

Custom-engineered jet-milling system helps maintain world health

A company uses a toll processor's services to mill a heat-sensitive powder to a narrow uniform particle size distribution.

Custom processor

Knowing whether a vaccine is viable or a food product is edible ensures that people are properly vaccinated and able to avoid foodborne illnesses. Since aging (time) and exposure to high temperatures can negatively affect many pharmaceutical and food products, it's important for manufacturers and end users to have an accurate and reliable way to monitor a product's exposure to them. For more than 25 years, TEMPTIME Corp., Morris Plains, N.J., has been developing and manufacturing a variety of such monitoring devices, called time-temperature indicators. The indicators use a specially developed monomer reagent powder that irreversibly changes color from light to dark when exposed to excessive heat over time, ultimately signaling when a product's time-temperature profile is exceeded. When developing the reagent powder, the company worked closely with a toll processor to build a custom-engineered milling system to consistently and uniformly mill the powder.

The time-temperature indicators

TEMPTIME's time-temperature indi-

cators are a cost-effective and reliable way to monitor a pharmaceutical or food product's exposure to temperature over time. For instance, the company's HEATmarker vaccine vial monitor indicates whether a vaccine vial has been exposed to high temperatures, ensuring that only an active vaccine is administered. And the company's Fresh-Check Indicator for food products provides consumers a way to verify the freshness of the foods they bring home. To date, more than 3 billion time-temperature indicators have been sold for use in the health and food industries throughout the world. This includes all vaccines currently used by the World Health Organization (WHO) and the United Nations Children's Fund (UNICEF) for their worldwide vaccination campaigns.

"The key to our indicator technology is a diacetylene monomer reagent powder we call KE," says Dr. Carl Lentz, TEMPTIME senior vice president and chief operating officer. "The KE powder, which is sensitive to heat, is used to make an ink that's applied to a media surface that's made into the



The cryogenic jet-milling system uniformly mills a heat-sensitive powder to a mean particle size range of 8 to 12 microns.

indicators. Each indicator is basically a little white dot that has a reference ring around it, with the indicator's active piece inside the reference ring."

The indicator is small enough that it can be incorporated into an adhesive label that's affixed to a vial, ampoule, syringe, food package, or other small container. A new indicator starts out bright white and then over time progressively and irreversibly darkens at a predictable rate as it's exposed to heat or light. The company can custom-formulate the indicators to meet the stability criteria and time-temperature profile of a customer's product.

Developing the indicators

A US-based contract manufacturer produces the KE powder using a crystallization process. This results in a powder with a broad particle size distribution range with particles larger than 30 microns as well as some agglomerates. When developing the time-temperature indicator, the company's testing showed that the KE powder needed to have a more uniform and narrower particle size distribution to consistently ensure reliable results. To accomplish this, TEMPTIME needed to find a way to mill the

powder without exposing it to heat, light, or mechanical shear stress.

The company worked with several toll processors to test various milling equipment during the indicator's development phase. "We first tried using bead mills, but the heat generated during milling caused some color to develop in the powder," says Joe Stavisky, TEMPTIME quality control manager. "Then we tried a three-roll mill, but the mechanical shear generated heat, which also caused color to develop in the powder. And since the powder's color is affected by ambient temperatures as well, it needs to be stored and processed at a cold temperature. There didn't seem to be a good way to do that with either of those mills."

Finally, the company contacted a nearby toll processor that uses jet-milling equipment for size-reducing dry flowable materials, ranging from talc to diamonds. A jet mill reduces a material by creating high-speed particle-on-particle collisions in its grinding chamber. Material is fed at a constant rate into the mill's grinding chamber where tangentially oriented jet nozzles generate a spinning vortex of air or gas that moves the particles

around the chamber's interior at near sonic speeds, causing the particles to collide at a high velocity.

The company sent several powder samples along with the powder's MSDS to the toll processor's facility for milling tests using a closed-loop liquid-nitrogen cryogenic jet-milling system. The tests demonstrated that the toll processor's Micron-Master orbital jet mill could effectively reduce the powder to the required particle size and distribution range without generating heat.

The toll processor sent the milled powder back to the company along with a grinding report documenting such technical information as the jet mill operating parameters and the powder's initial and final particle analysis results. After its in-house particle analysis lab verified the test results and the milled powder's quality and color, the company decided to have the toll processor mill the powder to meet its production needs.

"The cryogenic jet-milling system proved ideal for our application since the powder isn't exposed to any heat during the milling process," says Lentz. "The jet mill also breaks up any agglomerates and effectively produces a narrow particle size distribution with a low mean particle size. In addition, we found it was better for us to have the powder toll-processed because they're the experts in jet-milling technology, and it would have taken too long and cost too much for us to develop and perfect the technology in-house."

To mill the powder to meet the company's handling and processing requirements, the toll processor developed a closed-loop cryogenic milling system to maintain a constant temperature of -40°F during the milling process and virtually eliminate the powder's exposure to ambient temperatures. The toll processor also custom-engineered a method for collecting the milled powder and incorporated it into the closed-loop sys-



An operator adjusts the jet-milling system's operating parameters to ensure a quality finished product with no material loss.

tem to further protect the powder from exposure to heat or light. Additionally, the toll processor integrated a volumetric feeder into the milling system to ensure reliable feeding of the low-density powder.

The toll processing facility and equipment

The Jet Pulverizer Co. Inc., Moorestown, N.J., manufactures jet-energy milling equipment and provides custom toll processing and R&D services to companies in the dry bulk solids industries. The supplier operates a 15,000-square-foot toll processing facility with 15 dedicated processing stations and a 10,000-scfm, oil-free, compressed-air source. Each processing station is a self-contained cell that operates as a complete size reduction operation and includes precrushing, blending, drying, classifying, mechanical milling, jet milling, and particle size analysis capabilities. The supplier can process abrasives, pigments, cosmetics, food additives, heat- and moisture-sensitive materials, non-sterile cGMP materials, and many other dry materials, but it doesn't handle hazardous, toxic, or carcinogenic materials. The toll processing facility is ISO 9001:2000-certified and FDA-registered and operates according to cGMP standards.

The facility has 2-, 4-, and 8-inch jet mills for laboratory work and 12-, 15-, 20-, and 24-inch jet mills for production milling. The production-size jet mills have ceramic-lined material contact surfaces to eliminate contamination or discoloration of the material being milled. Stainless steel or resilient liners are available for milling softer materials. In addition, the facility performs low-temperature grinding and inert milling with nitrogen gas on a test or production scale, and crushing, hammer-milling, and blending equipment is available for full-scale production runs.

The facility's particle analysis laboratory's equipment includes laser-diffraction particle size analyzers with wet and dry modules, sieve classifiers, an optical microscope, a bio-

metric density meter, a sub-sieve sizer, and a tap density analyzer.

The supplier custom-designs each milling application to meet or exceed a customer's requirements and welcomes customers who want to witness the grinding tests. The toll processing facility can produce test and production quantities ranging from a few grams up to truckloads.

Besides operating the toll processing facility, the supplier also manufactures five types of jet mills, each of which can be custom-designed to suit a customer's application. Available jet mill sizes range from 1 to 42 inches with outputs from 0.2 to 10,000 lb/h. The jet mills can reduce materials to less than 10 microns, with averages ranging from 1 to 5 microns. If required, an average particle size of 0.25 micron can be achieved on many materials.

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Toll processor proves its worth

TEMPTIME has been using the toll processor's jet-milling services for more than 10 years to uniformly mill the KE powder to a mean particle size range of 8 to 12 microns, with 100 percent of the particles being less than 30 microns. "We've continued working with the toll processor over the years because they're able to produce a top-quality final product every time," says Stavisky. "This enables us to reliably produce the inks for the time-temperature indicators so that they have the same reactivity and rheological properties from lot to lot. The toll processor has been very accommodating to our schedule throughout the years and can process the powder as we need it, which give us flexibility when producing the indicators."

"It's worth our while having the powder milled outside of our facility because the toll processor makes it easy for us to work with them," says Lentz. "They implement their technology in a business-friendly manner and are willing to adapt and custom-engineer their milling systems to accommodate our needs. And since they have laboratory-size mills, as we've developed different formulations or monomers they've handled them at the lab scale for us during the testing phase and then scaled-up the process to their larger production-size mills. They're a full-service provider from development through full-scale manufacturing, and they've been with us every step of the way." **PBE**

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