HIGH-SHEAR MIXING
Don't Fall Victim to Common Misconceptions

During the scaleup of rotor/stator mixers, beautiful lab results often turn ugly

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The high-shear rotor/stator mixer (HSM), once relegated to a relatively narrow niche of mixing applications, has become a mainstay in many applications in the chemical process industries (CPI). The ability to apply intense shear and shorten mixing cycles gives these mixers broad appeal for applications that require immiscible fluids to be formulated into emulsions, or agglomerated powders to be dispersed into a liquid medium.[1]

Especially during the last decade, the emergence of new variations on the original rotor/stator mixer concept has extended the HSM's usefulness to more diverse applications. For instance, conventional HSMs, in both top-entering batch configurations and inline versions, are widely used today for high-intensity mixing, dispersion, disintegration, emulsification and homogenization. Applications range from dispersions involving gums, pigments, fumed silica, calcium carbonate and active drugs, to emulsions such as cosmetic creams, lotions, and flavors. (The fundamentals of rotor/stator operation are reviewed in the box, p. 47).

However, despite the growing popularity of HSMs in many industries, they are still widely misunderstood. Industry-based and university researchers have focused mainly on working out the dynamics of conventional low-shear mixing technologies, such as axial- and radial-flow turbines. With only a few notable exceptions, high-shear mixing has been largely overlooked in terms of fundamental research to unlock its mysteries and help users to better predict mixing outcomes, particularly during scaleup.

Since the body of literature available for predictive engineering related to rotor/stator mixing is extremely thin, the application of HSMs is often approached empirically — with heavy emphasis on application-specific testing and development by individual manufacturers in the process industries. A few users have invested heavily and achieved impressive success with HSMs in narrowly defined applications such as ones involving emulsion polymers and pigment dispersions. Others have been less successful on their own. Most prospective users of HSMs rely on the recommendation of mixer manufacturers, who often keep their proprietary application guidelines a closely guarded secret.

The result of this lack of publicly

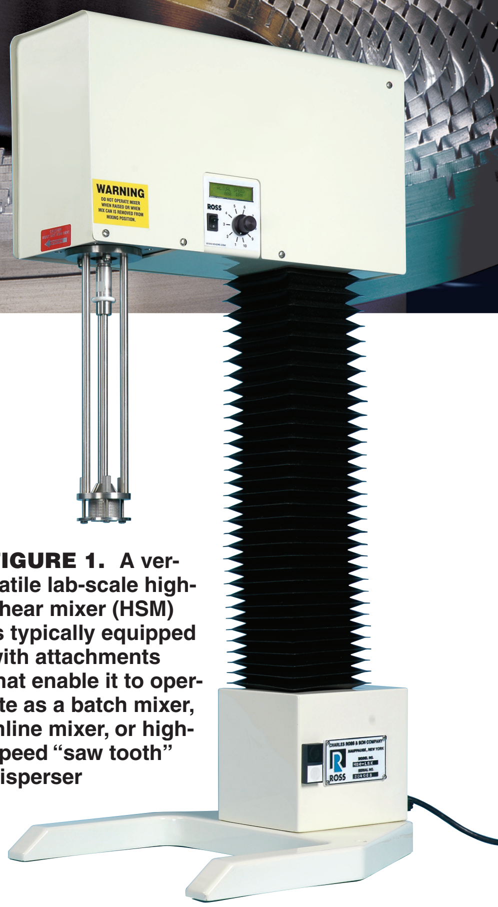

FIGURE 1. A versatile lab-scale high-shear mixer (HSM) is typically equipped with attachments that enable it to operate as a batch mixer, inline mixer, or high-speed "saw tooth" disperser

1 For more on high-shear mixing, see Taking high-shear mixing to the next level, *CE*, April 2004, pp. 24–26.

THE BASICS OF ROTOR/STATOR MIXERS

In a batch rotor/stator mixer (Figures 2 and 3), a rotor turns at high speed within a fixed stator. Materials from the surrounding batch are drawn up into the high-speed rotor from below, where they are accelerated and expelled radially, at high velocity, through the openings in the stator. With each pass through the rotor/stator, the fluid is subjected to mechanical and hydraulic shear that reduces droplet/particle size quickly.

Depending on a multitude of variables — including rotor/stator diameter, the clearance between the rotor and stator, the configuration of the stator, the peripheral speed of the rotor, tank capacity, mixer position in the tank, and the physical and chemical properties of the material being mixed — the process will progress quickly at first, but will then approach an asymptotic limit. Beyond this point — which we informally call the point of mixing equilibrium — the product will not change significantly, despite additional mixing. ❑

FIGURE 2. As the high-speed rotor turns within the stationary stator, fluids are expelled radially through the openings. The resulting mechanical and hydraulic shear forces reduce droplet/particle size quickly

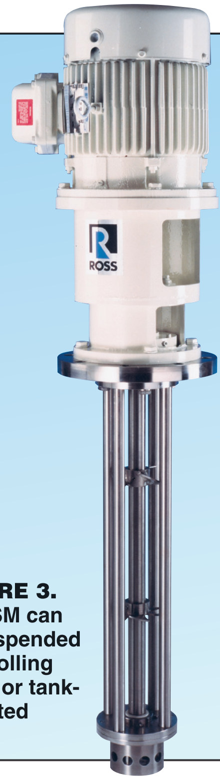

FIGURE 3. An HSM can be suspended on a rolling stand or tank-mounted

available knowledge about high-shear mixing is that misconceptions regarding the proper application and use of HSMs have proliferated. There are numerous commonly held misconceptions and commonly made application errors. Readers who are able to avoid these errors will save time and money in their search for the best rotor/stator mixer, and reduce their risk of choosing a mixing system configuration that looks fine in the laboratory but fails to perform adequately on the plant floor.

Scaling up

In virtually any application, scaleup is a critical process that impacts your business in a multitude of ways, from proper planning of plant floor design and equipment configuration, to operating procedures, to the net operating- and capital-cost impact on the bottom line. In laboratory-scale trials, misjudging the time required to achieve mixing equilibrium by just a few seconds can ultimately cost your company millions of dollars, not to mention wasted time and effort and increased wear-and-tear on the equipment, during commercial-scale production.

The laboratory table-top HSM usually represents the first step in exploring the particular benefits of rotor/stator technology for a given application. This familiar laboratory tool is generally equipped with a variety of interchangeable attachments that allow it to operate in a variety of mixing modes — as a conventional HSM, as a propeller mixer, and as a high speed "saw tooth" disperser (Figure 1). Such versatility is vital in bench-scale development, because it allows the research-and-development person to quickly test many diverse processing strategies.

However, as valuable as the lab-scale mixer may be, it is also the source of one of the most common and costly mistakes in the scaleup from laboratory-scale HSM to pilot-scale and production machines. Unless laboratory testing is conducted systematically and with great care and accuracy, subtle errors in over-processing on the benchtop can produce enormous errors in scaleup projections. Such errors are particularly common, because many engineers underestimate the lab-scale mixer's extraordinarily high throughput-to-product-volume ratio.

Before we move further, let's pause to explore one more concept: equilibrium mixing results. For practical purposes, this is the point at which the mixed product has acquired a target characteristic — such as a specific droplet or particle-size distribution — that will not change significantly,[2] no matter how long you continue to process the product. When we work with dispersions, this is the point at which we reach the equilibrium particle size. For emulsions, it's the equilibrium droplet size (Figure 4).

2. For our purposes in this article, we are focusing on the target average droplet or particle size. With additional processing, this will not change significantly. However, additional procesing (sometimes called overprocessing) will generally affect the particle or droplet size distribution, which can be an important parameter in many applications. For the sake of simplicity and clarity in this section of our discussion, we defer this issue to a later section.

Whether we are working with emulsions or dispersions, this much is certain: we will reach equilibrium much faster with a lab-scale mixer than with a scaled-up pilot or production unit. Depending upon the application and the rotor/stator design we use, we may reach this mark in one tank turnover or in several hundred tank turnovers.[3]

Now, consider this typical real-world scenario involving a test with a lab-scale mixer. Take a two-liter beaker and add the following ingredients to prepare an emulsion:

- Water phase
- Oil phase
- Water- or oil-miscible surfactant

Now, lower the batch-type lab HSM into the liquid. But before you push the start button and head down the hall for another cup of coffee, consider this:

- That little 1-3/8-in. rotor/stator generator on your mixer may operate with a throughput of 100 liters per minute or more
- With a 2-liter batch in the beaker,

3. "Tank turnover" refers to the process of subjecting one complete batch of material to a specific mixing action. In a batch process, this is almost always a theoretical approximation based on mixer flowrate and vessel capacity. Actual results conform to a normalized, Gaussian distribution. A true batch tank turnover, in which the entire batch is literally subjected to exactly one high-shear event, occurs only when the batch material is piped from one vessel, through the mixer, into a second vessel.

that translates to one complete batch turnover every 1.2 seconds

- Presuming that in this application 10 tank-turnovers produce the desired emulsion (a plausible number for many simple emulsions), this means that you may reach mixing equilibrium in just 12.0 seconds!

In the real world, this is where human nature takes over. As you go for coffee, you keep the tabletop batch going for five minutes, and when you check the results you find that the droplet size distribution of your emulsion is right where you want it to be. A success!

But what really happened? You processed the batch for five minutes, turned the batch over 250 times, and reached the right endpoint. But your product did not change once it had reached its mixing equilibrium in just 12 seconds — so the remaining four minutes and 48 seconds produced no appreciable change in the mixed product. That's the margin by which you actually overshot your mixing equilibrium.

In a lab-scale example, overprocessing by four minutes and 48 seconds may not seem like a big deal — but consider the implications in terms of productivity, energy costs, labor, and wear and tear when such an error is propagated during scaleup to a larger pilot- or production-scale unit.

Now, fast-forward to your scaleup requirements using the above example. Consider that you will need to produce this product in 500-gallon batches. If you assume that you will need 250 tank turnovers to accomplish your process goals (instead of 10, which is really all you need), then you will select a top-entering, batch HSM that will process 125,000 gallons through its rotor/stator generator in an acceptable period of time.

Drawing from experience, we assume that a 30-hp unit with a 7-in.-dia. rotor will pump roughly 500 gal/min. Therefore, our 250 tank turnovers (125,000 gallons) will require 250 minutes (4 hours, 10 minutes). This projects to a capacity of roughly two batches per 8-hour shift, or 10 per single-shift week.

If, at the lab scale, we had better understood that the process goal was reached in just 12 seconds (10 turnovers), we could have projected that the same production unit would complete the task in about 10 minutes. This projects to roughly 240 batches per week — an increase of 230 batches per week.

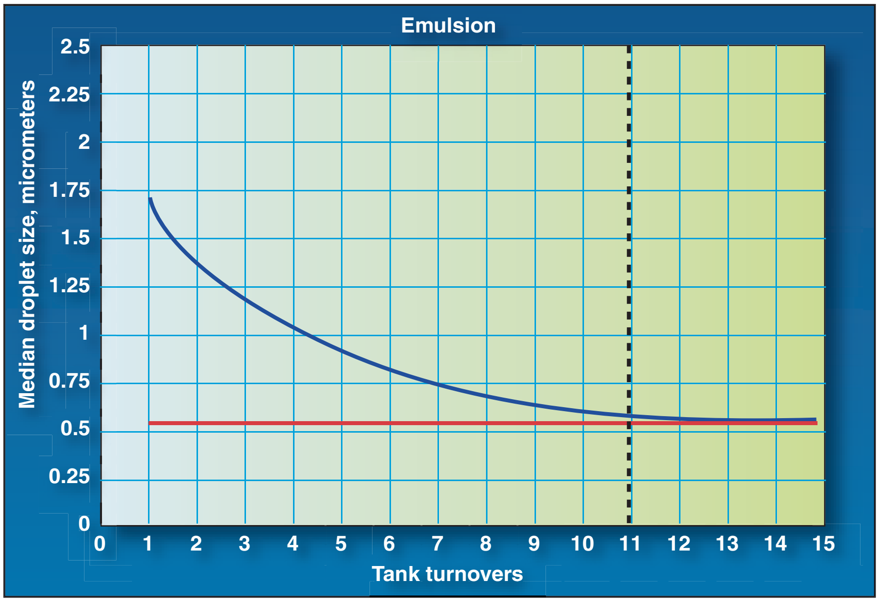

FIGURE 4. This graph shows the mixing equilibrium curves for a typical emulsion. The horizontal line represents the asymptote; the vertical line represents the equilibrium point. During high-shear mixing, the desired characteristic is reached in a given amount of time. Beyond this point, additional mixing will just result in wasted effort, energy and wear and tear on the equipment

Batch versus inline mixing

The emergence of an inline HSM represented a profound step in the evolution of high-shear rotor/stator mixing technology. The innovation was a breakthrough, but the essential concept was simple: First, take the same rotor/stator generator that works in the top-entering batch HSM and install it in a housing with inlet and outlet connections. Next, drive the rotor through a shaft seal and you have a rotor/stator mixer that behaves like a centrifugal pumping device (Figure 5).

The inline HSM offers many benefits. Because the inline mixer is positioned in a flowing stream, the mixing process is more closely controlled than in a batch configuration, so the number of passes through the high-shear zone can be monitored with greater confidence. Solid and liquid additions can also be injected into the flow and dispersed with well-understood results.

Inline HSMs also provide practical solutions for real-world problems on the plant floor. For tanks that are already equipped with low-shear, gentle-mixing agitators, for example, the use of an inline HSM lets operators add a high-shear mixer without disturbing pre-existing equipment. The inline mixer can simply be positioned on the floor alongside the tank. Batch materials can be tapped from the tank for processing through the high-shear rotor/stator generator, and then returned to the vessel.[4]

This configuration eliminates all the difficulties of trying to squeeze a top-entering mixer into the vessel along with pre-existing mixers, baffles and other obstacles. It allows the plant engineer to forget about headroom issues that sometimes arise when long-shafted batch HSMs are retrofitted to existing tanks. It also simplifies maintenance, since the inline HSM doesn't need to be removed from the tank for periodic maintenance.

The appeal of the inline alternative is strong, but how do we translate a batch mixing process to an inline

4. For additional efficiency, the inline mixer can be rolled up to the tank, operated for only a portion of the overall mixing cycle, then rolled to another tank to perform a similar function. In this way, a single, portable inline mixer can serve numerous tanks rather than sit idle in a fixed installation for a substantial portion of the mixing cycle.

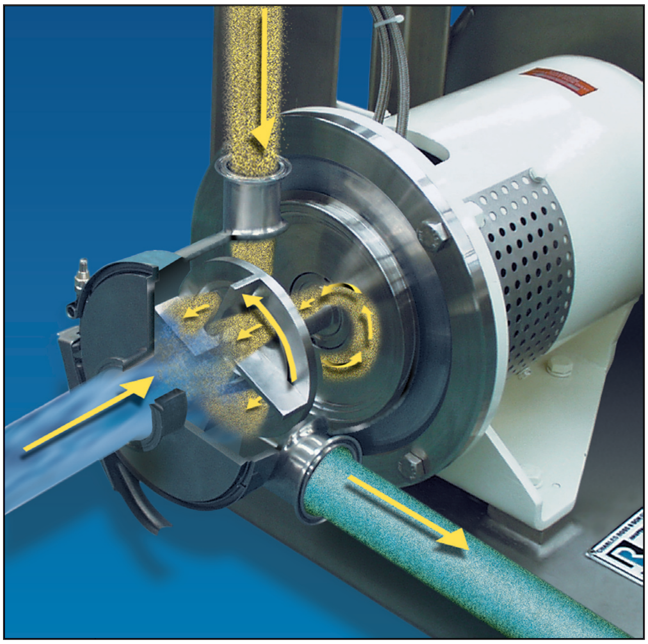

FIGURE 5. Shown here is an inline HSM specially modified to inject powder or liquid additions directly into the high-shear zone, where they are instantly dispersed without clogging

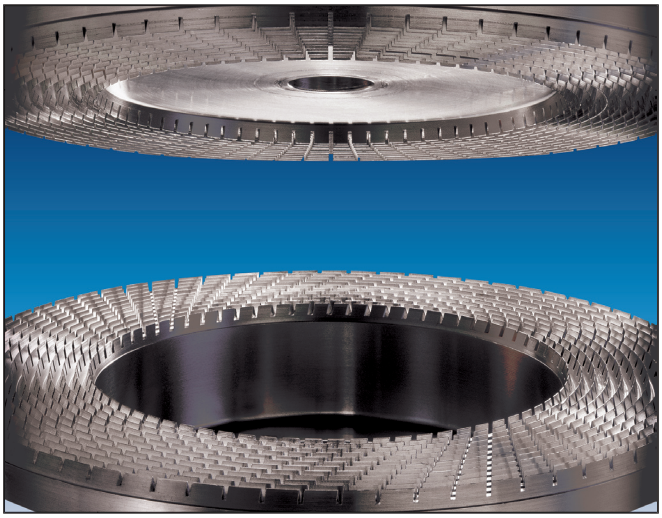

FIGURE 6. The rotor and stator of an ultra-high shear mixer (UHSM) are made up of many concentric rows of intermeshing teeth. The rotor turns at extremely high speed — reaching tip speeds as high as 18,000 ft/min. Fluid begins at the center of the generator and moves outward through radial channels cut in the rotor/stator teeth. As the mix material moves toward the outlet, it is subjected to intense mechanical and hydraulic shear — and thousands of shearing events in each pass

equivalent? Starting with our earlier example involving the 30-hp batch HSM with a 7-in.-dia. rotor, your first impulse might be to swap it for a 30-hp inline HSM with a 7-in.-dia. rotor. This is a presumption that many process engineers make every day, but it overlooks an essential difference between batch and inline HSMs.

Unlike the batch HSM, whose discharge is restricted only by the fluid surrounding the rotor/stator, the discharge of an inline HSM is severely restricted by the mixing chamber, the pressure drop from the outlet connection, and all other downstream sources of pressure drop.

To understand the magnitude of the flow reduction in the inline HSM, consider a 30-hp batch HSM with a 7-in.-dia. rotor that produces a throughput of roughly 500 gal/min in a low-viscosity liquid. An inline HSM driven with equal horsepower will pump less than 250 gal/min. Adding long piping lengths, elbows, valves and other restrictions will lower the throughput even further.

So, how does the limited flow of the inline HSM affect scaleup? Consider the 30-hp batch HSM mounted in a 1,000-gal. tank. In our hypothetical application, the process requires 10 tank turnovers, so it will require 20 minutes to reach our process goal. On the other hand, the 30-hp inline HSM, servicing the same 1,000-gal. vessel will take 40 minutes — twice the processing time. Over a year — or even just a week of single-shift processing — the accumulated impact of this disparity will become enormous and can easily make the difference between profitable and unprofitable production.

If an inline mixing solution is necessary (that is, if a batch solution is simply impractical with your current equipment, available space or throughput requirements), you will need to consider a substantially larger inline unit to duplicate the processing capacity of the batch unit. In this case, to match the batch mixer's 500-gal. flowrate, you would have to step up from the 30-hp inline mixer to a 50-hp inline unit equipped with a 11-in. rotor/stator generator.[5]

The essential principle to remember here is that an inline rotor/stator mixer is not a drop-in replacement for a batch mixer of equivalent horsepower. You will have to compromise on throughput or invest in a more substantial inline mixer. The correct choice will depend on your business and processing priorities in each application. You should also consider whether a switch to an inline configuration will provide additional advantages of value in your application — such as the ability to inject hard-to-disperse powders into your batch using the same inline mixer.

5. This comparison is meant to illustrate generale scaleup relationships without regard to numerous variables that would normally be considered as well. In actual practice, we would also consider pipe diameter.

High shear vs. ultrahigh shear

Since most industrial processes don't take place in a beaker, we must always consider the real-world behavior of high-shear batch mixers in large vessels. Thinking on a molecular level, we ask, "How many times does each particle or droplet pass through the high-shear zone?" Backing up to see the process from a wider perspective, we ask, "How consistent are my results? How uniform is the distribution of particle or droplet sizes in my batch?" In many applications these are critical questions because they can profoundly influence the properties of your end product.

The daily challenge in high-shear rotor/stator mixing is to reach the target droplet or particle size and achieve a satisfactory particle-size distribution in the most cost-effective manner. This requires a careful balance of rotor/stator design, cycle time at a required batch size, capital cost, and per-cycle operating costs.

Any batch mixing process — whether the process goal is particle dispersion, particle-size reduction or emulsification — generates a Gaussian distribution of results. The greater the ratio of product volume to HSM throughput, the broader the distribution will be. Of course, the goal is usually to produce the narrowest distribution possible with an equipment solution that meets the site-specific process and business requirements. The question is simply, "What is the most effective, economical, and practical way to produce the required particle- or droplet-size distribution?"

As discussed earlier, at mixing equilibrium we have reached the target average particle size. Additional processing will gradually narrow the distribution curve, but extending the process for this purpose alone almost always amounts to a substantial waste of time and energy.

An alternative strategy is to increase the size of the HSM. This will increase the ratio of HSM throughput-to-product-volume and narrow the curve. But this will also increase both the initial capital investment and the ongoing energy costs. The attractiveness of this solution depends heavily on the value of the product being manufactured, its competitive strength, and the overall business case for investing heavily in equipment.

A radical strategy for narrowing the particle-size distribution would be to use two tanks and a conventional inline HSM. You would pump directly from the first tank, through the inline HSM, and into the second tank. The process would then be reversed: pumping from the second tank, through the inline HSM, and back into the first tank. This cycle can be repeated over and over until the product reaches the desired characteristics.

With this method, the shear history of the product could be more closely monitored than in customary batch or batch-recirculation processes. Of course, the expense of purchasing and tying up two tanks is usually prohibitive. This technique would also be extremely time-consuming, and it would require either constant operator intervention or complex automation.

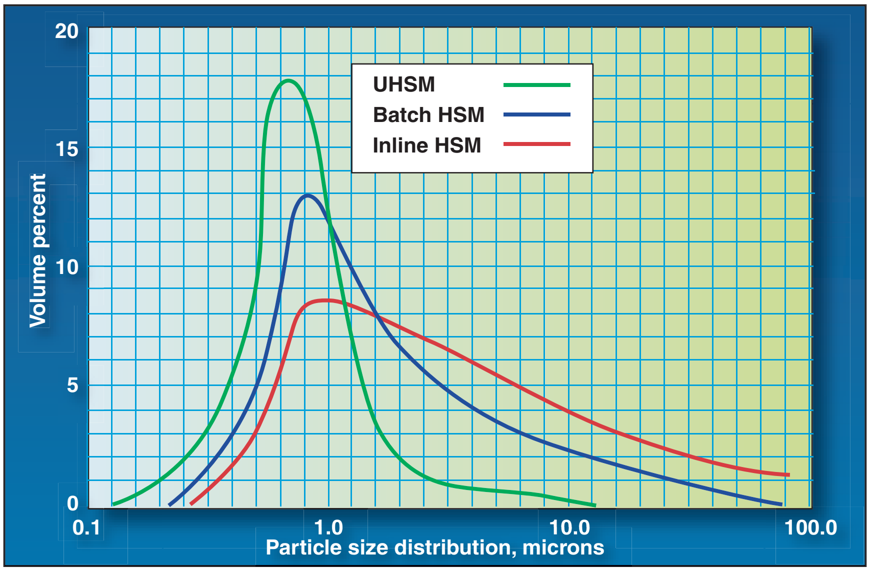

FIGURE 7. Shown here are the particle-size distributions one can expect to achieve with a batch HSM, inline HSM and UHSM. Both the batch and inline HSMs shown here are 10-hp units operating with a 100-gallon tank, recirculating for the required four minutes. In applications where a narrow particle-size distribution is required — signifying an extremely uniform emulsion or dispersion — the UHSM should always be evaluated as a potentially cost-efficient design alternative

Try an ultrahigh-shear mixer

The best solution is often to switch to an ultrahigh-shear inline mixer (UHSM). In select applications, an UHSM can achieve the desired process goals in a single pass, allowing this device to process batches of product in a "terminal" approach (that is, with one single pass of the tank contents through the UHSM, and then on to the next phase of the process). Although the UHSM typically represents a higher capital investment than a traditional single-stage HSM, its ability to complete the process in a single pass often allows us to step down to a lower-throughput unit. This reduces initial equipment cost without an unacceptable sacrifice in production.

The single-pass performance of the UHSM is attributable to the extraordinarily high shear it applies and the large number of shearing events to which it subjects the mix material in each pass (Figure 6). This also accounts for its ability to produce an exceptionally narrow particle size distribution with a single pass (Figure 7). For products that require great uniformity in a dispersion or emulsion, this can be a decisive advantage.

A recent example of this solution involved a manufacturer in the asphalt industry. In this case, a clay filler is added to asphalt at 400°F to boost its viscosity for use as a thixotropic automotive undercoating. After an initial pre-mix using a large-sweep agitator in a 2,000-gal. tank, the material requires mixing under high shear to deagglomerate and disperse the clay so that it develops its thixotropic properties.

An initial test on the benchtop reached the specified endpoint (defined in this case as target viscosity) in 30 minutes. A scaleup calculation based solely on mixer flowrate suggested that a 25-hp inline mixer with a 4.5-in.-dia. rotor would be an appropriate choice for production. Operating at 3,600 rpm, and with a flowrate of 150 gal/min, this would be a logical choice in most applications. But in this case it was impractical. The 30-minute cycle on the benchtop produced roughly 900 tank turnovers. Projecting to full-scale production in a 2,000-gal. tank, the process would require 7,200 minutes — or five days.

The best solution proved to be a switch to a UHSM operating at 30 hp with a 6-in.-dia. rotor and a flowrate of 20 gal/min. After the pre-mix, the UHSM successfully dispersed the clay and built up the viscosity in one pass, a batch that required 100 minutes — just 1 hour and 40 minutes. The material was then passed directly to filling equipment, where it was dispensed into final packaging.

Don't assume...test!

Understanding high-energy rotor/stator mixing is a daunting challenge on any level. It is far more complex than traditional low-shear mixing, which makes rigorous modeling and academic study extremely difficult. In the absence of such guidance, operators must rely on the instinct, experience, empirical testing and other technical resources offered by their high-shear-mixer manufacturer to find the most efficient and cost-effective HSM solution.

The best strategy is to consult with a mixer manufacturer that can provide a well-equipped laboratory with quantitative analytical support for a thorough process test. Bring your own ingredients, and specify process conditions carefully to accurately simulate conditions on your process line. Most important, test using a variety of equipment, from traditional single-stage batch and inline rotor/stator mixers to ultrahigh-shear devices. Even with an expert guiding you through the selection process, you cannot possibly know for sure that you have chosen the best equipment for the job until you've tested numerous possibilities.

In most of the CPI, competition is more intense today than ever before. So, even small gains in production efficiency can be vital to build your product's competitive strength. Measure your benchtop results meticulously, because every second counts and projections to full-scale operations can compound the impact of small, lab-scale errors. Evaluate your process results quantitatively with appropriate instruments such as particle-size analyzers. Leave nothing to chance or supposition. And above all, project carefully from the benchtop to full-scale production before you buy any scaled-up systems. An extra measure of rigor and diligence at this stage can certainly mean the difference between success and failure on the plant floor and profitability in the marketplace.■

Edited by Suzanne Shelley

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