Mixing Technologies for the Production of Low to High Viscosity Adhesives

A WHITE PAPER PREPARED BY
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ROSS

Mixing Technologies for the Production of Low to High Viscosity Adhesive Applications

Introduction

The preparation of almost all adhesives begins and ends with adequate mixing. From the homogenization of adhesive emulsions, to the dissolution of polymers into solvents, or mastication of rubber and let-down of master batches, the type of mixing equipment and method hugely dictate over-all processing efficiency and end-product quality. This whitepaper seeks to provide an overview of effective and updated mixing technologies being implemented across many of today's competitive adhesives manufacturing plants, as well as new equipment designs increasingly being recognized by the industry as potential solutions to prevailing mixing challenges.

High Shear Mixing

Throughout the adhesives industry, the **ROSS line of rotor/stator High Shear Mixers (HSM)** are widely used in the preparation of stable emulsions of adhesives, polymers and resins. Early equipment used to dissolve polymers into solvent was based on low-speed propeller, turbine or rake type agitators in vessels (known as churns). These devices relied heavily on the solvent's softening action on the polymers and predictably yielded very long cycle times. Mixing in a churn for as long 12 – 24 hours was typical. The operator would load the vessel with raw materials, turn on the mixer in the morning and shut it off in the evening or the next day. This problem was exacerbated when the resin was supplied in pellet or slab form, making it difficult to dissolve. Even with the introduction of saw-tooth type high-speed dispersers (HSD), batch times could take several hours just to dissolve the resin.

To accelerate this process, a ROSS High Shear Mixer (HSM) is recommended. Available in batch (vertical) or inline (horizontal) configurations, High Shear Mixers are comprised of a rotor that turns at high speed within a stationary stator. As the blades rotate, materials are continuously drawn into one end of the mixing head and expelled at high velocity through the openings of the stator. The hydraulic shear generated promotes fast mixing and polymer particles are broken down into smaller and smaller pieces which get easier and easier to dissolve. Fillers such as fumed silica are dispersed faster with a high shear mixer compared to lower energy devices. Rotor tip speeds between 3,000 to 4,000 ft/min are typical.

For example, a manufacturer of PVC solvent cements switched to the ROSS rotor/stator (HSM) mixer from a high speed disperser and reduced their cycle time from four hours to just under 45 minutes when PVC pellets are used, and under 30 minutes when the PVC is in powder form.

Another company making insulation adhesives experienced the same >80% reduction in mixing time when they replaced their disperser with a ROSS high shear mixer (HSM). The batch rotor/stator efficiently grinds up pieces of neoprene rubber and assists in a more rapid dissolution into a blend of toluene, hexane and acetone solvents. The use of a closed and jacketed mix vessel prevents the loss of solvents and also allows the operator to control batch temperature.

The ROSS high shear mixer (HSM) is not only useful for dissolving solids into liquids, but also for preparing emulsions. A manufacturer of protective films and tapes used for a variety of surfaces including carpets, windows, marble, and steel was looking for a faster way of blending two liquid components of an adhesive emulsion. They were using a propeller type mixer which they had to run for one hour to ensure a homogenous low-viscosity mixture made of 95% acrylic emulsion and 5% polyisocyanate solution. Target droplet size was 0.30 microns or below. Laboratory tests revealed that a ROSS inline (HSM) rotor/stator mixer was able to achieve the desired product characteristics in just a single pass. This translated into a dramatic improvement in mixing time using a relatively small inline mixer.

In a stable emulsion, the dispersed phase is suspended uniformly as droplets throughout the continuous phase, the two phases immiscible with each other. Big and small drops coexist in the emulsion and their size distribution gives the best description of the emulsion, affecting both stability and viscosity. The length of time in which a considered stable emulsion does not change its aspect is relative.

For emulsification to take place and remain in equilibrium as long as practical, sufficient mixing energy is required. A common generalization is that the higher the shear put into creating the emulsion, the finer the droplets produced, and the more stable the emulsion. However, some emulsions are shear-sensitive such that droplets start to coalesce past a certain level of mixing.

Of course, many other factors apart from shear input affect emulsion stability including individual properties of the dispersed phase and the continuous phase, temperature, presence, and type of surfactant, etc. In other words, the “ideal” average drop size varies from one formulation to another. Accordingly, one cannot generalize what droplet size a particular mixer can achieve. Droplet size is a parameter unique to each specific formulation. Mixer comparison experiments, to be truly meaningful, must be carried out using the same raw materials and their relative percentages.

In a good number of cases, due to the drastic reduction in mixing time, shifting from a low-speed, low-shear mixing system to a ROSS HSM high shear rotor/stator generates significant savings in power consumption which results in a full return of investment within a very short period of time.

In an inline high shear rotor/stator mixer, the greatest extent of droplet size reduction occurs within the first few passes. This phenomenon is true for almost any emulsion. Past this phase of sharp decrease in droplet size, the emulsion hovers at an equilibrium size despite subsequent recirculation. The same trend applies to batch rotor/stator mixing systems although the actual number of product turnovers is not as easy to define. Identifying the number of passes that it takes to achieve the desired or equilibrium droplet size is very useful to avoid over-processing. Over-processing not only unnecessarily consumes time and power but may also heat up the emulsion to the point of causing droplets to recombine or induce an irreversible change in viscosity.

ROSS HSM-100 Series Batch High Shear Mixer with Mobile Lift

ROSS HSM-400 Series Inline High Shear Mixer

High Shear Mixing and Powder Dispersion into Liquid

Powders behave differently when added into liquid, and some require more coaxing to dissolve, hydrate, or disperse completely than others. The “easier” ones need only gentle agitation as provided by low-speed propeller, turbine, or paddle agitators. More challenging powders benefit from higher speed devices such as open disc type blades which generate a powerful vortex into which the powders are added for faster wet-out. When dealing with solids that tend to form tough agglomerates which do not easily break apart, a high shear mixer is often installed to replace or supplement the existing propeller or disc type disperser in the vessel.

Adding hard-to-disperse powders slowly into a small batch of vigorously agitated liquid is not an issue in lab-scale batches. However, in a full-scale production setting, this method of addition is very costly and time-consuming. Moreover, if powders are added too slowly, an uncontrolled viscosity build-up can occur mid-processing thus preventing the rest of the solids to be fully dissolved. On the other hand, charging too fast can cause some powders to clump up. These agglomerates can solvate to form a tough outer layer which prevents complete wetting of the interior particles. These "fisheyes" lead to solution defects such as grainy texture and reduced viscosity. The high shear conditions usually needed to break up these agglomerates may overshear the already hydrated or dispersed particles leading to a permanent loss in viscosity. To correct below-target viscosity, many manufacturers will resort to adding more solids than is really needed and subsequently filtering agglomerates out of the mixture, which not only drives up raw material costs but also wastes power, lowers productivity, and constrains over-all production.

A key development in rotor/stator design is the **ROSS Solids/Liquid Injection Manifold (SLIM) Technology**, a high-speed powder induction system available on ROSS High Shear Mixers. The modified rotor/stator assembly is specially designed to create negative pressure (vacuum) behind the rotor, which can be used as the motive force to inject powdered (or liquid) ingredients directly into the high shear zone.

ROSS Inline High Shear Mixer
with SLIM

ROSS Batch High Shear Mixer
with SLIM

The SLIM is particularly useful in inducting hard-to-disperse powders such as fumed silica, carboxymethyl cellulose (CMC), hydroxyethyl cellulose (HEC) starch, pectin, talc, carbomers, etc. into a liquid phase. These powders are notorious for driving up processing costs in the form of labor and reduced production.

Even with a strong vortex in an open vessel, they resist wetting out and often float on the surface for hours. In the SLIM system, solids are added not to the top of the liquid but right in the mix chamber where they are instantly subjected to intense shear. As solids and liquids are combined and mixed simultaneously, agglomerates are prevented from forming because dispersion is virtually instantaneous.

For years, a developer of automotive adhesives and coatings had been using a high-speed dispersion mill to prepare a high volume adhesive premix. Crumbs of chlorinated rubber, different grades of carbon black and other fillers were dispersed into a solvent solution. Although adequate in attaining the desired grind prior to the sand milling step, the dispersion mill was prone to overheating and required a great deal of maintenance. Looking for a better way to prepare their premixes, this company tried the ROSS HSM Inline SLIM at their facility. Extensive trials confirmed that the SLIM system offered a three-fold advantage to their dispersion mill: lower processing temperature (85°F vs. 140°F), higher solids loading capability (28-33% vs. 8-12%), and faster, dust-less powder induction.

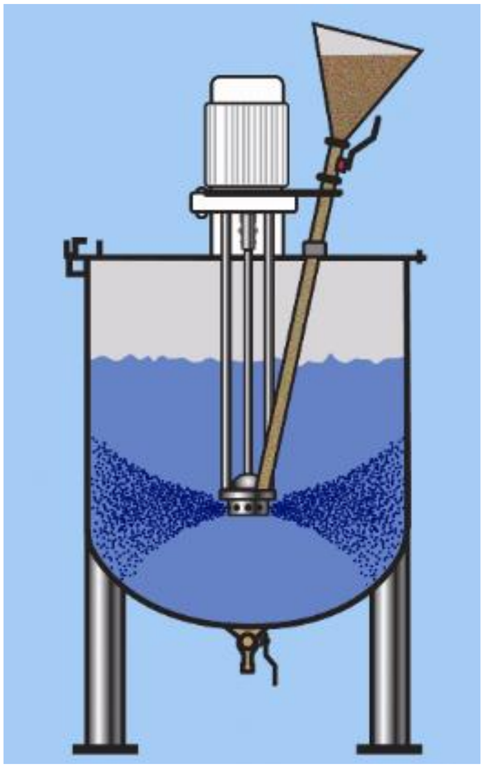
The inline configuration of the SLIM is a superior design compared to earlier venturi or eductor systems. In eductor systems, the process liquid is pumped at high velocity into a venturi chamber and passes into a downstream inline mixer. The combination of the pump, venturi and the pumping action of the mixer creates a vacuum in the venturi chamber. Powder fed through an overhead hopper is drawn by this vacuum into the eductor where it joins the liquid flow. A rotor/stator then mixes the powder and liquid, propelling the flow downstream. While this set-up eliminates the dusting and floating issues of batch systems, it also presents serious limitations. With three separate devices in series, maintenance is intensive. Balancing theperformance of the pump, eductor and mixer is often difficult, and in many applications, downtime is quite high.

But the most serious limitation relates to the inherent operating limitations of the venturi or eductor. Clogging is routine. The system is temperamental and requires a lot of operator experience and attention to operate successfully. Since the feed rate of the eductor relies on thevacuum created by a fast-moving stream, it is also extremely viscosity dependent. As the viscosity of the stream rises, velocity falls and the efficiency of the eductor drops offs steadily until it finally stops.

The ROSS HSM SLIM design is a breakthrough based on one simple idea — eliminate the eductor. In the older powder induction designs, solids are combined with the moving liquid stream in the eductor, and then mixed farther down the line. The distance between the eductor and the mixer is critical. Material that had been combined but not yet mixed intimately could clog the pathway before reaching the rotor/stator mixer where agglomerates could be dispersed.

In addition, clumps produced in the venturi chamber would form that tough outer layer which prevent interior particles from being wetted out.

ROSS Batch HSM SLIM



ROSS Inline HSM SLIM

A producer of pressure sensitive adhesives was using a steam-jacketed mix tank with a center agitator blade and a counter rotating sweep blade to disperse rosin ester resin powders into a surfactant-water solution. This process took 5 to 6 hours to complete. Temperature was closely monitored and kept below 113°F. At higher temperatures, the resin particles would soften and begin to agglomerate into sticky clumps which affected product quality and made cleaning difficult. When this point was reached, cooling was necessary to reverse the agglomeration. Evaluation of a 25HP inline SLIM system with a hopper attachment revealed that resin powders can be inducted under high shear at an impressive rate of around 75lbs/minute. Due to the very short cycle time, the 45% resin mixture is kept well within the 113°F limit and a cooling step is not required.

For more information about ROSS SLIM Technology click here.

Inline Ultra-High Shear Mixing

In some instances, conventional four-bladed rotor/stator mixers are unable to provide the required level of shear for dispersion, emulsification or particle size reduction. In these cases, adhesive manufacturers may turn to high-pressure homogenizers or colloid mills to make their products. Yet, several issues are typically encountered which make the use of homogenizers and mills less than ideal. These issues include low throughput, labor-intensive and time-consuming clean-up procedures, and high maintenance (i.e., seals have relatively short service life due to the extreme operating conditions). In addition, high-pressure homogenizers have a high comparable initial cost.

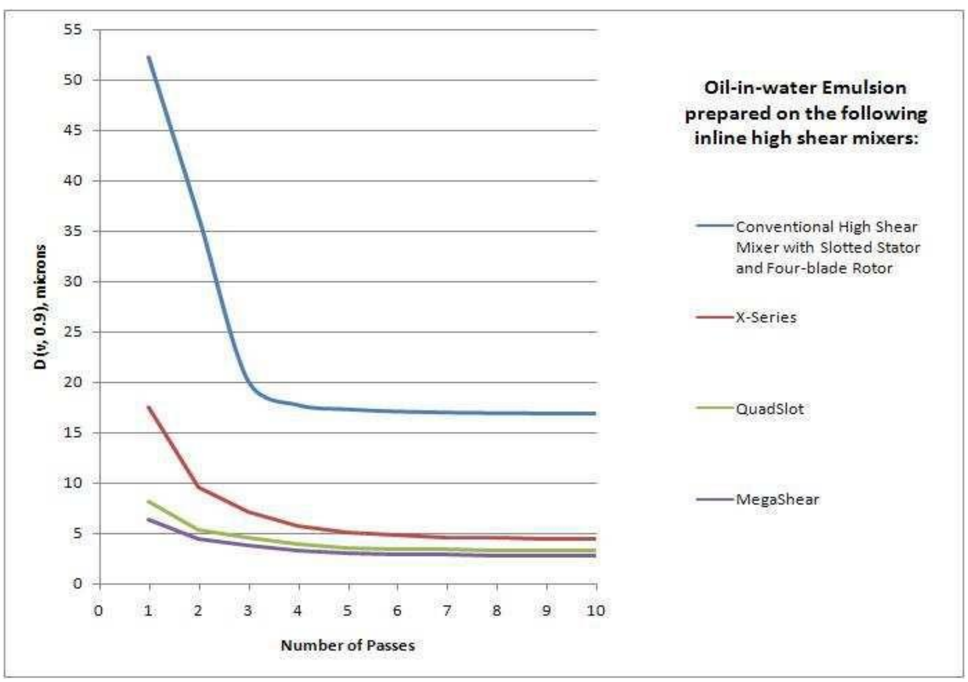
A different and welcomed approach comes in the form of new generation ultra-high shear mixers; an emerging technology being recognized across a wide range of process industries and not just in the field of adhesives.

Ultra-high shear mixers have unique rotor/stator geometries and run at tip speeds as high as 18,000 fpm. The combination of extremely close tolerances, specially designed channels and grooves in the rotor/stator generator and high tip speed, not only produces sub-micron dispersions and emulsions, but also disintegrates solid polymers into extremely small particles in just a single pass.

The Ross MegaShear, X-Series and QuadSlot Ultra-High Shear Mixers represent a quantum leap over the conventional rotor/stator mixer technology. Speeds, rotor/stator gap settings and residence time can be controlled to prevent over-shearing, which can produce a broken emulsion or degrade the polymer.

In the manufacture of adhesive emulsions, ultra-high shear mixers, compared to lower energy devices, produce finer droplet sizes and may allow the use of less surfactant which in turn can reduce the cost of the end product. One problem associated with wetting agents and defoamers is that they can be adsorbed by the polymer particles, thereby altering the coating performance of the emulsion. This often leads to product rework and/or scrap. Thus, by using less surfactants and defoamers, one can save in raw material costs and also produce more consistent batches. Ultra-high shear mixers present adhesive manufacturers this option.

On the right is a graph comparing the equilibrium droplet size distributions of an oil-in- water emulsion prepared on a regular rotor/stator and on the X-Series, QuadSlot and MegaShear ultra-high shear mixers (UHSM's).



The **ROSS 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. Tolerances are extremely close, and the rotor runs at very high tip speeds typically up to 11,300 fpm. This combination subjects the product to intense shear in every pass. The gap between adjacent surfaces of the rotor and stator can be set as close as 0.003" and is adjustable for fine-tuning shear levels and flow rates.

X-Series

The **ROSS QuadSlot** mixing head is a multi-stage rotor/stator with a fixed clearance. This generatorproduces higher pumping rates and requires higher horsepower compared to an X-Series rotor/stator set running at similar speeds.

QuadSlot

The **ROSS MegaShear** head (US Patent No. 6,241,472) operates at the same tip speed as the X-Series and QuadSlot heads but is even more aggressive in terms of 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.

MegaShear

For more information about ROSS High Shear and Ultra High Shear Mixers click here.

Batch Mixing of Viscous Formulations

Generally, the bulk of the solid portion of an adhesive is the bonding agent and the solvent is only a carrier to provide an easy method of application. Therefore, adhesives with a higher solids percentage usually contain more usable adhesive per gallon. Many adhesive slurries, cements and pastes are high-solids, viscous formulations that cannot be processed in a single disperser or rotor/stator mixer. This is when multi-shaft mixers are employed.

ROSS Dual-Shaft (CDA) and Triple Shaft Mixers (VMC) are comprised of two or more independently driven agitators working in tandem. A low-speed anchor compliments one or two stationary high shear devices, such as anopen disc style disperser blade or a high shear mixer rotor/stator assembly. On its own, a disperser blade will produce acceptable flow patterns in batches up to around 50,000 cP: the rotor/stator up to around 10,000-20,000 cP. Hence, for higher viscosities, there is a need for a supplemental agitator to improve bulk flow, deliver material to the high-speed devices and constantly remove product from the vessel walls for better heat transfer.

The most common low speed agitator designs are the two-wing and three-wing anchor. For added efficiency, especially in terms of axial flow, a three-wing anchor can be modified to feature helical flights in between wings.

In combination, stationary high shear devices and an anchor will process formulations that are several hundred thousand centipoise.

ROSS CDA-25 Dual Shaft Mixer

ROSS Sanitary VMC-10 VersaMix

Aside from the extended capability of multi-shaft mixers from a viscosity standpoint, another design advantage is that they are closed systems that offer the benefit of optional vacuum or pressure mixing.

When processed under vacuum, certain adhesives and composites develop higher densities and possess better tensile properties as a result of improved shearing and contact of the different components. With other adhesive products, vacuum mixing keeps entrained oxygen and moisture to the minimum which ensures longer shelf-life and improved stability (nitrogen blanketing is another technique). Mixing under vacuum also gets rid of unwanted voids that agitation under atmospheric conditions can produce. Pulling vacuum while mixing can also eliminate costly downstream de-aeration steps and shaves overall processing time.

It is common for multi-shaft mixers to function as specialized reactors equipped with automated controls and PLC-based recipe systems. For example, in a polymerization reaction, the batch has to be kept constantly homogenous and carefully monitored for temperature, level, pressure, etc. The properties of the end product are directly influenced not just by the purity of raw materials and reaction chemistry but also by the efficiency of mechanical mixing. Undoubtedly, identical formulations produced in different mixing systems can result in dissimilar stability, adhesion performance, and heat resistance.

For more information on ROSS Multi Shaft Mixers click here.

Double Planetary Mixing

As product viscosity continues to build up, a multi-agitator mixing system will eventually fail to produce adequate flow as can be characterized by an anchor carving a path through the batch (instead of pushing product away from the walls and into the center) or by high-temperature zones near the disperser and rotor/stator assemblies. At this point, stationary shaft agitators no longer suffice and a move to a **ROSS Planetary Mixer** is recommended. The agitators of a planetary mixer rotate on their own axis while being revolved around a central axis in the vessel. This motion allows the blades to pass through every point within the batch, not just along the periphery.

The ROSS Double Planetary Mixer (DPM) can be equipped with rectangular stirrer blades, finger blades or High Viscosity "HV" blades (US Patent No. 6,652,137). The HV blade design generates a vertical mixing action owing to its precisely angled helical contour. This sweeping curve firmly pushes the batch material forward and downward, a unique mixing action that solves the 'climbing' problem commonly experienced when processing highly filled materials. In addition, the HV blades do not have a lower crossbar so they can be cleanly lifted out of a very viscous batch and can pierce right through it just as easily.

ROSS Double Planetary Mixer (DPM)
with High Viscosity "HV" Blades

ROSS Double Planetary Mixer (DPM)
with Rectangular Blades

The order of raw material addition in a double planetary mixer is a crucial parameter. One method is to start with all or majority of the solids (resins, fillers, antioxidants, curing agents and other additives) and gradually add liquids (oils and tackifiers). Unless there are waxes or resins that need to be melted, it is recommended to artificially raise the viscosity by withholding some of the liquids. The higher the product viscosity during mixing, the greater the shear that the planetary blades can impart into it.

There are adhesive products that require a two-step approach to assure proper dispersion. For instance, after blending all ingredients in a double planetary mixer, the entire batch is transferred to a single shaft, high-speed disperser to provide the extra shear needed for completion. This cumbersome, two-step process is highly labor intensive and time consuming. To improve production efficiency, manufacturers can utilize a hybrid planetary mixer that combines the traditional thorough mixing action of a planetary mixer with the added benefit of a high-speed disperser. Both the planetary blade and the high- speed disperser rotate on their own axes while revolving around a central axis.

The planetary blade orbits through the mix can in a circular manner, continuously sweeping the vessel walls, as well as the vessel bottom, and carrying material toward the high- speed disperser. The planetary blade also insures that any heat created by the disperser is evenly distributed throughout the mix. Variable speed allows precise control of shear rates to minimize the degradation of any shear-sensitive components.

Like multi-shaft mixers, double planetary and hybrid planetary disperser mixers used in the production of adhesives also benefit from vacuum processing capabilities for the same reasons: to minimize exposure to oxygen, remove unwanted voids and improve dispersion.

- **Easier cleanup.** A vertical mixer design has no shaft seals, bearings, packing glands or stuffing boxes submerged in the product zone. In addition, the agitators are raised and lowered in/out of the mix can by a hydraulic lift. This allows easy access for cleaning between batches. Mix cans are interchangeable for designation to a particular formulation and/or color. There is no concern for cross contamination from batch to batch.
- **Less required floor space.** Footprint of the double planetary mixer is considerably less than that of a double arm/ sigma blade mixer.
- **Everyday energy savings.** Since the double planetary mixer uses less motor horsepower to operate, everyday energy/operating costs will be less. This can be significant over time.
- **Semi-continuous operation.** With the use of extra mix cans, the double planetary mixer can produce material in a semi-continuous basis: one can is being charged while other cans in the loop are being mixed, QC'd, discharged, and cleaned.

High Speed Planetary Mixing

Some highly filled and highly viscous formulations benefit from the **ROSS PowerMix (PDM),** a hybrid planetary mixer which combines the traditional thorough mixing action of a planetary mixer with the added advantage of a high speed disperser. Both the planetary blade and the high speed disperser rotate on their own axes while revolving around a central axis. The planetary blade orbits through the mix can continuously sweeping the vessel walls, as well as the vessel bottom, and carrying material toward the high speed disperser. The close tolerance sweeping action of the planetary blade also ensures that the heat which can be created by the disperser blade is evenly distributed throughout the batch. Variable speed allows precise control of shear rates to minimize the degradation of any shear-sensitive components.

ROSS PDM-300-gallon PowerMix


ROSS Planetary Dual Dispersers PDDM-40 gallon
with multiple mix cans and Discharge Systems

For more information on ROSS Double Planetary Mixers click here.

Value of Testing

A manufacturer of corrosion-resistant, trowellable adhesives had developed a new product line of two-component adhesives. At that time, six different mixes were being produced in laboratory scale and the production plant intended for the new line was just being built. A large order came in and engineers had to scramble to get the production equipment.

The adhesive products were relatively viscous (700,000 to 1,500,000 cps) and difficult to handle. Attempts to mix it properly in the plant's existing sigma blade mixer were not successful – the product had too many voids and there was evidence that the reinforcing fibers were being degraded.

Proof-of-concept trial runs confirmed that a double planetary mixer was appropriate for the application. The adhesive company rented a production-scale unit for further evaluation at their plant. Burnout tests and visual examination showed that the planetary mixing action did not degrade the reinforcing fibers in the mixture. The resulting product possessed the required high strength and exhibited maximum tensile properties.

The mixer performed very well and the plant decided to keep it, as well as purchase two extra mix cans to accommodate the mixing of multiple components and multiple batches. Once a mix is finished in one vessel, it is taken to the packaging area for emptying while another mix can is positioned under the double planetary mixer. During production, the plant tries to reserve a can for each recipe. As long as the components are not being changed or left in the vessel longer than specifications permit, clean-up can be avoided. The planetary blades are easily cleaned by lifting the mixing head and wiping them with the appropriate solvent.

Conclusion

The success of modern adhesive chemistries can only go so far as our capability to produce them cost-effectively. It is essential for process engineers to be updated on the different mixing technologies that are being improved or made available. Many of these mixers' uses and functions overlap such that certain applications can actually be successfully produced by two or more types of mixing systems. For example, many types of epoxy-based adhesives, contact cements, hot-melts and pressure-sensitive adhesives can be made in heavy-duty multi-shaft, double planetary and hybrid planetary mixers. In these cases, economics rules out the more costly initial investments but must also take into account the difference in efficiencies, i.e., how much faster is one mixer able to make a batch compared to another system or how much less power is required?

A visit to a mixer manufacturer's test center often helps make the selection process easier. Be sure to test a variety of equipment and techniques using your own raw materials, simulating conditions as close to your actual process as possible. Quantitative test results provide the best assurance that you have chosen the best mixing system for your particular adhesive product.

To learn more about ROSS Mixers visit mixers.com.