Simpler, quicker, cheaper

Freeze drying: a new take on an old technology

Frozen granular material as produced in the freezing step with Active Freeze Drying

Freeze drying (lyophilization) has been used industrially for decades to preserve foodstuffs and biological materials, but traditional tray-type freeze dryers are slow and labor-intensive. A new "Active Freeze Drying" process promises to eliminate these drawbacks.

Dr. Ir. Peter G. J. van der Wel

The history of freeze drying goes back to the ancient Incas, who preserved potatoes and other vegetables simply by exposing them to the air. Low winter temperatures turned the water in the food to ice, which then evaporated in the very dry mountain air. In the 1930s, industrial freeze drying was developed as a way to preserve surplus coffee in countries such as Brazil. Nescafé and other famous brand names were founded at this time, and instant coffee is now the best-known freeze-dried product. During the Second World War, freeze drying was also used for wartime medical supplies such as penicillin and blood plasma. In the following decades the use of freeze drying expanded to the point where today the technology is used in the production of around 400 different foodstuffs and many pharmaceuticals.

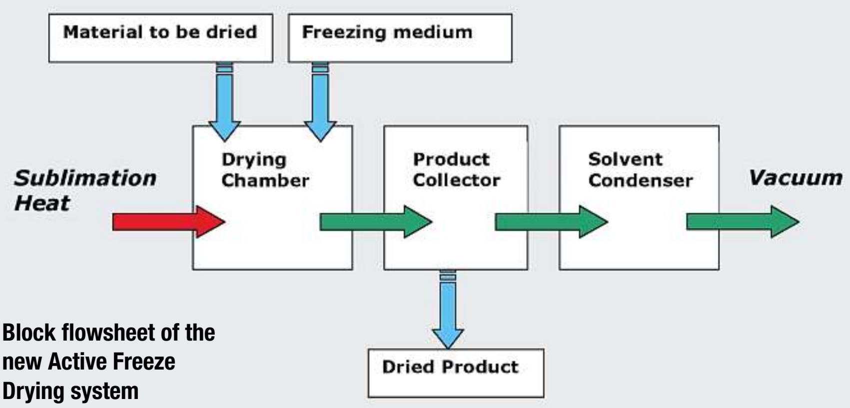
The author is with Hosokawa Micron BV, Doetinchem/Netherlands.
Phone: +31 (0)314/373-570

Problems with traditional method

Ever since the 1930s, industrial freeze drying has depended on a single type of equipment: the tray-type freeze dryer. In a tray freeze dryer, the material to be dried is placed on open trays and supported on shelves equipped with cooling and heating systems. The trays are arranged within a chamber that is connected to a vacuum pump.

First, the material is frozen at atmospheric pressure by lowering the temperature of the shelves. The drying chamber is then evacuated to a pressure typically in the range 0.01–1 mbar. Gentle heat applied to the shelves causes the ice in the product to evaporate without passing through the liquid phase (sublimate). The deep vacuum allows this to take place at temperatures of –20 °C or even lower. If necessary, for instance where organic solvents are used, solvent is recovered from the vacuum pump exhaust by a low-temperature condenser.

Despite successful operation over many decades, tray freeze dryers have three significant disadvantages. First, loading and unloading is labor-intensive, especially since large units may contain several hundred trays. Second, the freeze-dried cake removed from the trays often requires grinding to convert it to a free-flowing granular product. Third, the formation of a "skin" of dried product on the surface of the tray hinders the passage of vapor, making this method of freeze drying very slow.



Block flowsheet of the new Active Freeze Drying system

Keep the product moving

Hosokawa Micron has developed a quicker and less labor-intensive freeze drying process that can produce free-flowing powders from a single process vessel. Known as Active Freeze Drying technology, the new process uses continuous motion to improve mass transfer and hence cut processing time, while also eliminating the need to transfer product to and from drying trays and downstream size reduction devices.

In Active Freeze Drying, the product is first frozen in a specially-designed drying vessel fitted with an agitator. Whether the material to be dried starts off as a liquid, granular solid or paste, the forced motion inside the drying vessel ensures that it freezes in the form of free-flowing solid granules. The size and shape of these granules can be adjusted by changing the geometry of the drying chamber and the speed of the agitator.

Once the product is fully frozen, vacuum is applied and sublimation starts. Heat applied to the vessel jacket is distributed effectively through the product thanks to the motion of the agitator. The initially coarse granules gradually shrink as the ice structure connecting the frozen material sublimes away, yielding a loose powder consisting of dry particles.

As the solvent disappears the product temperature starts to rise, until finally it equals the jacket temperature. This marks the end of the drying process, at which point all the material has been transformed into a fine, free-flowing powder. Once the vacuum has been released, the chamber is opened and the powdered product can be discharged with the aid of the agitator.

Wide application

The main applications of Active Freeze Drying are in the pharmaceutical industry, such as antibiotics and electrolytes. Other products typically produced by freeze drying include proteins, hormones, viruses, vaccines, bacteria, yeasts, blood serum, liposomes and transplant materials like collagen sponge. For all these materials, freeze drying is popular because it preserves the product structure, requires only low temperatures, and yields a final product that is easy to handle.

Another fast-growing market for Active Freeze Drying on a larger scale is in materials, in particular for nanomaterials. Dynamic freeze drying of nanomaterials produced via wet processes helps the suspended particles to remain separate during freezing as well as drying. As sublimation progresses the particles become separated from one another, but the continuous motion of the material induces the formation of weak agglomerates. The product consists of loosely-bound single particles forming a fine cohesive powder.

Active Freeze Dryer batch volumes can range from a few liters for laboratory work and small-scale production, up to bulk drying applications involving hundreds of liters. Whatever the size, the advantages of this new process are obvious: rapid drying, simple product handling and unique product quality. Over the last year Hosokawa Micron has carried out a large number of tests for customers in different fields of application. Materials tested range from regular pharmaceuticals to insects, nanomaterials, bacteria, high-value herbs and other sophisticated food ingredients. The future may even see a continuous version of the process, creating even higher capacity for a given equipment volume. ■

process-worldwide.com
For further information:
www.process-worldwide.com
InfoClick 263725