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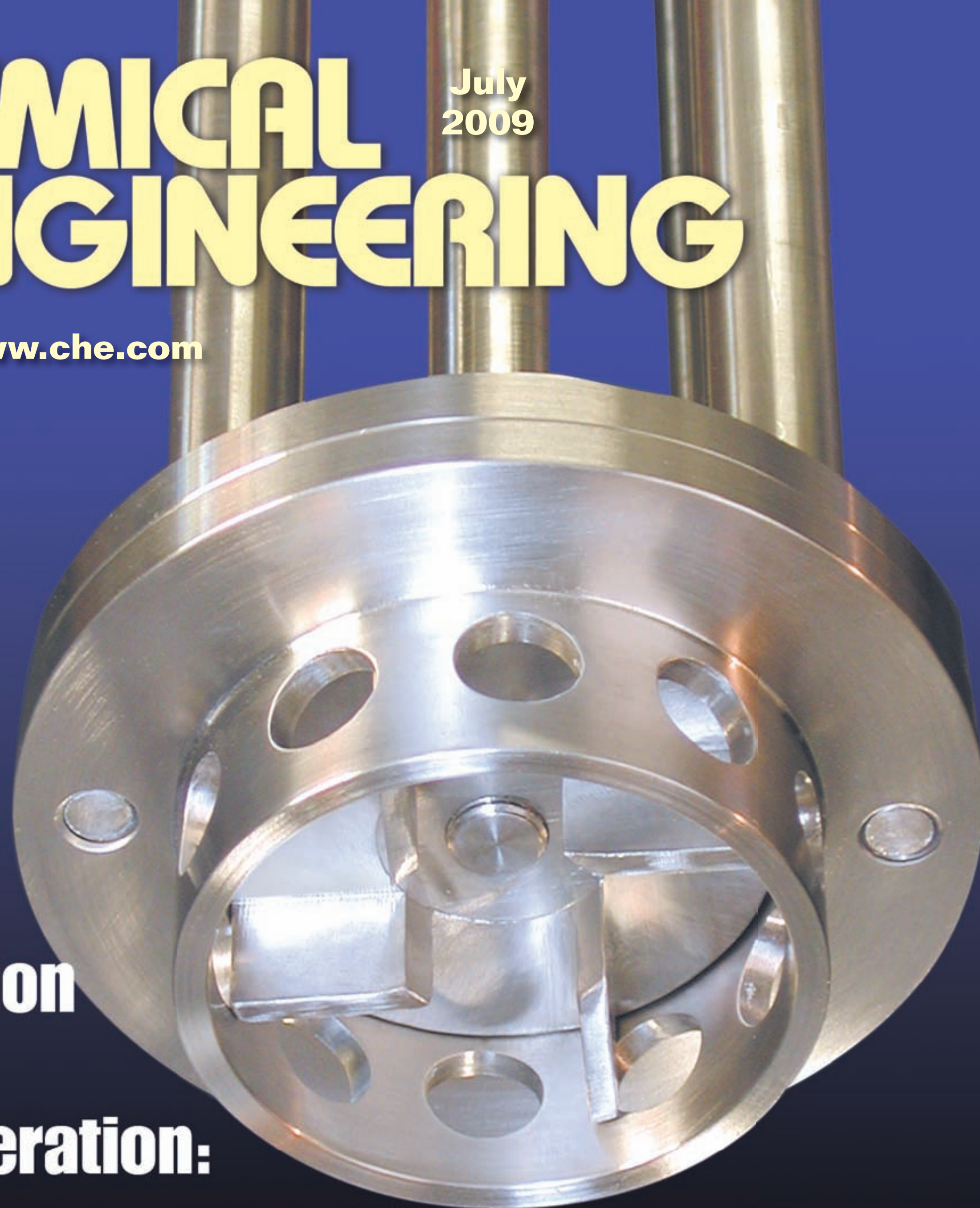

Dispersion Without Agglomeration:

Keep it Clean!

Access Intelligence

Cover Story

Disperse Difficult Solids

Recent advances in mixing technology offer increased efficiency in dispersing powdered additives into liquids for both low- and high-viscosity applications

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FIGURE 1. In a high shear rotor/stator mixer, a rotor turns at high speed within a fixed stator. As the blades pass each opening in the stator, the mixer applies intense shear to the liquid material, which is rapidly accelerated and ejected radially through the stator

It's easy to understand why R&D engineers love performance-enhancing additives like fumed silica, carbomers, cellulose gum, alginates and bentonite clay. These all-purpose ingredients offer incredible versatility for a multitude of products from cosmetics to ketchup, wallpaper paste and the thermal grease used to bond a heat sink to a microprocessor. They can serve as thickeners and fillers. They can impart rheological properties such as thixotropy or pseudoplasticity. They can bind moisture or promote the free flow of solids. They can correct the mouthfeel of an artificially sweetened drink or improve the tear strength of silicone rubber.

Despite the immense value and universal appeal of these additives for product designers, process engineers facing the day-to-day reality of full-scale production face a unique set of challenges. Dispersing these powdered additives into a liquid is one of the most formidable challenges in the chemical process industries (CPI). Although most can be dispersed fairly easily in a common laboratory mixer, when scaled-up for batch, semi-continuous or continuous production, it's much more difficult, time-consuming and costly.

Only a few years ago, in a less intensely competitive business environment, the long mix cycles devoted to dispersing these additives did not receive much attention. The fact that inefficient mixing often led to under-performance of additives, and therefore excessive loading to compensate, was also overlooked. Today, competitive pressures have amplified the importance of every possible improvement in process efficiency — especially those that might yield a significant competitive advantage.

Because of the ubiquitous use of "hard-to-disperse" additives (Table 1) and the inefficiency of the old-fashioned mixing techniques generally used to disperse them, modern mixing techniques present an extraordinary opportunity for manufacturers throughout the CPI. Recent advances in mixing technology enable dramatic gains in process-line efficiency and end-product quality.

A recipe for "fish eyes"

The mechanisms by which thickeners and other modifiers operate vary considerably. However, when they are added to an open vessel with a propeller generating a vortex, the results are usually the same: many hours of mixing, an imperfect dispersion, and often an unsafe plant environment.

In a traditional batch-mixing process, lightweight powders often float persistently on top of the liquid batch. A variety of factors may contribute to this familiar and frustrating sight, including the material's low surface energy, low molecular weight and hydrophobic properties. The material simply drifts on the surface and resists wetting, even when subjected to vigorous agitation.

Over a period of hours, a low-shear, top-entering agitator will gradually coax these materials to submerge into the batch. However, most will readily hydrate to form clumps with a tough outer layer that inhibits dispersion of the particles within. Especially when using low-shear mixing devices, such as turbines and propellers, the dispersion can take many hours to complete. Even in the best-case scenario, this process produces a dispersion of reasonable quality, but only after many hours of processing. All too often, the final mix includes a variety of solution defects, such as a grainy texture, viscosity below target levels and insoluble particles that resemble "fish eyes."

The cost of this imperfect dispersion can be measured in numerous ways.

TABLE 1. MATERIALS APPROPRIATE FOR HIGH SPEED INDUCTION

Material	Typical Applications
Alginates	Paper and textiles, beverages and soups, cosmetics, dental and prosthetic molds
Alumina	Coatings, ceramics
Aluminum Isopropoxide	Lubricating greases
Bentonite clay	Drilling mud, coatings, cement, adhesives, ceramic bodies and glazes, cat litter and rocket nozzles
Boric acid	Specialty lubricants
Calcium carbonate	Building materials, road-building materials, drilling fluids, latex gloves, adhesives and sealants, decorative fillers, ceramic glazes
Carbon black	Adhesives, inks, coatings
Carbomers	Pharmaceuticals, cosmetics, personal care products
Cellulose gum / Carboxymethylcellulose (CMC)	Adhesives, ceramics, coatings, detergents, mining, paper products, textiles, pharmaceuticals, food, cosmetics, personal care products
Fumed Silica	Defoamers, coatings, pharmaceutical gels, cosmetics, personal care products
Hydroxyethyl Cellulose	Coatings, drug capsules, dental gels
Milk Powder	Food
Rosin Ester Resin	Water-based adhesives, coatings
Starch	Food, paper, adhesives
Sugar	Food and beverages
Talc	Pharmaceuticals, adhesives, cosmetics, ceramics
Titanium Dioxide	Textile chemical, inks, coatings, food coloring
Xanthan Gum & Sodium Cyanurate Powders	Swimming pool water stabilizer

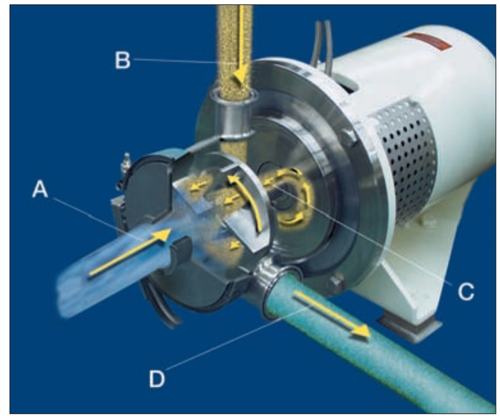

FIGURE 2. The liquid stream (A) enters the mixer and immediately encounters the powder addition. Drawn into the mixer by a powerful vacuum, the powder (B) is injected through the ported rotor directly into the high shear zone (C), where it is subjected to intense hydraulic shear. With particles instantly dispersed, the resulting dispersion is ejected from the mixer (D)

Even if the product is deemed adequate to proceed to downstream processing, these defects usually reduce the efficacy of the additive. This in turn requires more solids to be added in order to generate the desired properties, which drives up the cost of raw materials. Every hour wasted on unnecessary mixing also wastes power, lowers productivity and constrains overall production.

There are also indirect costs that can be traced to inefficient mixing of these additives. For example, in a batch-mixing environment, fluffy powders like fumed silica, carbon black, and many other pigments and flavorings are notorious for "dusting" in the plant. When they are poured into the open vessel, a cloud of airborne particles immediately swirls into the air. This can require a great deal of labor to clean up. It can also elevate the risk of contamination and expose workers to significant safety hazards.

Batch high-shear mixing

A switch to a high-shear rotor/stator mixer is the first essential step toward improving the dispersion of difficult solids. In its simplest form, this mixer consists of a rotor that turns at high speed within a stationary stator (Figure 1). Tolerances are close (0.010–0.015 in. typically), and as the blades of the rotor pass each opening in the stator, they apply intense shear.

In a batch configuration, the portable rotor/stator generator is suspended in the vessel, slightly off-center. Material is expelled at high speed through the stator and into the surrounding mix, which applies hydraulic shear to surrounding material and stimulates vigorous flow. As fast as material is expelled, more material is drawn into the rotor/stator generator from below, which promotes continuous flow, a strong vortex beneath the mixer, and thorough mixing.

For dispersing troublesome powders, the traditional rotor/stator mixer is far more efficient than a low-shear propeller or turbine, but in a batch configuration, it still presents significant limitations:

- Once powders are wetted out, they are dispersed readily. But first, they must be drawn into the mix by the vortex created on the surface. Materials that float or "raft" persistently resist even a vigorous vortex.
- Exposure to intense shear is not sufficiently controlled for all the material in the batch. While the operator waits for the remaining powder on the surface to wet out, solids already hydrated are subjected to more passes through the high shear zone. For some materials, such as synthetic carbomers, this overshearing can result in a permanent decrease in viscosity.
- As batch size increases, the size of the rotor/stator mixer required to generate adequate flow goes up, too. As the mixer size increases, power consumption rises and portability eventually becomes impractical. Mixers exceeding 10 h.p. are generally installed in a permanent, fixed-tank configuration.

Inline high-shear mixing

Inline rotor/stator units provide a high-shear mixing process that is closed and far more controlled than batch units. A liquid stream enters the mixer (Figure 2), and it is immediately subjected to intense shear in the rotor/stator generator. It may be mixed with a powder (or another liquid) in the high shear zone, where the addition is immediately dispersed with highly predictable results.

In an inline, high-shear rotor/stator mixer, the point at which powdered ingredients are added to the stream is a critical factor in determining maximum effectiveness of the device. Early design concepts first combined powdered ingredients with the liquid stream using an eductor. Note that in this scenario, the solids and liquids are first combined; then they travel downstream to the rotor/stator generator, where mechanical shear is applied. During the transit from the point of simple combination to high-shear mixing in the rotor/stator generator — even if it is a distance of just inches — agglomerates are likely to form, which makes the device highly vulnerable to clogging or fish-eye formation.

The rotor/stator generator easily breaks agglomerates apart, but only if the device does not clog between the eductor and the rotor/stator. This realization led to the next major advance in rotor/stator design: mixers that combine ingredients and subject them to high shear simultaneously.

Inline powder dispersion

The many ancillary benefits of an inline, high-shear rotor/stator mixer have been recognized for years, but they were mainly considered little more than a welcome bonus. The primary function of this mixer has always been high-speed dispersion, emulsification and suspension with the application of high shear. The fact that a rotor/stator generator behaves like a centrifugal pump, for example, merely adds convenience and value to the unit. In fact, these mixers often provide sufficient pumping capacity to eliminate the need for an auxiliary pump to move product downstream. These mixers also have the benefit of enabling the easy introduction of powder and liquid additions.

During the first 50 years of rotor/stator mixer design, development continued to focus mainly on the application of intense shear. In the last surge in development — since the early 1990s — multi-stage rotor/stator generators became quite sophisticated, and their ability to create sub-micron emulsions and dispersions improved dramatically. Then, about 10 years ago, mixer manufacturers began to recognize the value of driving rotor/stator design toward even higher levels of shear while also focusing on the optimization of rotor/stator design for the injection of solids into a liquid stream.

Note the path of solids in Figure 2 as they are sucked into the mixer by a powerful vacuum generated by a specially modified rotor and stator. Once they have entered the mixer, they are re-routed through the ported rotor into the interior of the rotor/stator generator. There, the solid ingredient meets the liquid stream for the first time, and together they are immediately subjected to intense shear forces. The dispersion is then ejected centrifugally through the mixer outlet with sufficient force to eliminate the need for a downstream pump in many applications.

With simultaneous combination and high-shear mixing, agglomerates have no chance to form. Agglomerates are sheared and injected into a high-quality dispersion virtually instantly. By reducing the risk of clogging, this design concept has removed a significant constraint on production.

Direct injection for batch mixers

The principle of simultaneous combination and high-shear rotor/stator mixing can also be applied to batch rotor/stator mixers. While the rotor/stator generator is running beneath the surface of the batch, a feed tube allows powdered ingredients to be added from above. As in the inline mixer, the solids are drawn down the tube by a vacuum created by the rotor/stator generator, remaining dry until the moment they enter the high-shear zone and are simultaneously combined with the liquid batch and dispersed.

While this configuration does not offer the close process control of the inline system, it greatly improves the performance of the batch mixer and accelerates the dispersion of solids that would otherwise float on the surface and drive up processing costs.

FIGURE 3. Equipped with helical blades, the double planetary mixer's operating viscosity range is extended from approximately 2 million cP to at least 8 million cP. The mixer's ability to disperse powders rapidly is also dramatically improved. As the helical blades advance, they continuously push powders forward, down and inward

Rotor/stator powder injection

For most veteran process engineers, appreciating the production gain possible with rotor/stator powder injection requires "out of the box" thinking. The scale, in many cases, is quite large, with cuts in the mix cycle ranging from 50% to as much as 98%.

Certain variables are of high importance in their effects on the process line. Viscosity, for example, is a key driver in optimizing the configuration of the powder injection equipment. Depending on the peak viscosity encountered during the mix cycle, performance of the device may be improved with additional agitators that stimulate flow and homogeneous mixing throughout the vessel. Other important process variables include the shear-sensitivity of the solids added to the batch as well as other ingredients already present. Rotor/stator mixers can be modified to inject shear-sensitive powders like carbomers while minimizing the risk of damaging the polymers.

New rules for rising viscosity

The high-speed, powder-injection equipment discussed thus far is ideal for low-viscosity mixes. In a batch configuration, as viscosity rises above approximately 20,000 cP, however, supplemental agitation from a multi-shaft mixer may be required. A slow-speed anchor agitator generates additional flow needed to move material from the vessel walls and bottom toward the interior of the batch and "feed" the high-shear rotor/stator device. Even with this addition, the mechanism remains essentially the same: the injection device sucks flowable powder and liquid into the rotor/stator generator, where it applies intense shear and drives the powder into dispersion. This process works well for products that flow with or without additional agitation, but materials that do not flow easily require a completely different strategy. Instead of drawing free-flowing material to the mixing head, we must literally bring the mixer's agitators to the non-flowing batch material.

Many products — from structural adhesives to aerospace composites and fuel-cell-electrode pastes — require dispersion at the high end of the viscosity range, for which the double planetary mixer is most often chosen. Compared to multi-agitator mixers for mid-range viscosities, this mixer is distinguished in that its agitators are not stationary. Each of the two rectangular planetary blades turns on its own axis as they orbit the vessel on a common axis. While continuously moving material from the vessel walls and bottom toward the interior of the batch, the blades travel through the vessel and physically contact every point in the batch within only a few revolutions.

The drive components and agitators in a double planetary mixer are engineered to apply immense power and move material that is often so dense you can easily stand on it, yet the mixer was limited for decades to materials of about 2-million cP, or even less when working with sticky materials like silicone sealants that are inclined to "climb" the blades during the mix cycle and collect in the upper region of the process container.

Helical blades extend capacity

The most dramatic recent advance in planetary mixing is the development of helical planetary agitators. Unlike the vertically oriented agitators used for decades, these precisely-angled, helical agitators slope gracefully. As they travel through the batch, they continuously force material forward, down and inward (Figure 3). With close tolerances, they also remove material efficiently from the vessel sidewalls. The result is that powder additions, such as carbon black and fumed silica, are quickly wetted out and thoroughly dispersed, and 'climbing' is eliminated.

Compared to traditional agitators, the slope of the helical blades greatly

OBSERVED INDUCTION-RATE TABLE & SUPPORTING DISCUSSION

A key step in applying high-speed powder-induction technology to any application is sizing the mixer to match target production rates, operating budget, and the flow characteristics of the materials being dispersed. This table reports maximum induction rates observed for a variety of materials, for both inline and batch injection systems of various sizes. In general, larger mixers — operating with greater horsepower and larger rotor/stator generators — will apply more energy and produce higher powder-injection rates and greater production.

For a mixer of any particular size and configuration (batch or inline), variation in injection rates is due to a number of factors, including material density, particle size and shape, electrical charge and the presence of moisture. Injection rates for any mixer size and configuration, as well as scaleup performance, are highly predictable. ■

Rotor/stator mixer size & batch/in-line configuration		Sample materials & maximum induction rates by rotor/stator mixer size (lb/min)							
h.p.	Rotor diameter (in.)	Alumina	Calcium carbonate	Carboxy-methyl cellulose (CMC)	Carbomers	Fumed silica	Starch	Sugar	Titanium dioxide
Inline rotor/stator powder induction mixers									
30	7.0	500	590	340	280	110	450	700	560
25	3.5	90	105	60	50	20	80	125	100
15	2.5	45	53	30	25	10	40	63	50
Batch rotor/stator powder induction mixers									
50	10.5	270	315	180	150	60	240	375	300
25	4.5	158	183	105	88	35	140	219	175
5	3.5	68	79	45	38	15	60	94	75

reduces the drag induced by the motion of the agitators through the batch. The absence of a horizontal crossbar also contributes to a reduction in drag (though the principal benefit of eliminating the crossbar is the greater ease with which the agitators may be lifted out of a viscous, sticky batch). Also unlike vertical rectangular blades, the sloped helical blades pass one another with a slicing motion in the vessel. With tolerances very close, this prevents the sudden spike in power that typically occurs when the vertical flights of rectangular blades pass one another.By reducing drag and suppressing this power spike, the working viscosity of a planetary mixer equipped with helical blades is extended from 2 million cP to 8 million cP or higher. This offers manufacturers some surprising new possibilities for fast dispersion needed to produce both high-viscosity and low-viscosity products.

Dispersions for high viscosity

Compared to the high speed devices discussed earlier, the rotational and orbital motion of double planetary agitators is plodding. This slow-motion contact with mix materials does not mean that the double planetary is necessarily a low-shear mixer. Instead, with subtle manipulation of agitator speed and batch viscosity, it can be either a low-shear or high-shear mixing device.

With close clearances between the blades and between each blade and the vessel wall, shear increases quickly as agitator speeds increase. At low speeds, the mixer gently disperses such shear-sensitive materials as micro-spheres to produce syntactic foams and lightweight composites. At faster speeds, it quickly crushes agglomerates and disperses non-shear-sensitive materials such as carbon, alumina, fumed silica, and PTFE to produce battery and fuel cell components.

...and for low viscosity products

Keeping speed constant, shear can also be elevated and dispersion accelerated simply by increasing the viscosity of the batch. In some cases, this may even provide a superior approach to mixing a low-viscosity product.

A case in point is a high-temperature polymeric insulating material used for electrical applications in aerospace. The material consists of micronized calcium carbonate dispersed in resin. The final viscosity of the product is only about 500 cP, but a test in a multi-agitator mixer equipped with a slow-speed anchor and high-speed injection equipment resulted in a final particle-size distribution that was too wide.

Further tests revealed that the process required high-viscosity mixing in a double planetary mixer. The viscosity was artificially raised to approximately 5 million cP by withholding a significant amount of solvent at the beginning of the mixing cycle. At this initial level of viscosity, the friction induced by the planetary blades within the batch generated intense shear, which quickly dispersed all agglomerates of polymer particles and produced a narrow, sub-micron particle size distribution in only 30 minutes. Afterward, the mixed material was let down quite slowly to avoid over-lubricating the product and creating a new generation of agglomerates. Overall, the batch duration was approximately 60 minutes.

Hybrid dispersion strategies

The drive to explore the crossover possibilities between high-viscosity and low-viscosity mixers for dispersion has led to a variety of additional strategies for mixing. One such design strategy produced the hybrid mixer concept that combines a single planetary blade and a high-speed disperser. As in a double planetary mixer, the two agitators orbit the batch on a common axis while each turns on its own axis. This design is extremely versatile, but it is especially suitable for high-viscosity materials that are heat-sensitive but not shear-sensitive. With the high-shear device moving through the batch and the planetary blade continuously feeding material to it, the mixer is quite effective at dispersing heat generated by the high-speed disperser. This lowers the risk of creating a localized buildup of heat that might damage sensitive ingredients. Another approach to "crossover" mixing involves mixing an initial stage in a low- to mid-viscosity mixer, then finishing the process in a high-viscosity mixer. ■

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