



The future of FREEZE DRYING

The history of freeze drying goes back to the ancient Inca's, who preserved their food stuff by freezing it in the mountains in winter time. At the same time the frozen water is removed through the low vapor pressure of the water in the surrounding air at those high altitudes. Of course this process was rather slow, but during the drying process the quality of the food was maintained due to its frozen state. Once dried the food materials like potatoes and vegetables showed a remarkably increased quality over time. It is only since the WWII that this way of drying is converted into an industrial method for the preservation of materials. Especially the need for improving shelf live of pharmaceuticals like penicillin and the preservation of blood plasma in war time was a drive for the development of the modern tray-type Lyophilizer as it is still used in many industries today. Another catalyst for the development of the freeze dry technology was the surplus of coffee in countries like Brazil in the late 30's of the previous century. Also here the tray-type freeze dryer was used for the sublimation of the water from the frozen coffee granules. Brand names like Nescafe were founded at that time. Instant coffee is now the most well-known freeze dried product.

After the war period the technology emerged into its current popularity for the drying of many different temperature sensitive materials like pharmaceuticals and food materials. There exist already 400 different freeze dried food materials alone.

Tray freeze dryers

The tray freeze dryers require the use of a large chamber for freezing and a vacuum pump for removing the moisture. In the drying chamber the material to be dried is put on trays and the trays are placed on cooling/heating shelves. First the material is frozen by lowering the temperature of the shelf. The material is frozen in a solid layer of ice. After freezing, the product is placed under vacuum by closing the drying chamber. This enables the frozen solvent in the product to vaporize without passing through the liquid phase, a process known as sublimation. Typical vacuum levels during drying are between 1 mbar and 0.01 mbar. For example when water is sublimated at such deep vacuum levels the vacuum will ensure sublimation temperatures of -20°C or lower.

Heat is supplied via the shelves to the frozen product in order to accelerate the sublimation process. However when the process evolves the formation of a dried layer of product will hinder this sublimation considerably. This makes freeze drying by far the slowest method of drying available. The drying system further consists of a low-temperature condenser removing the vaporized solvent from the vacuum chamber by converting it back to a solid. This completes the separation process of solvent and liquid. After completion of the drying process the trays are removed from the drying chamber and the dried cake is taken out of the tray, often some degree of grinding is necessary to convert the dried matter into a fine powder. Large-scale installations can contain as many as hundreds of tray. So besides the long drying times, intensive handling is a major draw-back of this technology.

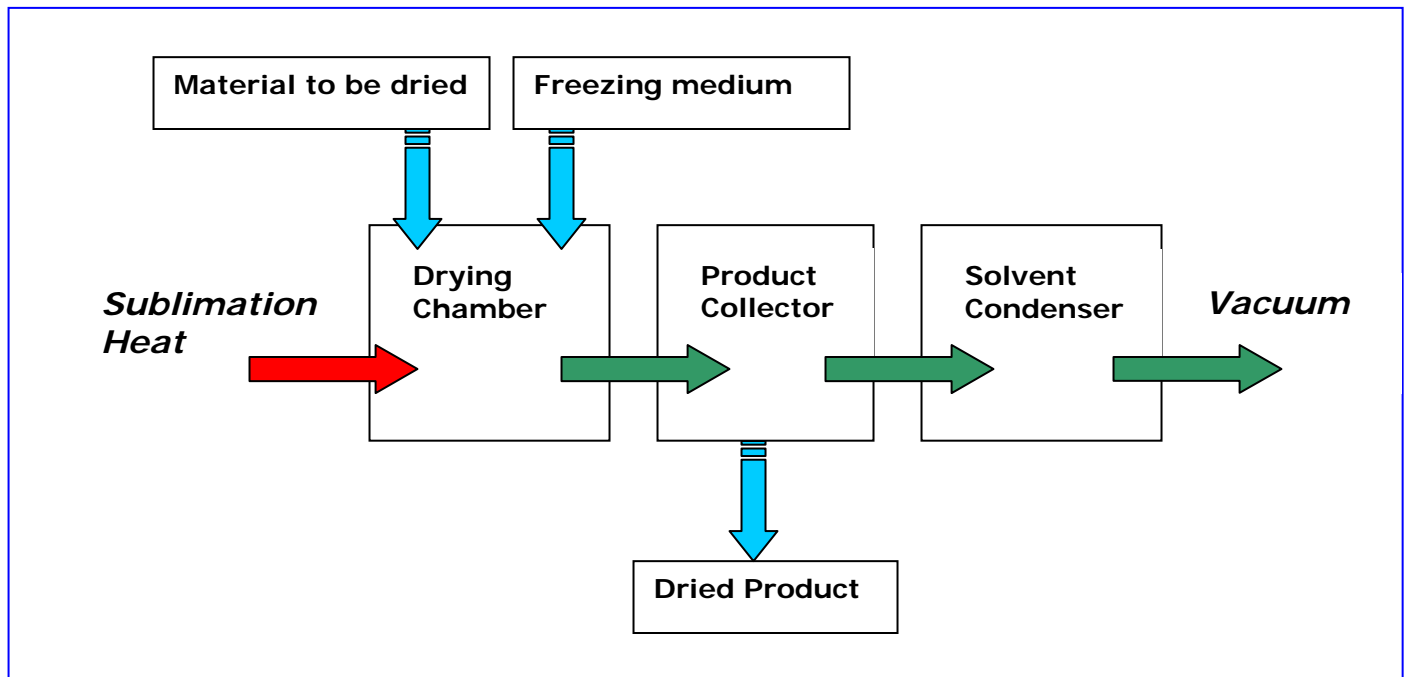


New technology

A quicker and less labor intensive freeze drying process and the possibility of producing loose and free-flowing powder at low temperatures and low pressures, all in one vessel.

Even this short introduction illustrates that the newly developed Active Freeze Drying technology can be seen as a big step in the world of freeze drying and powder technology.

Early experiments with a drying forced drying chamber operated at low temperatures and low pressures showed that it is possible to obtain an efficient freeze drying process. The result is a lump-free, free-flowing product. Active Freeze Dryers exhibit a better heat transfer rate due to the continuous motion of the product. This shortens the drying process because the dried product no longer hinders the sublimation. Finally the freeze drying process is simplified because all process steps can be done in a single processing unit in stead of handling trays filled with product between freezing units, drying chambers and crushers. This results in easy handling of the product especially when compared to the traditional tray dryer equipment.



Schematic drawing of the newly developed Active Freeze Drying system

The positive results of these initial experiments have led to the development of a commercial dynamic freeze drying technology. In a dedicated designed drying chamber the material to be dried is frozen. Due to forced motion of the material, being a liquid, paste or solid, it will be transformed into solid granules. The granules can have sizes and shapes which are controlled by the dynamics of the machine. Once the freezing step is completed the drying chamber is closed and vacuum is applied. After evacuation of the freezing agent the sublimation process will start. From this stage the product temperature is dictated by the vacuum level. During sublimation the heat is supplied through the jacket and efficiently distributed



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throughout the product by the chamber design. The initially coarse granules will gradually reduce in size due to the sublimation of the connecting ice structure in between the frozen material. The released dried particles will make up a loose powder. Towards the end of the drying process when most of the frozen solvent is sublimated the product temperature will start to rise. Finally the product temperature will equalize the wall temperature, indicating that the drying process is finished. By then all material is transformed into a fine and loose powder, After breaking the vacuum the dryer can be discharged easily as a free-flowing material from the dryer vessel, assisted by the transporting characteristics of the drying chamber.

Applications

The main application of this dynamic freeze drying technology is found in the pharmaceuticals, e.g. antibiotics macromolecules and electrolytes are being produced by freeze-drying. Other products typically produced by freeze-drying are: proteins, hormones, viruses, vaccines, bacteria, yeasts, blood serum, liposomes and transplant materials like collagen sponge. For all these products the decisive factor to use freeze-drying is the preservation of the product structure, particle size and the minimal temperature load.

Another fast growing market for the application of Active Freeze Dryers on a larger scale is the materials business, in particular for nano-materials. By using dynamic freeze drying for these wet base processed materials special advantages are obtained. The suspended particles remain separated during the freezing as well as the drying process. During the progress in sublimation single particles will become separated, but the continuous motion of the material will induce the formation of weak agglomerates. The final product will consist of loosely bound single particles forming a fine cohesive powder.

ACTIVE FREEZE DRYING: the future

With the introduction of this new dynamic way of freeze drying a new type of production technology has been made available. Active Freeze Dryer batch volumes can range from a few liters for lab-scale and small scale production applications to bulk drying of hundreds of liters. In all sizes the advantages are obvious: rapid drying, simple product handling and a unique product quality. Over the last year a large number of tests for customer in various fields have been performed. Materials tested are ranging from regular pharmaceuticals to insects, nano-materials, bacteria, exclusive herbs and other high-end food ingredients. Recent developments are aiming to convert this unique technology into a continuous production process, allowing a further increase of capacity.