic shear by continuously drawing product components into the rotor and expelling them radially through the openings in the stator. Due to the differential speed and close tolerance between the rotor and stator, this mixer delivers greater shear and faster deagglomeration compared to devices with an open-blade geometry, even those that run at higher tip speeds.

Many solid dispersions, however, require more aggressive mixing than can be accomplished in conventional dispersers and four-blade rotor/stators. Thus, manufacturers use these machines for preparing a pre-mix or intermediate which is then fed into more shear-intense equipment such as a high-pressure homogenizer, colloid mill or media mill. These expensive devices are however commonly associated with a number of issues including low throughput, tendency to clog, time-consuming clean-up and intensive maintenance. Across the process industries today, there is indeed huge room for improvement in the preparation of very fine dispersions.

New developments in rotor/stator technology endeavor to answer this particular need. Manufacturers looking to update their traditional dispersion processes may find any of the following Ultra-High Shear Mixers (UHSM's) to be a suitable and more cost-effective solution.

PreMax Batch Ultra-High Shear Mixer

The Ross PreMax is a top-entering batch Ultra-High Shear Mixer equipped with the patented "Delta" rotor/stator assembly. Turning at 5,000 ft/min, the rotor is specially contoured for high pumping capacity and shear intensity. Product is drawn from above and below the mix chamber then expelled radially through the stator slots at high velocity. This generates upper and lower vortexes allowing for extremely efficient powder additions and rapid turnover rates.

Depending on the formulation, the PreMax can produce results comparable to one or two passes through a media mill. Hence, for an application that currently requires media milling, the PreMax can potentially reduce the number of mill passes or eliminate milling entirely. Raw materials can thus be combined and brought to the final dispersion in a single vessel. One-pot processing in the PreMax eliminates transfer steps and simplifies clean-up, dramatically cutting overall cycle time while reducing cost.

PreMax Batch Ultra-High Shear Mixer
with "Delta" rotor/stator
(US Patent No. 6,000,840).

Series 700 Inline Ultra-High Shear Mixers

Ross Series 700 Mixers are inline devices available in three ultra-high shear rotor/stator designs namely the X-Series, QuadSlot and MegaShear (see next page for detailed descriptions of each design). Running at tip speeds over 11,000 fpm, a Series 700 mixer is capable of far greater flowrates compared to a similarly-sized high pressure homogenizer or colloid mill. It is also easier to clean and disinfect in place. Based on user experiences, the shorter cleaning time equates not only to a faster changeover procedure but also to longer intervals between cleaning cycles (longer production runs).

A comparably-sized inline UHSM costs considerably less than a high pressure homogenizer while being less sensitive to clogging and changes in viscosity. In almost all applications, this technology delivers greater particle size reduction compared to traditional colloid mills.

Ross Series 700 Inline Ultra-High Shear Mixer (production model)

SAMPLE ULTRA-HIGH SHEAR MIXER APPLICATIONS

- Adhesives
- Agrichemicals
- Chemical additives
- Clay dispersions
- Cosmetic creams
- Dressings and condiments
- Electronic and printing inks
- Flavor emulsions
- Food supplements
- Greases and lubricants
- Gum dispersions
- Nanodispersions
- Paints
- Pharmaceutical suspensions
- Pigment dispersions
- Soft polymer disintegration
- Specialty coatings
- Sunscreen lotions
- Syrups and beverages
- Wax emulsions

Rotor/Stators for Series 700 Inline Ultra-High Shear Mixers

X-Series

The X-Series head (US Patent No. 5,632,596) consists of concentric rows of intermeshing teeth. The product enters at the center of the stator and moves outward through radial channels in the rotor/stator teeth. The combination of extremely close tolerances and very high tip speeds (11,300 fpm or higher) subjects the product to intense shear in every pass through the rotor/stator. The gap between adjacent surfaces of the rotor and stator are adjustable from 0.010" to 0.180" for fine-tuning shear levels and flow rates.

QuadSlot

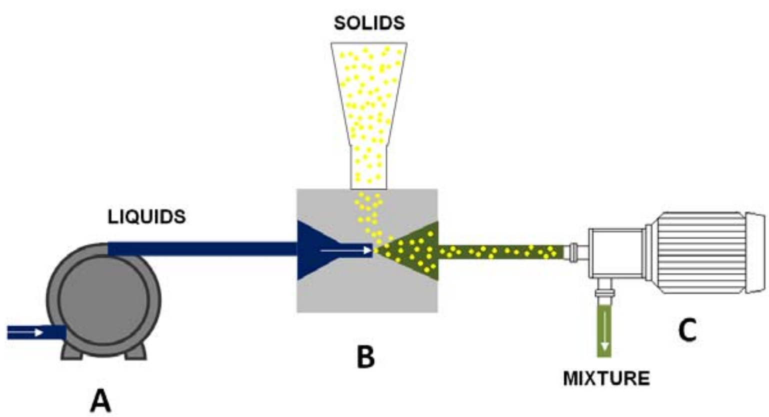
The QuadSlot mixing head is a multi-stage rotor/stator with a fixed clearance. It produces high pumping rates and intense hydraulic shear energy.

MegaShear

The MegaShear head (US Patent No. 6,241,472) is capable of the highest peak shear and throughput levels. It consists of parallel semi-cylindrical grooves in the rotor and stator towards which product is forced by high velocity pumping vanes. Different streams are induced within the grooves and collide at high frequency before exiting the mix chamber.

Sub-surface Powder Injection

Aside from a shear level standpoint, the use of rotor/stators has also evolved in recent years to include the capability for sub-surface powder injection. Earlier versions of this technology operated based on the venturi principle: a pump (A) accelerates liquid into an eductor (B) creating a vacuum; powder fed through an overhead tube is drawn by this vacuum into the eductor where it joins the liquid flow; finally, a rotor/stator device (C) mixes the powder and liquid, and propels the flow downstream.

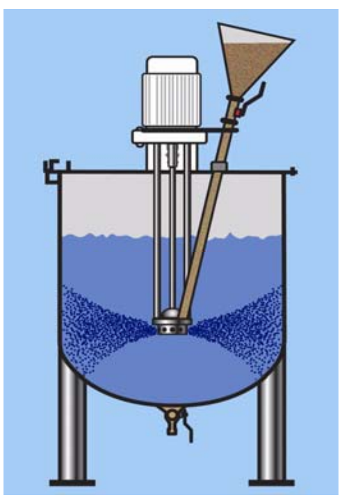


First generation eductor-based powder injection system

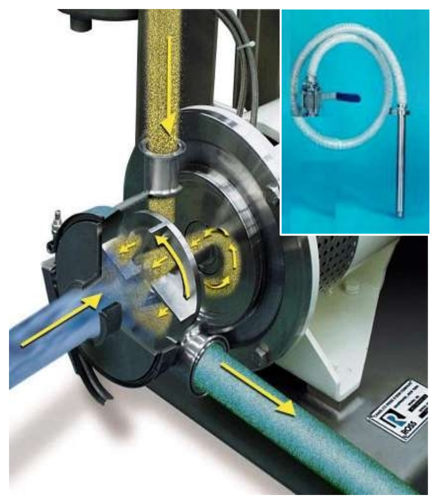
The theory is sound but in practice balancing the performance of the pump, eductor and mixer is often difficult. Downtime is quite high due to routine clogging which happens in many applications. The system is temperamental and requires the attention of an experienced operator. Most set-ups can only handle liquids below 100 cP with low solids concentration. Finally, with three separate devices in series, clean-up and maintenance are intensive.

New generation rotor/stator designs such as the Ross Solids/Liquid Injection Manifold (SLIM) addresses the above limitations by utilizing a specially designed rotor/stator mixer that also executes the functions of both the pump and the eductor. The ported rotor generates an intense vacuum which draws powders right into the high shear zone of the mix chamber, where they are dispersed instantly with the liquid stream. Without the need for an auxiliary pump and eductor, operation is simpler and more straightforward. The SLIM technology simplifies material handling, accelerates dispersion, reduces dusting and improves operator safety.

Using the SLIM, solids are combined with the liquid sub-surface and immediately subjected to intense shear. Solids meet the liquid at precisely the point where flow is most turbulent, preventing agglomerates from forming in the first place. Dispersion is virtually instantaneous and complete. The SLIM works optimally at solid loadings as high as 70% depending on the application and within a wide viscosity range: from water-like to up to 10,000 cP during powder injection. After all the solids are added, product viscosity may continue to climb and a properly-sized SLIM mixer can keep on recirculating the mixture or pump it downstream.



Batch SLIM. In the above mixer configuration, powders are loaded into a hopper. As soon as the rotor reaches operating speed, the hopper valve is opened and solids are quickly drawn into the batch via the vacuum generated by the rotor.



Inline (Continuous) SLIM. The liquid stream (blue) enters the mixer and immediately encounters the powder addition. Drawn into the mixer by a powerful vacuum, the powder (yellow) is injected through the ported rotor directly into the high shear zone. The resulting dispersion (green) is expelled centrifugally through the stator openings and pumped at high velocity.

In lieu of the feed hopper, a "hose & wand" attachment (inset) may be used to draw extremely dusty powders straight from within the bulk bag or container.

Typical Solids Injected through the SLIM

- Acrylic polymers
- Alginates
- Alumina
- Bentonite and kaolin clays
- Calcium carbonate
- Carbomers
- Carbon black
- Carboxymethylcellulose (CMC)
- Carrageenan
- Citric acid
- Dye powders
- Fly ash
- Fumed silica
- Granulated sugar
- Graphite
- Guar
- Gum Arabic
- Hydroxyethylcellulose (HEC)
- Iron oxide and other pigments
- Latex powders
- Magnesium hydroxide
- Metal chlorides
- Metal oxide powders
- Milk
- Pectin
- Polycarbophil
- Potassium sorbate
- Precipitated silica
- Pulp dust
- Silicon powders
- Sodium carbonate (soda ash)
- Sodium gluconate
- Starch
- Sweeteners
- Talc
- Titanium dioxide
- Vanilla powder
- Whey
- Xanthan gum
- Yeast

Medium Viscosity and Low Shear Applications

Flowable solid-liquid mixtures with viscosities above 50,000 cP require mixing blades with a larger diameter and larger surface area to generate sufficient flow. Anchor agitators and bow tie mixers, as well as blades in the shape of ribbons, screw or paddles, belong to this category. The mixing mechanism in any of these mixers is relatively low shear, thus suitable for solids and liquids that readily combine or mixtures requiring high uniformity but not necessarily a fine dispersion. Otherwise, a Multi-Shaft Mixer capable of both low and high speed agitation through the use of two or more different style blades would be more effective. The following section will elaborate on the design advantages of this type of mixer configuration.

Medium Viscosity and High Shear Applications

Thickened solutions and mixtures with high solids loadings often arrive at viscosities that render single-shaft devices inadequate. Furthermore, if the starting liquid vehicle is already viscous, it quickly becomes hard to incorporate solids using a single-shaft mixer, especially if those need sufficient shear to disperse completely.

For such requirements, manufacturers generally rely on multi-agitator mixers which are extremely efficient at turning over viscous batch material within the mix vessel. In this type of mixing system, a low-speed anchor agitator typically complements one or two high speed devices, such as a saw-tooth disperser blade and/or a rotor/stator assembly. Note that a modified rotor/stator may be installed to allow for sub-surface powder injection. Multi-Shaft Mixers are fairly robust and can process formulations that are several hundred thousand centipoise. Batch temperature is easily controlled through use of a jacketed mix vessel and scrapers attached to the anchor agitator.

Aside from the improved capability of Multi-Shaft Mixers over single-shaft devices from a viscosity and heat transfer standpoint, another design advantage is that they are closed systems and can offer benefits in vacuum mixing. When processed under vacuum, certain applications develop higher densities and achieve better tensile properties as a result of improved shearing and contact of the different components. Mixing under vacuum also eliminates unwanted air voids in the finished product. With other formulations, vacuum mixing keeps entrapped oxygen to a minimum, ensuring longer shelf life and improved stability.

There are several techniques that operators follow when mixing under vacuum. One is to load the liquid phase first into the mix vessel and then pull vacuum to release any entrapped air. Running the anchor slowly during vacuum application assists in breaking up air pockets within the batch and helps to control the liquid level, preventing product from rising to the mixer cover. After this step, solids are charged on top of the liquid and full vacuum is reestablished in the vessel before any agitators are turned on. The vacuum environment causes powder agglomerates to explode. After the batch is mixed and returned to atmospheric pressure, liquids fill interstitial spaces between solid particles resulting in a void-free mixture.

Some sample configurations of Ross Multi-Shaft Mixers.

Sample Batch Procedure on a Multi-Shaft Mixer

The following summary of a polymer dispersion process provides a step-by-step look at a typical Multi-Shaft Mixer Application and a good indication of the flexibility possible with this type of mixer.

Specs of 300-gallon Triple-Shaft Mixer

- Three-Wing Anchor (max speed: 30 rpm)
- High Speed Disperser (14" saw-tooth blade, max speed: 1,090 rpm)
- SLIM High Shear Mixer (7" rotor and slotted stator, max speed: 1,800 rpm) equipped with a powder injection manifold.
- All drives are independently controlled and offer a 10:1 variable speed ratio.
- The mixer is equipped with a jacketed vessel as well as Teflon scrapers on the anchor agitator.

Phase 1, Mixing:

150 gals. of base oil is added to the vessel and heated to 350°F. The anchor is run at 25 rpm and as the liquid begins to warm, 300 lbs. of solid rubber polymer is added to the mixer. The disperser is started at 1,090 rpm to initially reduce the solid particles into smaller pieces that could flow through the stator openings of the high shear mixer. Once the polymer has been broken down sufficiently, the rotor/stator is turned on to start the fine grinding process. Soon, the batch reaches the target temperature and at this point, the rotor/stator is doing most of the high shear work; the saw-tooth disperser blade is mainly contributing to batch circulation. Meanwhile, the anchor is constantly moving material from the vessel perimeter to the interior where the two high speed devices pull it into their localized flow patterns.

Phase 2, Powder Injection and Deaeration:

Using the powder injection manifold of the SLIM rotor/stator, the following raw materials are then charged into the batch, sub-surface: 100 lbs. of fumed silica in 5 minutes, 25 lbs. of carbon black in 30 seconds, and 20 gals. of minor liquids in 15 seconds. Product viscosity is around 10,000 cP. To complete this mixing phase, vacuum is pulled to 29.5"Hg and the batch is allowed to deaerate. Because the material's viscosity is strongly temperature-dependent, deaeration is most efficiently done during this step – before the product cools in the following phase.

Phase 3, Cooling and Final Raw Material Addition:
The first step in this stage is to shut down the high shear mixer and reduce the disperser speed to 110 rpm to minimize energy input. The anchor agitator, set at 20 rpm, continues to scrape the vessel walls, hastening heat transfer between the batch material and the cooling liquid running in the jacket. The rotor/stator is not run again in this cycle. This is for two reasons: intense shear is not required anymore; and, with the batch temperature lowered by 180°F, viscosity will rise to 100,000 cP – beyond the range of the rotor/stator, even with the added flow generated by the anchor agitator and disperser blade. When the batch has cooled, 50 lbs. of a fine polymer solid is added to the vessel. The powder injection system cannot be used at this viscosity so the polymer is charged directly to the batch surface and vacuum is reestablished to deaerate the powder. The disperser is then set again to 1,090 rpm to create a vortex for drawing the solid polymer into the batch.

Phase 4, Discharge:
Vacuum is released and the finished product is discharged through a flush tank valve while the anchor turns at 10 rpm. Scrapers clean the vessel sidewalls and bottom as the product level falls. When discharge is complete, the walls and bottom surfaces of the mix can have been scraped clean. After a quick wipe down, the mixer is ready for the next batch.

High Viscosity and Low Shear Applications

As product viscosity climbs to a million centipoise and higher, agitators with a fixed axis of rotation eventually fail to produce adequate flow. At this point, shifting to a planetary-style mixer is recommended. The agitators of a planetary mixer rotate and travel through the mix vessel, passing through every point within the batch and not just along the periphery. Batch components are constantly recombined and physically moved from one part of the vessel to another until a homogenous state is achieved.

Shear-sensitive viscous formulations such as certain gels, creams and pastes that require low speed but thorough blending are successfully produced in Double Planetary Mixers (DPM's). This type of mixer moves material by rotating two identical blades on their own axes as they orbit on a common axis. At the right speed setting, these blades impart a gentle but efficient folding action, an important requirement of sensitive compounds with a tendency for irreversible viscosity loss if exposed to elevated levels of heat or shear.

Dilatant (shear-thickening) materials also benefit from the low speed agitation provided by a Double Planetary Mixer. These mixtures exhibit an increase in apparent viscosity as the rate of shear increases. Typically made up of a high concentration of solids (polymers, metal or oxides) dispersed in liquid, such compounds behave like a fluid under relatively low shear conditions but transition into a more solid-like behavior once greater stress is applied. It is this property which makes mixing dilatant formulations a unique challenge.

The Double Planetary Mixer is very appropriate for processing dilatant materials not only because of its low speed and low shear agitation, but also because its robust design. Some degree of shear is required for proper dispersion of the solid components and DPM can be built with enough torque to accommodate the inevitable shear-thickening effects.

High Viscosity and High Shear Applications

Although the blades in a Double Planetary Mixer turn at relatively low speeds, this does not mean that the mixer cannot implement high shear conditions. In fact, the DPM can finely disperse solid components in high viscosity mixtures up to around 6 million cP when equipped with helical stirrers, such as the Ross High Viscosity "HV" blades (US Patent No. 6,652,137). More traditional blade designs such as the rectangular open paddle and finger blades are ideal for processing mixtures up to approximately 3 million cP.

Rectangular Blades

Finger Blades

High Viscosity Blades
(US Patent No. 6,652,137)

The DPM can handle viscosities as low as 50,000 cP but its ability for high shear dispersive mixing is more efficient at higher viscosity levels. For instance, as the viscosity of a heavy paste approaches 1 or 2 million cP, shear in the batch increases steadily, agglomerates disintegrate and average particle size drops quickly. One processing technique on the DPM is to follow an order of addition that artificially raises the viscosity of a batch to obtain a good dispersion. Once the target level of dispersion is achieved, remaining liquids are added at a suitable rate to bring the mixture down to its final viscosity.

For formulations between 100,000 cP to around 2 million cP, the combination of high speed and low speed mixing may be helpful in creating a fine dispersion. A Planetary Disperser like the Ross PowerMix (PDM) is ideal for this viscosity range because it can impart high shear when the batch is still too low in viscosity to benefit from the DPM's kneading action.

The patented PowerMix design combines the thorough mixing action of a planetary stirrer with the added benefit of a high speed disperser. Both the planetary stirrer and the disperser rotate on their own axes while revolving around a central axis. The planetary stirrer continuously sweeps material near the vessel walls and bottom, carrying them toward the saw-tooth blade. This accelerates dispersion and evenly distributes heat generated by the high speed disperser.

More challenging applications benefit from additionally powerful mixing, like that of two planetary stirrers and two high speed disperser shafts. The Ross Planetary Dual Disperser (PDDM) offers the combined mixing intensity of all four agitators which enables rapid incorporation of large amounts of solids into an already viscous starting liquid vehicle. Energy per unit volume is extremely maximized and particle size reduction is accomplished even faster than in a Planetary Disperser. Shear levels and flow patterns in the PDDM are easily fine-tuned because the stirrers and dispersers are independently driven and controlled.

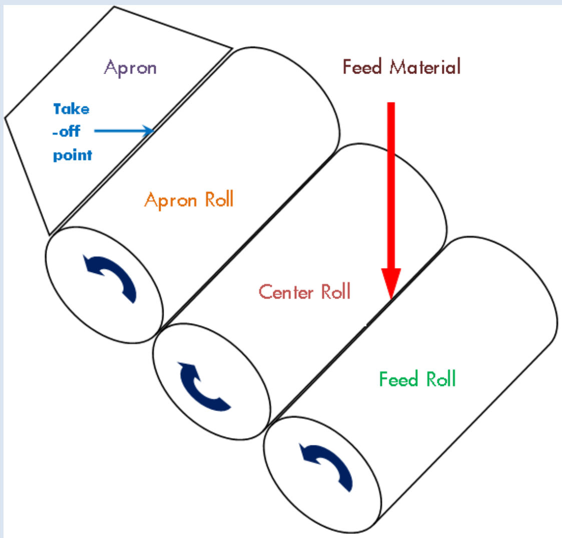
Apart from its robust processing power, the Ross PDDM offers a unique flexibility which is very useful for manufacturers looking to produce multiple products in a single mixer. Because each agitator is removable, the PDDM can be operated as a classic Double Planetary Mixer or as a PowerMix Planetary Disperser.

Ross PowerMix Planetary Disperser (PDM Model) *US Patent No. 4,697,929*

Ross Planetary Dual Disperser (PDDM Model)

When optimizing solid dispersion in your viscous formulation, it is always useful to perform mixing trials comparing two or more mixers and utilizing your own raw materials. This helps to determine not only the most efficient mixer design but also the ideal method and order of addition. If necessary, the level of dispersion achieved in a planetary mixer may be further improved by passing the product through a Three Roll Mill for a final polishing step.

What is a Three Roll Mill?



A Three Roll Mill is composed of three horizontally positioned rolls rotating in opposite directions and at different speeds. The material to be milled is placed between the feed and center rolls, quickly adhering to the rotating surfaces and spreading out evenly. Shear forces between the adjacent rolls break up the agglomerates in the product. When the apron roll is engaged, the material transfers to it while being subjected to additional shear forces at even higher intensities.

The distance between each roll is adjustable and impacts both shear level and throughput. Gap settings in the range of 0.001″ are common. Milled material is scraped from the apron roll by a take-off knife. The cycle can be repeated to improve dispersion quality or until an equilibrium particle size distribution is reached.

Sample Application

At an electronics facility, thick film paste is batched in a Ross Double Planetary Mixer. After the mixing cycle, the paste is weighed and transferred to a Ross Three Roll Mill. A first pass is performed with the feed roll set at 0.002″ and the apron roll at 0.001″. Depending on the formulation, multiple passes may be performed. The rolls are heated or cooled, as desired. After milling, the final product is subjected to a grind gauge reading to make sure it does not exceed 50 microns.