Freeze-Drying of Foods

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Contributor: Ewa Jakubczyk

Freeze-drying is a process in which water is sublimated by the direct transition of water from solid (ice) to vapor, thus omitting the liquid state, and then desorbing water from the “dry” layer.

Keywords: lyophilization ; freeze-drying ; food

1. Introduction

Freeze-drying is a process in which water is sublimated by the direct transition of water from solid (ice) to vapor, thus omitting the liquid state, and then desorbing water from the “dry” layer [1][2][3][4][5]. It is widely used for the stabilization of high-quality food, biological materials, and pharmaceuticals, such as proteins, vaccines, bacteria, and mammal cells. In the process, the quality of the dried product (biological, nutritional, and organoleptic properties) is retained [6][7]. This is due to the fact that freezing water in the material prior to lyophilization inhibits chemical, biochemical, and microbiological processes. Therefore, the taste, smell, and content of various nutrients do not change. Raw food materials contain a lot of water, ranging from 80% to 95%. The removal of water by sublimation results in the creation of highly porous structure of the freeze-dried products, and the rehydration of lyophilisates occurs immediately [8][9].

The water in the products can be free water or water bound to the matrix by various forces. Free water freezes, but bound water does not freeze. In the freeze-drying process, all ice water and some bound water must be removed. Therefore, lyophilization is a highly complex and multi-step process that consists of [1][6]:

- The freezing of the product, most often under atmospheric pressure.
- Primary drying—proper freeze-drying—ice sublimation, most often at reduced pressure.
- Secondary drying—desorption drying—drying the product to the required final humidity.

The effect of freeze-drying should be considered from the economic aspect and the quality of the freeze-dried material. The cost of a product mainly depends on the freeze-drying time. Therefore, process parameters and other conditions of its course are often set so that its time is as short as possible. Setting parameters to speed up the process can lead to the deterioration of the product’s properties. For example, increasing shelf temperature can lead to the defrosting of the product and the collapse of the structure or to the thermal degradation of heat-sensitive food ingredients.

The conditions of the freeze-drying process should be selected in a way that does not melt the water. Liquid water is the reaction medium and changes the rheological properties of the product. The presence of liquid water during the freeze-drying of food products may result in many changes in the composition, morphology, and physical properties of foods (e.g., shrinkage). It may also reduce the period of ensuring high quality during storage [10]. The color and structure–texture properties are crucial in the quality evaluation of food by consumers. Therefore, the dependence of these food properties on the parameters of freeze-drying is extremely important.

The effect of freeze-drying conditions on the nutritional properties, antioxidant activities [11][12][13][14], and glass transition characteristics [12][15][16] of different food materials can be found in the literature. It is widely believed that freeze-drying is the best method of drying. However, improperly selected process parameters may cause unfavorable changes in the material, such as shrinkage, color change, collapsed structure. Therefore, the aim of this review was to characterize all stages of the freeze-drying process, discuss the phenomena taking place during those stages, present their impact on the course of the process, and explain the effect of the process conditions on the selected physical properties of different food products.

2. The Characteristics of the Freeze-Drying Process

During the three stages of the freeze-drying process (sublimation, primary drying, and secondary drying), six main physical phenomena can be distinguished that have a significant impact on the course of the process, the quality of the obtained material, and the overall costs of the process. Those are:

- The phase transition of the water contained in the product into ice.
- The ice to vapor phase transition.
- The desorption of water molecules from material structures.
- The obtainment of a sufficiently low pressure.
- The re-sublimation of water vapor removed from the material on the surface of the condenser.
- The removal of a layer of ice from the surface of the capacitor.

Both the kinetics of the process and the properties of the obtained product depend on the parameters in which these phenomena occur.

The main feature of freeze-drying, the only one that distinguishes it from vacuum drying, is the need to keep free water frozen. This is one of the most difficult problems in freeze-drying.

Freeze-drying is a mass exchange process that requires heat transport. The heat of sublimation is 2885 kJ/kg [17][18]. If too little heat is supplied, the process will be slow, which will increase its costs. If the supplied heat flux is too high, it will cause an accumulation of heat in the material and an increase in its temperature, consequently leading to the possibility of the appearance of liquid water. Hence, it is extremely important to maintain a balance between the amount of heat supplied and used. One way to assess whether the amount of heat supplied is too high is to monitor the temperature of the lyophilized material [19]. Its value must not exceed the value of the cryoscopic temperature for a given material or the glass transition temperature for a given water content. If the glass transition temperature is exceeded, the structure may collapse (porosity reduction), which is highly disadvantageous due to the reduction of the specific surface of the product. As a consequence, the time of the second drying stage lengthens, the rehydration capacity of the product deteriorates, the product has a higher final water content [20]. Moreover, it may result in lower product stability during storage.

Maintaining a constant, low temperature (according to the pressure in the chamber) during the sublimation period proves that the balance between the amount of heat supplied and used for sublimation is maintained. However, it does not mean that the process runs at the maximum possible sublimation rate under given conditions. Too low a value of the supplied heat flux may limit the sublimation rate. On the other hand, an increase in temperature may indicate that the heat input is too high. It can be also the effect of the possible heat consumption by sublimation due to increase of the heat transport resistance. Therefore, for more complete control, changes in the water content should also be monitored simultaneously [19].

The parameter that determines the amount of heat supplied is the heat transfer resistance, while the resistance to mass movement (water vapor), both inside and outside the material, determines the amount of heat used for evaporation. Therefore, the course of lyophilization is determined by all the factors affecting the value of both these resistances. These factors are related to the process parameters at each stage, the design of the freeze-dryer, and the properties of the lyophilized material.

3. Summary and Conclusions

The literature on the analysis of the phenomena occurring during the entire freeze-drying process comes mainly from the area of pharmacy. Maintaining the biological activity of this group of products, both after the process and after the storage period, is the overriding goal. Therefore, many studies concerning the influence of the individual stages of freeze-drying on the final activity of the product have been carried out. Therefore, a modern technologist should make every effort to preserve not only substances that are noticeable by the consumer but also vitamins and other bioactive substances, often very labile, that are degraded during processing. The many publications also showed the effect of process conditions on the physical properties of freeze-dried foods. This has been analyzed by some authors, mainly concerning the shrinkage and porosity of dried material. The texture of a freeze-dried product obtained at different parameters of the process, such as the shelf temperature, chamber pressure, and the freezing rate, were investigated in a small range. Texture is a major feature assessed by consumers. This was surprising because the textural properties of food are strongly linked with the sensory analysis of food products. More publications can be found in regard to the color of the dried material. This is also important because changes in color during the freeze-drying process provide some information on the degradation of bioactive compounds such as antioxidants.

It should be understood what changes a selected material is subject to at each stage of freeze-drying, and the parameters of each stage should be selected based on the specificity of a raw material. The selection of proper conditions for the freeze-drying of a food material should be performed based on the characteristics of raw materials, such as composition (water content, the presence of sugars, proteins, and bioactive compounds), type of material (tissue, liquid material, semi-liquid material, and gel), and the glass transition temperature of foodstuff.

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