Cryogenic size reduction can provide benefits for many processing applications, including thermoplastics and spices. Surprisingly, these disparate applications have key attributes in common that cryogenic grinding effectively addresses.

Cryogenic Grinding: A Multitasking Marvel

From spices to thermoplastics, cryogenic size reduction can provide benefits to a variety of industries.

By Khatera Mohd-Habib and Tim E. Boland, Air Products

Liquid nitrogen (LIN) has been used for more than 30 years in cryogenic grinding to "super-refrigerate" size reduction processes in industries such as food processing, plastics recycling, powder coating, viscosity control additives and tire recycling. Liquid nitrogen offers a number of unique benefits to size reduction processes.

- Liquid nitrogen is one of the coldest liquids on earth with a boiling point of -320°F (-195°C). This means that liquid nitrogen can be used to cool materials that require very low processing temperatures such as polyolefins or similar tough-to-grind materials.
- Because nitrogen does not support combustion, it can help make size reduction operations safer.
- Nitrogen is inert and does not react with other substances under normal conditions.
- Unlike liquid carbon dioxide, which forms carbonic acid when it dissolves in the moisture inherent to food products, nitrogen does not affect the pH of food products. In fact, liquid nitrogen has a lower boiling point than liquid carbon dioxide.

The basics of liquid-nitrogen-based cryogenic grinding technology are similar across many industries. Two diverse industries — the preparation of powdered spices and the grinding of thermoplastic resins for powder coatings — illustrate how liquid-nitrogen-based cryogenic grinding technology can be successfully applied by very different industries.

Both spice grinding and thermoplastic grinding require equipment designed

Recently developed cryogenic grinding mills can generate consistent yields of ultra-fine particles.

with the following features:

- Ease of cleaning.
- Ability to operate at extremely cold temperatures.
- Good control of the liquid nitrogen and material feed rates.
- Capable of being demonstrated in the manufacturer's test laboratory.

Ease of cleaning is equally important for both the preparation of powdered spices and the grinding of thermoplastic resins. Obviously, ease of cleaning is critical for systems that process spices, neutraceuticals and pharmaceuticals because of food safety and hygiene concerns. Ease of cleaning is also important in the processing of plastic resins because many processors like to use a single grinding system to process a range of products. As a result, these systems must be thoroughly cleaned to prevent cross-contamination among products. Achieving a high level of cleanliness when alternating among different products in the processing of thermoplastic powder coatings is especially important when, for example, different resin colors are involved.

Cryogenic grinding systems must be designed to operate at low temperatures (approximately -100°F (-73°C) or colder) to successfully grind many thermoplastics and spices, especially those with high oil content. The grinding systems must be well insulated, especially where the liquid nitrogen contacts the material being processed. Insulation is important to minimize the loss of liquid nitrogen refrigerant power to the surrounding environment. It also is important to protect the equipment operators from coming into contact with the liquid nitrogen. Liquid nitrogen can cause severe burns due to its extremely low temperature.

As the cryogenic grinding system cools down from ambient temperatures during startup, the system components will shrink. For example, a 10' length of stainless steel piping will shrink by more than 0.125" (3 mm) as it cools from ambient to -100°F (-73°C). A longer pipe will shrink even more. Therefore, the grinding system must be designed to accommodate this shrinkage — and the stresses generated. Because many metals such as carbon steel as well as some plastics become brittle at low temperatures and can fracture, the grinding system must be designed with components that can tolerate the low temperatures required without failure.

Maintaining good control of the relative flows of the material being processed and the liquid nitrogen being used to cool the material is crucial to the successful commercial operation of a cryogenic grinding system. For example, spices are temperature sensitive. The heat generated from the actual grinding can cause spices to lose their essential oils, distort their aroma and flavor, and alter their color, all of which reduce the quality of the spices. Additionally, if the spices are not processed at a low enough temperature, the oils and fats in the spices can cause agglomeration and clog up the mill. Good control of the operating temperature via good liquid nitrogen flow control can help prevent these issues.

Surprisingly, grinding thermoplastic resins presents similar challenges. While most thermoplastic resins are not as temperature sensitive as spices, poor control of liquid nitrogen flow can cause issues with the out-of-specification particle size due to high processing temperatures. In recycling processes, for example, off-spec and post-industrial waste of thermoplastics are reprocessed and added to other compounds as fillers to increase toughness and strength characteristics of the resin matrix. This process requires consistent fine particles to ease blending and achieve an eco-friendly process. Thus, fluctuations in particle size may interfere with the efficiencies of such processes. Production rates will also fall as the processing temperature increases. If the temperature gets too high, the resin may even partially melt and clog up the mill.

Testing is an important aspect of any size reduction system. Size reduction, especially of thermoplastics or elastomers, is still a very empirical technology. The equipment supplier should be able to demonstrate the performance of their equipment with the substrate being ground.

While grinding applications may differ, the desired end result is often the same — achieving the finest particle size and most uniform particle distribution while maximizing production rates and minimizing overall operational costs. Cryogenic size reduction technology using liquid nitrogen to remove heat produced during the grinding process can result in a finer, more consistent particle size distribution and higher throughputs for many products compared to conventional grinding methods. These products include adhesives and waxes, carpets, color concentrates and pigments, composites, grains, pharmaceuticals, plastics, powder coatings, metal, multi-component materials, rubber, and spices and herbs. **PC**

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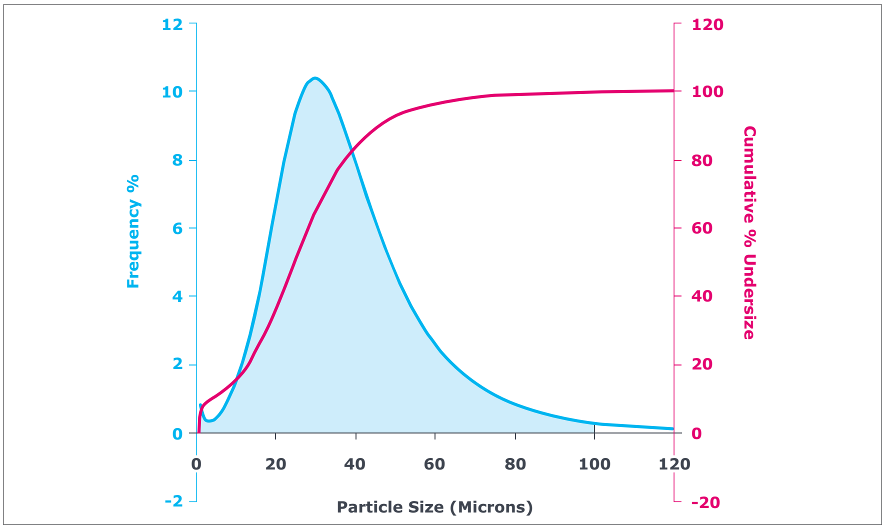

Figure 1: Nutmeg ground on Air Products PolarFit™ ultra-fine-grinding system

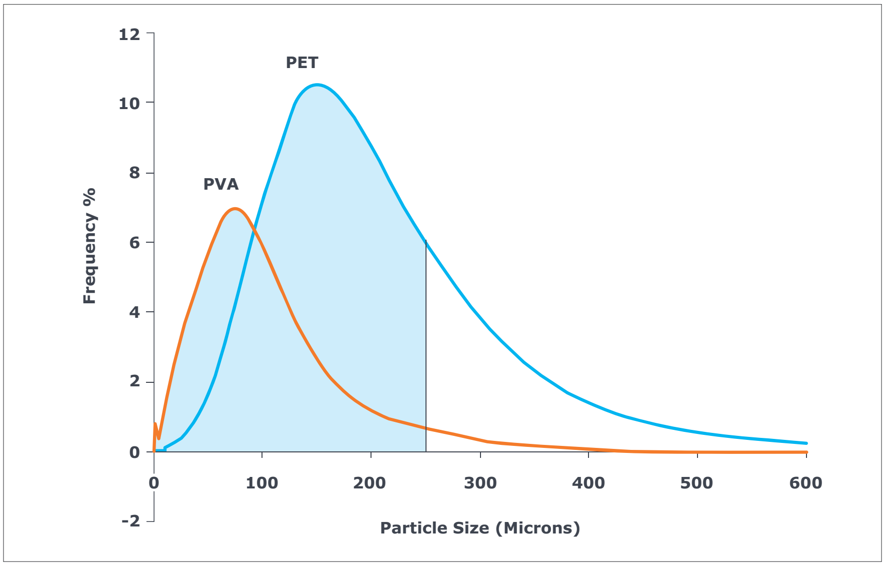

Figure 2: Various thermoplastics ground on Air Products PolarFit™ ultra-fine-grinding system

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■ Ultra-Fine Grinding

A combination of size reduction mechanisms — impact, attrition and particle-particle collision — help achieve much smaller particle sizes than other mills.

The PolarFit™ ultra-fine-grinding mill generates consistent yields of particles between 45 and 250 µm (60 and 325 mesh) and particles as small as 10 µm in some cases. Test results using this mill illustrate how a single system can be used across industries.

Figure 2 presents the same single-pass type results for processing various thermoplastics. In a single-pass test, the material is processed by the pulverizer, and all the resultant particles are collected and analyzed. Particle size classification equipment such as a screener is not used to classify the particles by size, and it recycles the over-size materials to the feed.

This type of test provides insight into the grinding mill's size reduction capabilities.

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