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Preservation of some food and fruits Species through Freeze Dryer- A Review

Asanya Emeka[1], Audu Ibrahim Ali[2], Ezeigwe C P[3],
[1] Department of Mechanical Engineering, Enugu State University of Science and Technology, Enugu State, Nigeria
[2] Department of Mechanical Engineering, The Federal Polytechnic Idah, Kogi State, Nigeria
[3] Science Laboratory Technology, Federal Polytechnic Oko, Anambra State, Nigeria

Abstract
Freeze-drying is a method of dehydrating frozen materials by a process of sublimation under high vacuum. This drying method is known for its ability to sustain food quality during the low temperature drying due to the minimum loss of flavor and aroma, negligible shrinkage and the absence of water, which minimize the chance of microbial growth. It can also be used to preserve the animals in the museum to attract tourist from different part of the world, which will generate revenue for an individual, State or Federal Government. This may be used to keep these animals in their original form for latter consumption. The benefits of the process include extended shelf life and cheaper storage and transportation costs relative to the liquid form of the product. It is comprised of freezing, sublimation (primary drying), and desorption (secondary drying). Lyophilization stabilizes the drug in formulation by slowing down the degradation process. It modifies the nature of materials by eliminating the solvent component to level that it no longer supports chemical changes to occur. Solvent removal is accomplished by freezing the formulation and immobilizing any solvent in the interstitial region between the crystals.

Keywords: freeze drying, lyophilization, Preservation, Food and Fruits

Introduction
It is common knowledge that processing may partially or totally affect the quality of a food product (*Agnieszka et al, 20114)* [1]. Various changes may occur in physical, chemical and/or biological characteristics of animal and foodstuffs during processing, storage and distribution. The criterion of quality is becoming of increasing importance to consumers' choice. Thus, industrial products and ingredients are expected to offer various convenient properties (taste, health promotion, safety, *etc.*) that correspond to those of fresh products (*Agnieszka et al, 2011)* [1]. At the same time, new market demands are emerging that could concern freeze-dried products, animal, for example dehydrated fruits to be added to corn, flakes, cereal bars, ice cream, or pastry making. Freeze-drying (lyophilization) is a drying process in which the solvent (usually water) and/or the suspension medium is crystallized at a low temperature and thereafter sublimated from the solid state directly into the vapor phase [Liu *et al*., 2008] [33]. Freeze-drying has become one of the most important processes for the preservation of heat-sensitive biological material [George & Datta, 2002; Dincer, 2003; Liu *et al*., 2008] [23, 12, 33]. Today, the field of its applications ranges from relatively simple preservable food, over complex biotechnological or pharmaceutical products, to proliferating bacteria and fungi [Tsinontides *et al*., 2004] [40]. In addition to food products (coffee, tea, crispy fruits and vegetables, ingredients for ready-to-eat foods and some aromatic herbs) [Pan *et al*., 2008; Chan *et al*., 2009] [7], it is suitable for other goods, including: flowers, microorganisms, pharmaceuticals, medical devices, and cosmetics, specially chemicals and pigments, enzymes and ceramic powders [Liu *et al*., 2008] [33]. Although today a great variety of foods, pharmaceuticals, *etc.* are produced by lyophilization, a successful application for long-term preservation of a living system, like *e.g.* cells, is still one of the greatest challenges for scientists in this field [Ciurzyńska & Lenart, 2009] [8]. Freeze-drying has become a standard processing technique in the bio-industry sector, where it enables stable products of high quality to be manufactured. Yet until quite recently, no monograph has been published that would analyse various aspects, scientific, engineering, economic, and regulatory ones, which make effective freeze-drying such a complex operation [Franks, 2000] [18]. The popularity of freeze-drying is based on some well-known advantages compared to competitive processes: sample stability at a room temperature, the easy reconstitution by the addition of water, the defined porous product structure, the reduction in weight, and the possibility of easy sterile handling. Since these advantages often result in the saving of time or money, the demands to develop or improve freeze-drying processes for more complex systems are continuously increasing. With increasing complexity of the biological material (structure, activity, and metabolism), the prospects for a successful preservation by freeze-drying are decreasing [Rindler *et al*., 1999] [44].

Air-drying is an ancient process used to preserve foods in which the material to be dried is exposed to a continuously flowing hot stream of air where moisture evaporates. The phenomenon underlying this process is a complex problem involving simultaneous mass and energy transport in a hygroscopic, shrinking system. Air-drying offers dehydrated products with their shelf life being extended by a year, but the quality of a conventionally-dried product is usually drastically reduced compared to that of the original foodstuff [Ratti, 2001] [42, 45]. These changes involve physical alterations, chemical reactions and biochemical effects. Physical alterations include shrinkage, increased or decreased porosity, and decreased ability to bind water and damage to microscopic structure [Chirife & Buera, 1995; Stapelfeldt *et al*., 1997; Witrowa-Rajchert & Lewicki, 2006] [11, 48, 50].

Compared to conventional food preservation technologies (conventional drying and others), the key benefits of freeze-drying include the following: retention of morphological, biochemical, and immunological properties, high viability/ activity levels, lower temperature, and shear conditions compared with other drying methods, high recovery of volatiles, retention of structure, surface area, and stoichiometric ratios, high yield, long shelf life, and reduced weight for storage, shipping, and handling [Dincer, 2003] [12]. The product from freeze-drying should also be much crisper than that from hot air drying [Pan *et al*., 2008]. Despite unmatched advantages, freeze-drying has always been considered the most expensive operation for manufacturing a dehydrated product owing to high energy consumption and high costs of both operation and maintenance. The analysis of energy requirements for the conventional drying methods and freeze-drying have shown that the basic energy required to remove 1 kg of water is almost double for freeze-drying than for the conventional drying [Flink, 1977] [19]. In addition, compared with air-drying, the cost of freeze-drying, is 4–8 times higher [Ratti, 2001] [42, 45]. Minimizing energy losses in freeze-drying is of outmost significance, for a great energy input is needed in the process to achieve low temperatures and low pressures, as well as heating to meet the requirement of drying [Dincer, 2002]. Ratti [2001] [42, 45] illustrated that, among the freeze-drying operations, sublimation accounts for about 45% of the total energy consumption in a freeze-drying cycle, while the energy consumption of freezing is about 4% of the total. Conditions for low energy consumption and high productivity were investigated by many scientists.

Principles of Freeze Drying

The freeze drying process consists of three stages: prefreezing, primary drying, and secondary drying. Prefreezing: Since freeze drying is a change in state from the solid phase to the gaseous phase, material to be freeze dried must first be adequately prefrozen. The method of prefreezing and the final temperature of the frozen product can affect the ability to successfully freeze dry the material. Rapid cooling results in small ice crystals, useful in preserving structures to be examined microscopically, but resulting in a product that is more difficult to freeze dry. Slower cooling results in larger ice crystals and less restrictive channels in the matrix during the drying process. Products freeze in two ways, depending on the makeup of the product. The majority of products that are subjected to freeze drying consist primarily of water, the *solvent,* and the materials dissolved or suspended in the water, the *solute.* Most samples that are to be freeze dried are *eutectics* which are a mixture of substances that freeze at lower temperatures than the surrounding water. When the aqueous suspension is cooled, changes occur in the solute concentrations of the product matrix. And as cooling proceeds, the water is separated from the solutes as it changes to ice, creating more concentrated areas of solute. These pockets of concentrated materials have a lower freezing temperature than the water.

Although a product may appear to be frozen because of all the ice present, in actuality it is not completely frozen until all of the solute in the suspension is frozen. The mixture of various concentration of solutes with the solvent constitutes the eutectic of the suspension. Only when all of the eutectic mixture is frozen is the suspension properly frozen. This is called the *eutectic temperature.*

It is very important in freeze drying to prefreeze the product to below the eutectic temperature before beginning the freeze drying process. Small pockets of unfrozen material remaining in the product expand and compromise the structural stability of the freeze dried product.

The second type of frozen product is a suspension that undergoes *glass formation* during the freezing process. Instead of forming eutectics, the entire suspension becomes increasingly viscous as the temperature is lowered. Finally the product freezes at the *glass transition point* forming a vitreous solid. This type of product is extremely difficult to freeze dry.

Primary drying: Several factors can affect the ability to freeze dry a frozen suspension. While these factors can be discussed independently, it must be remembered that they interact in a dynamic system, and it is this delicate balance between these factors that results in a properly freeze dried product.

After prefreezing the product, conditions must be established in which ice can be removed from the frozen product via sublimation, resulting in a dry, structurally intact product. This requires very careful control of the two parameters, temperature and pressure, involved in the freeze drying system. The rate of sublimation of ice from a frozen product depends upon the difference in vapor pressure of the product compared to the vapour pressure of the ice collector. Molecules migrate from the higher pressure sample to a lower pressure area. Since pressure is related to temperature, it is necessary that the product temperature is warmer than the cold trap (ice collector) temperature. It is extremely important that the temperature at which a product is freeze dried is balanced between the temperature that maintains the frozen integrity of the product and the temperature that maximizes the vapor pressure of the product. This balance is key to optimum drying. The typical phase diagram shown in Figure 1 illustrates this point. Most products are frozen well below their eutectic or glass transition point (Point A), and then the temperature is raised to just below this critical temperature (Point B) and they are subjected to a reduced pressure. At this point the freeze drying process is started.

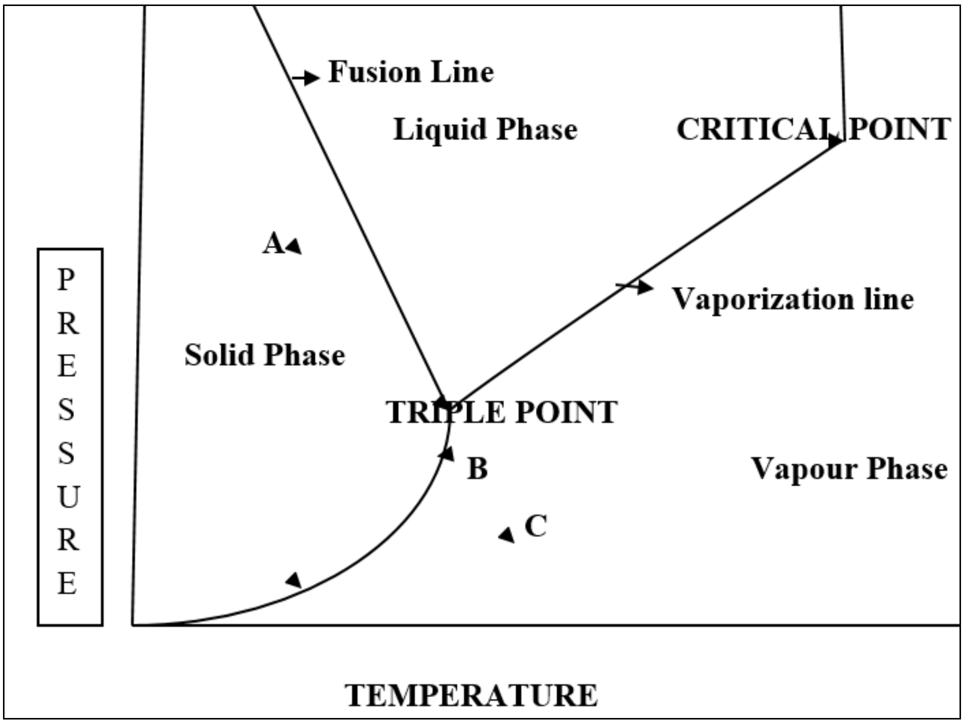

Figure 1: A Typical Phase Diagram (Labconco, 2004)

Some products such as aqueous sucrose solutions can undergo structural changes during the drying process resulting in a phenomenon known as *collapse*. Although the product is frozen below its eutectic temperature, warming during the freeze drying process can affect the structure of the frozen matrix at the boundary of the drying front. This results in a collapse of the structural matrix. To prevent collapse of products containing sucrose, the product temperature must remain below a critical *collapse temperature* during primary drying. The collapse temperature for sucrose is -32° C. No matter what type of freeze drying system is used, conditions must be created to encourage the free flow of water molecules from the product. Therefore, a vacuum pump is an essential component of a freeze drying system, and is used to lower the pressure of the environment around the product (to Point C). The other essential component is a collecting system, which is a cold trap used to collect the moisture that leaves the frozen product. The collector condenses out all condensable gases, i.e; the water molecules, and the vacuum pump removes all non-condensable gases.
Below is another detail phase diagram explaining the phase changes in lyophilisation

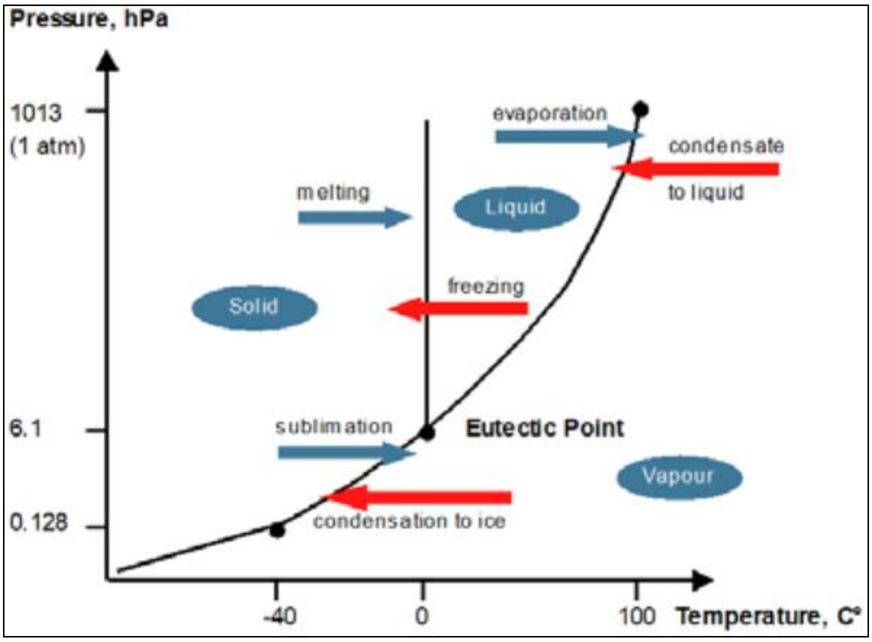

Figure 2: A Typical Phase Diagram (Labconco, 2004)

Primary drying

During the primary drying phase, the pressure is lowered (to the range of a few millibars), and enough heat is supplied to the material for the ice to sublime. The amount of heat necessary can be calculated using the sublimating molecules' latent heat of sublimation. In this initial drying phase, about 95% of the water in the material is sublimated. This phase may be slow (can be several days in the industry), because, if too much heat is added, the material's structure could be altered. In this phase, pressure is controlled through the application of partial vacuum. The vacuum speeds up the sublimation, making it useful as a deliberate drying process. Furthermore, a cold condenser chamber and/or condenser plates provide a surface(s) for the water vapour to re-solidify on. It is important to note that, in this range of pressure, the heat is brought mainly by conduction or radiation; the convection effect is negligible, due to the low air density.

Secondary drying

The secondary drying phase aims to remove unfrozen water molecules, since the ice was removed in the primary drying phase. This part of the freeze-drying process is governed by the material's adsorption isotherms. In this phase, the temperature is raised higher than in the primary drying phase, and can even be above 0 °C, to break any physico-chemical interactions that have formed between the water molecules and the frozen material. Usually the pressure is also lowered in this stage to encourage desorption (typically in the range of microbars, or fractions of a pascal). However, there are products that benefit from increased pressure as well.

After the freeze-drying process is complete, the vacuum is usually broken with an inert gas, such as nitrogen, before the material is sealed.

At the end of the operation, the final residual water content in the product is extremely low, around 1% to 4%.

How Freeze Drying Works

Refer to the phase diagram (Figure 1) and a typical sublimation cycle (Figure 2). The product is first cooled to below its eutectic temperature (Point A). The collector is cooled to a temperature approximately 20° C cooler than the product temperature, generally around -50 to -80° C. The product should be freeze dried at a temperature slightly lower than its eutectic or collapse temperature (Point B) since the colder the product, the longer the time required to complete primary drying, and the colder the collector temperature required to adequately freeze dry the product. After the product is adequately frozen and the collector temperature achieved, the system is evacuated using a vacuum pump (Point C). At this point, primary drying of the product begins and continues until the entire frozen matrix appears dry. Heat input to the product may be achieved by several means such as increasing the shelf temperature in the case of tray drying, or using a liquid bath for manifold drying. While the collector and vacuum pump create the conditions for allowing sublimation to occur, heat input is really the driving force behind the whole process.

Heat input to the sample can be enhanced by controlling the pressure in the system at some level above the ultimate capability of the vacuum pump. Some freeze dryers incorporate vacuum control systems that automatically regulate the pressure to the preset level. This allows additional gas molecules to reside in the system thereby improving the conduction of heat to the sample. This improves the sublimation rate, reducing process time and associated energy costs. Care must be taken to prevent the pressure within the system from exceeding the ice vapor pressure of the product or melting of the sample may occur.

Fig 3: A vacuum pump is essential to evacuate the environment around the product to be freeze dried (Labconco, 2004)

Application of Freeze drying

Freeze-drying (lyophilization), was developed to preserve bioactive molecules (DNA, enzymes, and proteins), pharmaceuticals products (antibiotics) and other delicate, solvent- -impregnated materials [Kusakabe & Kamiguchi, 2004] [31]. Freeze-drying is becoming an increasingly popular method for the long-term preservation of various biological materials. Although today a great variety of foods, pharmaceuticals, *etc.* are produced by lyophilization, a successful application for long-term preservation of living systems, like cells, is still one of the greatest challenges for scientists in this field [Rindler *et al*., 1998].

Continuous innovation and process optimization have increasingly led to more and more new applications of this process on an industrial scale [Rey & May, 2001] [43]. Freeze-drying appears, therefore, as a promising technique for dehydration of thermal-sensitive materials, such as fruits [Marques *et al*., 2007] [35]. Processing conditions have an influence on the quality criteria used to evaluate freeze-dried fruits, and on freeze- -drying time. In fact, two criteria (rehydration and texture) are closely linked with the ultimate use of the freeze-dried product and cannot be considered as absolute criteria. Dehydrated fruits are intended primary to be added to products such as corn flakes, cereal bars, ice cream, pastry sauces, *etc*. [Hammami & Rene, 1997] [27]. Also Krokida *et al*. [1998] have shown that physical properties of freeze-dried materials depend on temperature during freeze-drying. For all materials, collapse occurs above the glass transition temperature and the phenomenon becomes more intense as the temperature increases, controlling the bulk density and porosity of the dried materials. The freezing step explains the instantaneous rehydration capacity of the product, and the texture loss due to the cell wall damage. However, freezing rate has no significant effect on the quality of freeze-dried strawberry, nor on the freeze-drying time [Hammami & Rene, 1997] [27]. While, Gawałek [2005] [22] has shown that slower freezing of apples before the lyophilisation process results in an increase in the rate and amount of absorbed water, as well as in greater dry matter losses in the rehydration process. Also the density of the freeze-dried apples obtained decreased slightly along with an increase in freezing time, *i.e.* a slower freezing rate in a decrease in drying shrinkage. Freeze-drying including precrystallization and rapid freezing, allows the functional properties of egg yolk, without ingredients, to be preserved. Since full contact rapid freezing makes it possible without liquid nitrogen, freeze-dried egg yolk can be produced even at marketable costs [Jaekel *et al*., 2008]. Investigations conducted by Marques *et al.* [2007] [35] have shown that in the freeze-drying of acerola, fast pre-freezing of the material contributes to the preservation of the original porous structure of the product and results in a powder material being little susceptible to degradation reactions. From the techniques tested by these authors, cryogenic freezing using N_2 has been found the most recommended technique for freezing the samples. Freeze-dried acerola fruits were characterized by minimum shrinkage. In addition, their rehydration capacity was high, because the samples did not suffer cellular rupture. With their values thus preserved, the freeze-dried acerola can be considered a good source of vitamin C [Marques *et al*., 2007] [35]. High rehydration capacity restricts the application of freeze-dried strawberry in liquid carriers because the texture of such dried strawberries may collapse. Therefore, a suitable coating needs to be developed to slow the rehydration rate of freeze-dried strawberries in order to maintain their taste over a long period of time. It was shown that a whey protein coating solution could be used to reduce the rehydration rate of freeze-dried strawberry pieces because of its physicochemical properties [Huang *et al*., 2009] [26]. For osmotically-dehydrated freeze-dried strawberries a decrease of rehydration (Figure 2) and sorption capacity in relation to freeze-dried fruit not subjected to osmotic dehydration was noticed [Ciurzyńska & Lenart, 2010] [10]. Freeze-dried fruit after previous osmotic pre-treatment were characterised by lower water contents after 120 min of rehydration than fruit not subjected to osmotic dehydration. It was also noted that osmotic dehydration in starch syrup caused a significant difference in rehydration in relation to the analogical process conducted in sucrose and glucose solution [Ciurzyńska & Lenart, 2010] [10]. The rehydration and sorption properties were related to structural changes in freeze-dried strawberries during osmotic dehydration and freeze-drying (Figure 3) [Ciurzyńska & Lenart, 2010] [10].

Freeze drying of food

The primary purpose of freeze drying within the food industry to extend the shelf-life of the food while maintaining the quality *(Ratti, 2008)* [9, 41]. Freeze-drying is known to result in the highest quality of foods amongst all drying techniques because structural integrity is maintained along with preservation of flavors. Because freeze drying is expensive, it is used mainly with high-value products. (*Ratti, 2001)* [42, 45] Examples of high-value freeze-dried products are seasonal fruits and vegetables because of their limited availability, coffee, and foods used for military rations and/or hikers (*Ratti, 2001)* [42, 45].

NASA and Military Rations

Because of its light weight per volume of reconstituted food, freeze-dried products are popular and convenient for hikers, as military rations, or astronaut meals. A greater amount of dried food can be carried compared the same weight of wet food. In replacement of wet food, freeze dried food can be easily be rehydrated with water if desired and shelf-life of the dried product is longer than fresh/wet product making it ideal for long trips taken by hikers, military personal or astronauts. The development of freeze drying increased meal and snack variety to include items like shrimp cocktail, chicken and vegetables, butterscotch pudding, and apple sauce (NASA, 2018) [27].

Coffee

Coffee contains flavor and aroma qualities that are created due to Maillard reaction during roasting (Dong *et al*, 2017) [15] and can be preserved with freeze-drying (Peter, 2017).Compared to other drying methods like room temperature drying, hot-air drying and solar drying, Robusta coffee beans that were freeze-dried contained higher amounts of essential amino acids like leucine, lysine and phenylalanine (Dong *et al*, 2017) [15]. Also few non-essential amino acids that significantly contributed to taste were preserved (Dong *et al*, 2017) [15].

Fruits

Freeze drying can be beneficial for fruit as it can make them available year-round and can preserve their taste and color, making them more similar to the fresh fruit than other dehydration methods (Serna Cork *et al*, 2014). With conventional dehydration, berries can degrade in quality as their structure is very delicate and contains high levels of moisture. Strawberries were found to have the highest quality when freeze dried; retaining color, flavor and ability to be re-hydrated (*Shishehgarha et al, 2002)* [47]. Additionally freeze-dried fruit can be powdered to make it more soluble and act as bases for food products, as well as being a natural source of antioxidants and coloring (Serna Cork *et al*, 2014).

Advantages of freeze drying

Freeze-drying is viewed as the optimal method of choice for dehydration because of the preservation of quality, meaning characteristics of the food product such aroma, rehydration, bioactivity, are noticeably higher compared to foods dried from other techniques (Ratti 2008) [9, 41].

Shelf-life extension

Shelf-life extension is a result from low processing temperatures in conjunction with rapid transition of water through sublimation. With these processing conditions, deterioration reactions, including non enzymatic browning, enzymatic browning, protein denaturation, are minimized. When the product is successfully dried, packaged properly, and placed in ideal storage conditions the foods have a shelf life of greater than 12 months (Peter 2017).

Rehydration

If a dried product can not be easily or fully rehydrated, it is considered to be of lower quality (Ratti 2001) [42, 45]. Because the final freeze dried product is porous, complete rehydration can occur in the food. This signifies greater quality of the product and makes them ideal for ready-to-eat instant meals (Ratti 2001) [42, 45].

Effect on nutrients and sensory quality

Due to the low processing temperatures and the minimization of deterioration reactions, nutrients are retained and color is maintained (Peter 2017). Freeze-dried fruit maintains it original shape and has a characteristic soft, crispy texture.

Equipment and types of freeze dryers

There are many types of freeze-dryers available, however, they usually contain a few essential components. These are a vacuum chamber, shelves, process condenser, shelf-fluid system, refrigeration system, vacuum system and control system.

Function of essential components

Chamber

The chamber is highly polished and contains insulation, internally. It is manufactured with stainless steel and contains multiple shelves for holding the product (*Nireesha et al 2013)* [39]. A hydraulic or electric motor is in place to ensure the door is vacuum-tight when closed.

Process Condenser

The process condenser consists of refrigerated coils or plates that can be external or internal to the chamber. During the drying process, the condenser traps water. For increased efficiency, the condenser temperature should be 20°C less than the product during primary drying (*Nireesha et al 2013)* [39] and have a defrosting mechanism to ensure that the maximum amount of water vapor in the air is condensed.

Shelf Fluid

The amount of heat energy needed at times of the primary and secondary drying phase is regulated by an external heat exchanger. Usually, silicone oil is circulated around the system with a pump.

Refrigeration System

This system works to cool shelves and the process condenser by using compressors or liquid nitrogen, which will supply energy necessary for the product to freeze.

Vacuum System

During the drying process, a vacuum of 50-100 microbar is applied, by the vacuum system, to remove the solvent (*Nireesha et al 2013)* [39]. A two-stage rotary vacuum pump is used, however, if the chamber is large then multiple pumps are needed. This system compresses non-condensable gases through the condenser.

Control System

Finally, the control system sets up controlled values for shelf temperature, pressure and time that are dependent on the product and/or the process. The freeze-dryer can run for a few hours or days depending on the product.

Contact Freeze Dryers

Contact freeze dryers use contact (conduction) of the food with the heating element to supply the sublimation energy. This type of freeze dryer is a basic model that is simple to set up for sample analysis. One of the major ways contact freeze dryers heat is with shelf-like platforms contacting the samples. The shelves play a major role as they behave like heat exchangers at different times of the freeze-drying process. They are connected to a silicone oil system that will remove heat energy during freezing and provide energy during drying times *(Nireesha et al 2013)* [39].

Additionally, the shelf-fluid system works to provide specific temperatures to the shelves during drying by pumping a fluid (usually silicone oil) at low pressure. The downside to this type of freeze dryer is that the heat is only transferred from the heating element to the side of the sample immediately touching the heater. This problem can be minimized by maximizing the surface area of the sample touching the heating element by using a ribbed tray, slightly compressing the sample between two solid heated plates above and below, or compressing with a heated mesh from above and below.

Radiant freeze dryers

Radiant freeze dryers use infrared radiation to heat the sample in the tray. This type of heating allows for simple flat trays to be used as an infrared source can be located above the flat trays to radiate downwards onto the product. Infrared radiation heating allows for a very uniform heating of the surface of the product, but has very little capacity for penetration so it is used mostly with very shallow trays and homogeneous sample matrices (Peter 2017).

Microwave-assisted freeze dryers

Microwave-assisted freeze dryers utilize microwaves to allow for deeper penetration into the sample to expedite the sublimation and heating processes in freeze-drying. This method can be very complicated to setup and run as the microwaves can create an electrical field capable of causing gases in the sample chamber to become plasma. This plasma could potentially burn the sample, so maintaining a microwave strength appropriate for the vacuum levels is imperative. The rate of sublimation in a product can affect the microwave impedance, in which power of the microwave must be changed accordingly (Peter 2017).

Conclusion

Freeze Drying has become an accepted method of processing heat sensitive products that require long term storage at temperatures above freezing. In other words, Freeze Drying is a dehydration technique. The aspect of the Freeze Drying process that makes it different from other dehydration techniques, is that dehydration takes place while the product is in a frozen state and under a vacuum. These conditions stabilize the product, minimizing the effects of oxidation and other degradation processes. The unique advantage of Freeze Drying is that the samples are kept at low temperatures and remain frozen during the entire drying process, thereby preserving thermo labile components (proteins, flavors, colors), all while maintaining the original shape and size. The dried product can then be stored for long periods without the risk of changing composition (i.e. enzymatic, genetic) or being infected by microorganisms, which is all made possible due to the lack of water.

References

1. Agnieszka Ciurzyńska, Andrzej Lenart. Freeze-Drying – Application in Food Processing and Biotechnology – A Review, Polish Journal of Food Nutrition Science, 2011.
2. Ammu K, Radhakrishna K, Subramanian V, Sharma TR, Nath H. Storage behaviour of freeze-dried fruit juice powders.m *J. Food Technol*,1977:12:541-554.
3. Babic J, Cantalejo MJ, Arroqui C. The effect of freeze-drying process parameters on Broiler chicken breast meat. Food Sci. Technol,2009:42:1325-1334.
4. Banjo AD, Lawal OA, Songonuga EA. The nutritional value of fourteen species of edible insects in southwestern Nigeria. Afr. J. Biotechnol,2006:5:298-301.
5. Bird K. Palatability of freeze-dried meats. Food Technol,1965:19:55.
6. Bourdoux, Siméon Li, Dan Rajkovic, Andreja Devlieghere, Frank Uyttendaele, Mieke. "Performance of Drying Technologies to Ensure Microbial Safety of Dried Fruits and Vegetables". Comprehensive Reviews in Food Science and Food Safety,2016:15(6):1056-1066. doi:10.1111/1541-4337.12224. ISSN 1541-4337.
7. Chan EWC, Lim YY, Wong SK, Lim KK, Tan SP, Lianto FS *et al*. Effect of different drying methods on the antioxidant properties of leaves and tea of ginger species. Food Chem,2009:113:166-172.

8. Ciurzyńska A, Lenart A. Colour changes of freeze-dried strawberries osmotically dehydrated before drying., Food Technology Operations new vistas Monograph, 2009, 217-223.
9. Ratti Cristina. Advances in Food Dehydration. CRC Press, 2008, 209-235. ISBN 9781420052534.
10. Ciurzyńska A, Lenart A. Rehydration and sorption properties of osmotically pretreated freeze-dried strawberries. J. Food Eng,2010:97:267-274.
11. Chirife J, Buera MP. A critical review of some nonequilibrium situations and glass transitions on water activity values of foods in the microbiological growth range. J. Food Eng,1995:25:531-552.
12. Dincer I. Refrigeration Systems and Applications., John Wiley & Sons, England, 2003, 534-536.
13. Di Matteo P, Donsi G, Ferrari G. The role of heat and mass transfer phenomena in atmospheric freeze-drying of foods in a fluidised, 2003.
14. Defense.gov News Article New Rations in Pipeline for Service Members". *archive.defense.gov*. Retrieved, 2018.
15. Dong, Wenjiang Hu, Rongsuo Chu, Zhong Zhao, Jianping Tan, Lehe. Effect of different drying techniques on bioactive components, fatty acid composition, and volatile profile of robusta coffee beans". Food Chemistry, 2017, (234).
16. Ellab white Paper The freeze drying theory and Process, 2018.
17. Euromonitor International Consumer Lifestyles in Kenya, 2017. Available online: http://www.euromonitor.com/ consumer-lifestyles-in-kenya/report (accessed on 29 May 2017).
18. Franks F. Freeze-drying/lyophilisation of pharmaceutical and biological products. Cryobiology,2000:40:381-382.
19. Flink JM. Energy analysis in dehydration processes. Food Technol,1977:31:76-83.
20. Forkwa Tengweh Fombong, Mik Van Der Borght, and Jozef Vanden Broeck Influence of Freeze-Drying and Oven-Drying Post Blanching on the Nutrient Composition of the Edible Insect Ruspolia differens, Insects,2017:8:102.
21. Food Process Engineering and Technology, FPET, 2013. DOI: http://dx.doi.org/10.1016/B978-0-12-415923-5.00023-X © 2013 Elsevier Inc. All rights reserved.
22. Gawałek J. Effect of convection and freeze drying conditions on the quality of dried carrot roots. Inż. Roln,2005:71:119-127 (in Polish).
23. George JP, Datta AK. Development and validation of heat and mass transfer models for freeze-drying of vegetables slices. J. Food Eng,2002:52:89-93.
24. Hassan M, Ahmed J. Physico-chemical and sensory characteristics of mango-milk beverage. *Ind. Food Packer*,1998:52:32-36.
25. Henry BS. Natural Food Colours. In: Houghton, Natural Food Colourants, G.A.F, 1996.
26. Huang L, Zhang M, Wei-qiang Y, Mujumdar AS, Sun. Effect of coating on post-drying of freeze-dried strawberry pieces. J. Food Eng,2009:92:107-111.
27. Hammami C, Rene F. Determination of freeze-drying process variables for strawberries. J. Food Eng,1997:32:133-154.
28. Hymavathi TV, Khader V. Carotene, ascorbic acid and sugar content of vacuum dehydrated ripe mango powders stored in flexible packaging material. J. Food Comp. Anal,2005:18:181-192.
29. Jayathilakan K, Khudsia Sultana, Pandey MC, Radhakrishna K. Effect of freeze-drying and storage on β-carotene and ascorbic acid stability of mango milk shake, *Intl. J. of Food. Ferment. Technol*, 2012.
30. Kinyuru JN, Kenji GM, Simon N, Muhoho MA. Nutritional potential of longhorn grasshopper (Ruspolia differens) consumed in Siaya district, Kenya. J. Agric. Sci. Technol,2010:12:32-46.
31. Kusakabe H, Kamiguchi Y. Chromosomal integrity of freeze dried mouse spermatozoa after 137Cs gamma-ray irradiation. Mutat. Res,2004:556:163-168.
32. Labconco. A guild to freeze drying for laboratory, Labconco Corporation, USA, 2004.
33. Liu Y, Zhao Y, Feng X. Exergy analysis for a freeze-drying process. Appl. Thermal Eng,2008:28:675-690.
34. Maugey M, Neri W, Zakri C, Derré A, Pénicaud A Noé L *et al*. "Substantial improvement of nanotube processability by freeze-drying". J Nanosci Nanotechnol,2007:7(8):2633-2639. doi:10.1166/jnn.2007.855
35. Marques LG, Ferreira MC, Freire JT. Freeze-drying acerola (Malpighia glabra L.). Chem. Eng. Process,2007:46:451-457.
36. Meissner U, Stahl Harald, Steinkellner D. "Detection of Silicone Oil Leakages in Freeze Dryers". PDA Journal of Pharmaceutical Science and Technology,2011:65(5):481-485. doi:10.5731/pdajpst.2011.00748. ISSN 1079-7440. PMID 22293837.
37. *NASA Food for Space Flight"), 2018. www.nasa.gov. SpaceFlight:JSC*. Retrieved 2018-05-18.
38. NASA Food Technology: Incredible Edibles from Space.
39. Nireesha GR, Divya L, Sowmya C, Venkatesan N, Niranjan Babu M, Lavakumar V. "Lyophilization/Freeze Drying - An Review". International Journal Of Novel Trends In Pharmaceutical Sciences, 2013, 3.
40. Tsinontides SC, Rajniak P, Pham D, Hunke WA, Placek J, Reynolds SD. Freeze drying-principles and practice for successful scale-up to manufacturing. Int. J. Pharm,2004:280:1-16.
41. *Ratti Cristina*. Advances in Food Dehydration. CRC Press, 2008, 209-235. ISBN 9781420052534
42. Ratti C. "Hot air and freeze-drying of high-value foods: a review". Journal of Food Engineering,2001:49:311-319.

43. Rey L, May J. Freeze-drying/Lyophilization of pharmaceutical and biological products. Book reviews. Eur. J. Pharm. Biopharm,2001:51:163-164.
44. Rindler V, Lüneberger S, Schwindke P, Heschel I, Rau G. Freeze-drying of red blood cells at ultra-low temperatures. Cryobiology,1999:38:2-15.
45. Ratti C. Hot air and freeze-drying of high-value foods: a review. J. Food Eng,2001:49:311-319.
46. Serna-Cock, Vargas-Munoz, Diana Patricia, Aponte, Alfredo Ayala "Structural, physical, functional and nutraceutical changes of freeze-dried fruit". African Journal of Biotechnology,2015:14(6):442-450. doi:10.5897/ajb2014.14189. ISSN 1684-5315.
47. Shishehgarha F, Makhlouf J, Ratti C. "Freeze-Drying Characteristics Of Strawberries". Drying Technology – via Academic Search Premier,2002:20(1):131-145
48. Stapelfeldt H, Nielsen BR, Skibsted LH. Effect of heat treatment, water activity and storage temperature on the oxidative stability of whole milk powder. Int. Dairy J,1997:7:331-339.
49. Steinbach G. Equations for the Heat and Mass Transfer in Freeze-Drying of Porous and Non-Porous Layers and Bodies, 1971, 674-683. Washington, DC: International Institute of Refrigeration.
50. Witrowa-Rajchert D, Lewicki PP. Rehydration properties of dried plant tissues. Int. J. Food Sci. Technol,2006:41:1040-1046.
51. World Health Organization. Global prevalence of Vitamin A deficiency in populations at risk 1995-2005. WHO database on Vitamin A deficiency, 2009. Available on www.who.int/nutrition/ publications/micronutrients/vitamin _a deficiency/ WHO_NUT_95.3/end/index.html. Accessed on 15 December 2010.
52. Zhai S, Taylor R, Sanches R, Slater NKH. Measurement of lyophilisation primar drying rates by freeze-drying microscopy. Chem. Eng. Sci,2003:58:2313-2323. bed. J. Food Eng,2003:59:267-275.