Review

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Posted Date: 13 September 2024

doi: 10.20944/preprints202409.0950.v1

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 doi:10.20944/preprints202409.0950.v1

Review

The Impact of Freeze Drying on Bioactivity and Physical Properties of Food Products

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Abstract: Freeze drying (FD) is a technique used to preserve the bioactive content and flavor of samples. Foods with a short shelf life due to nature have been preserved by FD in recent years. The success of the drying technique is thanks to the high retention ability of the bioactive compounds and the flavor of the sample. However, the high cost and energy consumption of the FD limit its usage in several fields. Freezing, primary drying, and secondary drying, which take place in the same device, are the stages of the FD. In addition, several parameters, including pretreatment, size, temperature, sample species, time, and pressure, affect the FD process. These parameters are controlled to increase the effectiveness of the FD on the samples. Moreover, FD was compared with other drying techniques including hot-air drying, microwave drying, vacuum drying, and solar drying to determine its protective capability. It has also been applied to the valorization of waste products, which is a common problem worldwide. For valorization, microencapsulation and powder production have been achieved by FD. In recent years, studies on the effects and use of the FD on various materials increased, providing new aspects for the future of science and food industry.

Keywords: freeze drying; physical property; bioactive property; flavor; waste by-product

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1. Introduction

For centuries, various techniques, including fermentation and drying, have been applied by people to preserve foods that require protection from the degradation of their bioactive and physical properties [1,2]. With increasing demands in the consumption and preparation of functional foods and their derivatives, drying has become essential to ensure the preservation of bioactive components of foods, especially throughout their shelf life [3–5]. Drying has been beneficial for centuries in protecting food and enabling long-term storage, especially in the science and food industry [6,7]. In another words, drying ensures that the bioactive content and flavor of the foods are conserved over a long time. [8,9]. In recent years, one of the newly applied drying techniques is the freeze drying (FD) technique, which has been used to maintain the quality of products by preserving the physical and bioactive characteristics of the products [10,11].

For a long while, foods have been used as spread product ingredients in several areas, including medicine and industrial manufacturing [12–14].Foods were dried using different techniques, primarily to enhance their long-term storage potential while minimizing the decomposition of their bioactive components [15,16]. Hot air drying, sun drying, and microwave drying are frequently used drying techniques in several industries and laboratories to produce high-quality dried materials [17]. Recently, FD has become popular in studies aiming to obtain maximum product quality from the samples [18]. Even though FD successfully acts on the prevention of the bioactive property, physical property, and flavor of the material, it also involves high costs and significant energy consumption due to the low temperature and pressure required [19]. Low temperature and pressure can be a source of the high product quality of the freeze-dried samples.

Freezing, primary drying, and secondary drying are the main processes required to achieve the maximum product quality in FD [20,21]. In the application of the FD technique, these processes are

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pursued, respectively. The duration of each drying process is crucial for managing energy consumption and cost. Also, FD is used for the conversion of waste products, which is an enormous problem for the global environment and cost [22]. Waste products, including peels, seeds, and pomace, with FD, transformed the effective materials, including microencapsulated bioactive content and powder, with high bioactive ingredients, to enhance the quality of the other products that are manufactured in the industry [23].

This review article examines the preservation effects of the FD on bioactive components of food products. It has been investigated how the parameters influencing freeze-drying, impact the bioactive content and flavor properties of the products [24]. Additionally, the freeze-drying of plants and foods, including fruits, vegetables, and other food products, as well as waste and by-products, is mentioned [25–31].

2. Freeze Dry Technique

Lyophilization, also known as freeze drying, is one of the widely used drying techniques for the preservation and production of foods in the food industry, especially in recent years, as well as for the production of nutraceuticals and pharmaceuticals in the pharmaceutical industry and medicine [32,33]. Although FD is a technique with disadvantages, including high energy consumption and cost, it can preserve the biological and physical contents of various food products, especially vegetables, fruits, and even food waste and by-products [34–36]. This technique, which is used in the preservation and storage of seasonal fruits, vegetables, and perishable foods, also preserves the sensory properties of these products [37]. These features popularize the use of the technique and expand its application area. FD consists of three main steps: freezing, primary drying, and secondary drying [38]. However, this process and its results are improved by integrating different techniques and devices into the freeze-drying process. Depending on the product to be dried, freeze-drying is combined with various pre-treatments, including pulsed electric field, ultrasound, blanching, and high hydrostatic pressure [39–41].

In FD, ice crystals formed during the freezing process are directly converted to water vapor and then condensed and stored as a liquid in another area of the freeze dryer. Enthalpy was explained as the amount of energy required for the sublimation of ice crystals to water vapor in FD [42]. It is calculated to determine the drying time and final product quality. It provides the administration of the expense and energy depletion, which forbids the usage of FD most frequently. In addition to these, the computation of enthalpy specifies the appropriate energy sources, the sublimation process time for ice crystals, and the quality of the final product.

Process of Freeze Drying Technique

The FD technique has processes such as freezing, primary drying, and secondary drying (Figure 1). The initial stage of FD is the freezing process, which is affected by manifold parameters to produce frozen materials at a special temperature for the used sample [43]. The parameters include a specific temperature, which varies depending on the content of protein, oil, and other components of the material that will be dried. Ice crystals formed during this process depend on the cooling rate, which affects the size and amount of ice crystals [38]. Additionally, the freezing and primary drying processes are influenced by the size and amount of the ice crystals, especially their application times [20]. The large and few ice crystals that are obtained by a slow cooling process, allow for rapid sublimation and reduce the drying time of FD [44]. Interestingly, the degradation of the cellular construction and cells of the material, which results from the large ice crystal formation, leads to an increase in the amount of some bioactive ingredients, which are present less in fresh food compared with freeze-dried material. In contrast, the application of rapid cooling causes the formation of smaller and larger quantities of ice crystals. This, in turn, leads to an increase in the time required for primary drying, and the amount of consumed energy by FD; however, it naturally reduce the degradation of the cellular structures of the material [45]. The extent of degradation is also heightened by rapid cooling, as this process prolongs the primary drying phase. However, the cellular structure remains intact.

Primary drying is the second main part of the FD technique. After freezing, ice crystals are sublimated directly to the vapor phase without passing through the liquid phase under low pressure and temperature [20,46]**.** As the processed food enters the liquid phase, a valuable portion of its components degrade. The temperature and pressure are determined at the triple point for sublimation. The sublimated vapor is converted to the solid phase, which is a part of the FD device, so the moisture content of the food decreases, but a small amount of the moisture is still in the food.

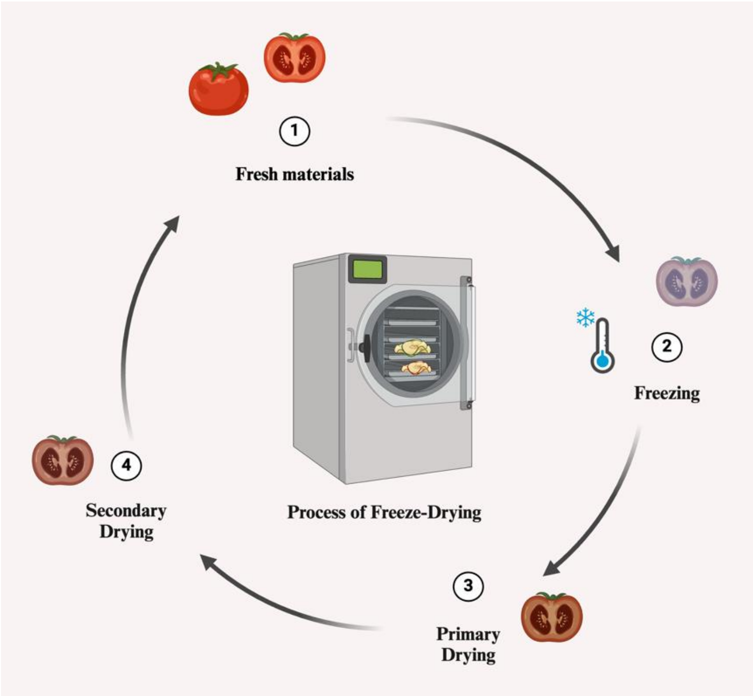

Figure 1. Process of freeze-drying technique.

The last part of the FD technique is secondary drying. In this process, the temperature is increased and the pressure is decreased for efficient drying [21]. Higher temperatures are not harmful to the food at this stage because there are no ice crystals present, so no liquid phase occurs [38,47]. The desorption term can be used for the secondary drying process. As a result of FD, 95-99.5% of the water in the food can be removed. Additionally, shorter drying times correlate with higher product quality.

FD can be enhanced by utilizing assisted technologies [48]**.** The importance of these technologies was demonstrated in an experiment by *Li* et al., where FD was compared with microwave-assisted FD [49]. Two different drying techniques were applied to the apple slices. In this study, vacuum microwave drying and FD were used as a hybrid system to obtain better results. Microwave-integrated FD was used to improve the efficiency of FD, reducing the drying time and promoting the nutritional value of the apple slices. Besides, greater puffing and better anti-moisture occlusion talents of the vacuum microwave and freeze hybrid dried apple slices were acquired, especially compared with apple slices that were freeze-dried alone.

Another technique is ultrasonic treatment, which can be used as an assistant to the FD [50]. Ultrasonically assisted atmospheric FD can be applied to achieve higher-quality products than non-assisted FD. In a study, dried button mushrooms were prepared with ultrasonically assisted atmospheric FD, and the study defined that the application of the assisted FD system improved the drying. Also, decreasing the drying time caused the reduction of the expense and energy depletion of the study. Results of the study demonstrated that ultrasonically assisted atmospheric FD was the best drying technique due to its drying rate with a shorter drying time and a nutritionally rich product. As an explanation, the biological and physical degradation of the sample was reduced by increasing the drying rate of FD. In contrast, the application of the ultrasonic power did not result in

 doi:10.20944/preprints202409.0950.v1

any significant change in the physical properties, especially texture, color, rehydration, or cell damage of the sample.

To improve the efficiency of FD, a dipping solution as an assistant to FD was applied during the drying processes. According to the research of *Cicerón Gonzâlez-Toxqui* et al., a dipping solution was applied to the FD processes to acquire a low drying time and energy depletion [51]. This application inhibited the drying time while protecting the bioactive properties of the sample. In addition, it used low temperatures, thus the cost of the study was decreased. Also in this study, coconut oil alkaline emulsion pretreatment was used to enhance the drying of the sample. As a result, the dipping solution and the pretreatment significantly reduced the energy consumption.

3. Parameters of freeze-drying

FD is impressed by several parameters, mainly containing temperature, length of the drying time, sample species, pretreatment, and pressure [52]. These parameters cause several results about the bioactive ingredients and flavor of material at the end of the FD processes. A wide variety of implications of the FD are exhibited stewardly to parameters (Figure 2).

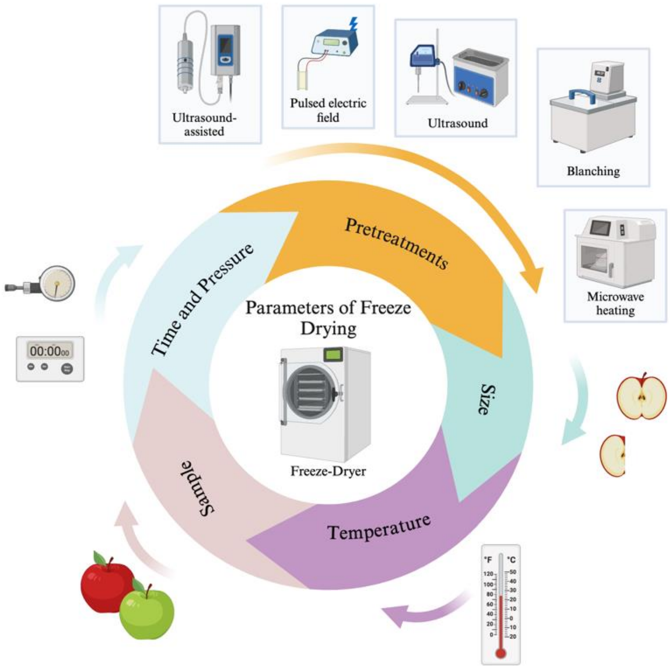

Figure 2. Parameters of freeze drying technique.

3.1. Pretreatments

Pretreatments include any technique used to enhance the preservation of the physical properties of the samples by increasing the bioactive properties of the samples and reducing the costs or achieving other desired outcomes [53]. In a study, blanching, ultrasound (US), and pulsed electric field (PEF) pretreatments were applied to improve the FD of red bell peppers [54]. As a consequence, these pretreatments similarly reduced the bioactive compounds' degradation of the sample and reduced the FD time. Results of the study showed that the blanching technique reduced the drying time and the amount of bioactive properties in the red bell pepper, while better preservation of the sample was observed when PEF and its combination with the US were applied. Hence, the protection of the product in all characteristics of the sample, including flavor, taste, color, and bioactive properties, was obtained by the appropriate choice of pretreatment techniques. In the FD application to the berries, fruits were used without cutting, peeling, or any physical changes to preserve their original shape and bioactivity; however, wrinkled products were obtained due to the outer skin barrier of the berries as an impediment to the flow of the water vapor [55]. Water vapor is generally produced during the sublimation period of FD. Also in this study, carbon dioxide (CO2) laser perforation was applied as a pretreatment to the skin of berries, which must be treated to obtain high-quality final products. The perforation improved the flow of the water vapor, which is produced by sublimation within the berries, and enhanced the drying processes. At the end of the study, the percentage of non-wrinkled products, which must be high to indicate that successful production occurred, increased from 47% to 86%. In other words, the product quality of the freeze-dried berries was improved with the perforation pretreatment. In brief, the low drying time and high product quality were obtained by pretreatment, which contributed to improving the FD of berries.

Ultrasound pretreatment was applied to explain its impact on the drying rate and rehydration rate of apple slices, and a positive correlation between FD and the pretreatment was examined [56]. The ultrasound pretreatment enhanced the product quality and improved the capacity of the apple slices to absorb water after FD. In another search about ultrasound by *N.C. Santos* et al., ultrasound pretreatment was applied for minutes to improve the product grade by increasing the bioactive properties, including the total phenolic compounds and antioxidant activity of the sample [57]. In this study, purple cabbage powder was prepared by FD with ultrasound pretreatment, and results obtained from the implementation of FD compared with convective, spouted bed, and spray drying according to the appearance of the dried powder. Consequently, convective drying, FD, and spouted bed drying generated erratic and rigid surfaces, while spray drying induced globular particulates.

Additionally, ultrasound-assisted FD was practiced to quince slices [39]. Freeze-dried samples that were exposed to ultrasound pretreatment showed a higher retention strength of bioactive properties, containing total phenols, antioxidant capacity, and ascorbic acid, of the slices compared to freeze-dried samples alone. Also, ultrasound pretreatment affected the physical properties, containing shrinkage, rehydration, color, water activity, and hardness of the freeze-dried samples, even more than dried samples without ultrasound pretreatment. Moreover, ultrasound was applied to improve the implications of FD on windfall apples [58]. The ultrasound application caused a high retention of the total phenolics, ascorbic acid, and antioxidant activity of the apples.

Two different pretreatments can be used as a hybrid to obtain maximum-quality products. For instance, a hybrid system of ultrasound pretreatment and blanching, which were applied before the FD processes, was used in a study [59]. As a result of the study, high retention of the total phenolics, total flavonoids, and quercetin and enhanced protection of the color of the sample were obtained by applying the hybrid system.

Another pretreatment for FD is high hydrostatic pressure (HHP) [60]. HHP was used to acquire improved results about the nutritional value and physical properties of carambola slices. As a result, HHP improved the retention of the total phenolic content, antioxidant activity, and other physicochemical properties of the slices by decreasing the drying time. However, HHP pretreatment is used less frequently by people who have limited budgets because of its high cost.

Also, in a recent study, three different plants-*Malva sylvestris* L., *Calendula officinalis* L., and *Asparagus officinalis* L.--were used to determine the effect of these novel pretreatment techniques on the FD. [61]. These samples were used by applying an infusion prepared from fresh plants to obtain the pretreated dried and non-pretreated dried samples. In conclusion, infusions of *Malva sylvestris* L. especially had higher amounts of polyphenols compared with other plant species. Also, the bioactive properties varied depending on the species and applied pretreatment to the samples. Interestingly, fresh infusions had lower amounts of bioactive properties, especially antioxidant activity and total polyphenols, compared to dried and pretreated freeze-dried infusions.

Furthermore, the FD technique was determined to be the best technique to preserve the bioactive properties of plants, suggesting that the impact of pretreatment was considerable by the end of the study. In the study by *M. Wang* et al., freeze-thaw pretreatment was applied to vacuum FD of goji berry (*Lycium barbarum* L.) to provide high retention of the bioactive properties of the berries during the drying processes [62]. The study determined that the pretreatment protected the physicochemical properties, including shrinkage and hardness of the berries, and conserved carotenoid content in the sample. Also, freeze-thaw pretreatment effectively decreased the drying time, energy consumption,

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and cost of FD compared to other traditional drying techniques. Another study investigated that the osmotic dehydration pretreatment was effective in the production of high-quality dried strawberries [63]. Osmotic dehydration improved the FD through its efficiency in decreasing the drying time and increasing the storage time and the product quality of the sample. As a result, this pretreatment increased the protection and retention of the color, hardness, total phenolic content, antioxidant activity, and vitamin C of strawberries.

3.2. Size

Large or small sizes, which are other parameters of FD, affect positively or negatively the drying time which impacts the protection of the bioactive and physical properties of the sample, as well as improves the quality of the final product. An experiment on blueberries found that the default moisture value of large berries was maintained for a longer time than that of small berries, and these results suggested that small samples led to a reduction in the drying time [64]. The outcomes provided that the sample size directly affects the drying time of the FD. Moreover, shorter drying times obtained by small samples resulted in higher biological value and more preserved polyphenolic compounds. Additionally, the decrease in the drying time reduced the cost of the technique.

In another study, different cuttings, including transverse and longitudinal cuts, were used to examine the effects of the size and shape of the ginger (*Zingiber officinale Roscoe*) on the drying processes and product quality [65]. Additionally, FD, hot air drying, vacuum drying, and catalytic infrared drying were applied to the ginger in this study. The study explained not only the effect of the size on the ginger quality in FD processes butalso determined the best drying technique to obtain the best dried ginger quality. The longitudinal cuts resulted in shorter drying times and better preservation of physical properties compared with other cuts. However, no change in the bioactive properties was observed when these cuts were applied to the ginger. Even though FD had a longer drying time compared to other techniques, it was chosen as the best technique among other drying techniques to obtain products that have maximum quality, especially in terms of the bioactive and physical properties. Because FD perfectly minimized the degradation of gingerol content, total phenolic, flavonoid content, and volatile compounds as bioactive properties of the sample. When longitudinal cut samples were dried by catalytic infrared drying, excellent results were obtained by reducing the drying time and developing the final product quality.

In some cases, the sample size may not be a determining factor in obtaining the maximum quality of freeze-dried products. For example, in the study of *Cristofel* et al., ginger was prepared as grated, sliced, and whole to determine the impact of the size of the sample on product quality and the drying rate of the FD [66]. Grating or slicing applications were ineffective in determining the effect of the sample size on the product quality compared with whole ginger.

3.3. Temperature

To obtain the improved final product, another parameter of the drying technique is temperature, which is determined specifically according to the application [7]. High temperatures cause color changes and increase the degradation of the bioactive properties of the sample. Also, various nucleation temperatures affect the drying time, thereby influencing product quality [43]. In addition, there are many results of the temperature difference, such as the size of pores used for the vapor flow of the sublimation process in FD and the rate that deals with the appearance of ice crystals. When a high cooling rate was used, a small pore size occurred. The nucleation temperature is an effective parameter that controls the structure of scaffolds for FD. In some cases, temperature may not significantly affect the drying process. For instance, in another study, no effect of temperature on product quality was observed in terms of alcohol-insoluble residue and water retention capacity [67]. Additionally, ultrasound was used to improve the drying rate, thereby decreasing the drying time.

3.4. Samples

The FD affects the product quality of the samples with its characteristic properties [68,69]. Various results were obtained by applying FD regarding the physical properties, metabolic processes, bioactive properties, and components of different samples [70,71]. The studies on the total phenolic content preservation of basil leaves (*Ocimum basilicum*) and purple carrots determined the effects of the sample species on the product quality of FD. The study concluded that an increase in the total phenolic compounds of basil leaves and a decrease in the total phenolic content of purple carrots were observed [72,73]. As a result, the FD process of the various samples showed different preservation levels of the bioactive contents and physical properties of the samples. For example, according to *R. Ma* et al., the internal structure and the number of main components of Gastrodia elata did not show any variation during FD [74]. However, in the research conducted by *C. Carrión* et al., FD changed the appearance, especially color and texture, and significantly preserved the amount of the bioactive content in button mushrooms [75].

In addition, samples that have different physical properties, including color, can cause unlike outcomes concerning physical and bioactive properties, after the FD process. For example, a study concluded that different degrees of whiteness as a result of FD when white and purple garlic samples were used [76]. The study also found that purple garlic slices showed better preservation ability on the bioactive properties including total phenolic content, antioxidant capacity, and allicin content, which are important for the valuable usage of garlic, compared with white garlic slices. Correlatively, in a study conducted by *Castro-mendoza* et al., FD of various sweet potato flours, including yellow and purple, yielded different implications, showing that yellow sweet potato exhibited better retention of the antioxidant capacity and polyphenol content of the sample than purple sweet potato [77]. Different degrees of retention may come from the divergent amounts of the bioactive ingredients, including raw fiber content and protein content. Also, oven drying was used for drying and showed low retention ability for bioactive compounds, especially the main polyphenols (coumaric acid) and caffeine, which were only detected in the yellow sweet potato after the drying.

3.5. Time and Pressure

In the research of *Nanchun Luo* et al., various ways were explained to decrease the drying time due to the high energy consumption and cost [78]. According to the article, a way among these was using pressure to decrease the drying time. As a result of this reduction, according to the article, pressure and time coordinatedly improved the dry matter quality. For example, in the study by *Suherman* et al., three different pressures and drying times were selected to determine the optimum conditions for achieving maximum quality in freeze-dried mango [79]. When the results of the study were evaluated, optimal values were determined as 13 hours of drying time and a pressure of 25 Pa. Additionally, in a study on orange puree, low pressure and processing time impacted the product quality positively due to preventing the degradation of the vitamin C, total phenols, β-carotene, antioxidant activity, and changes on the original color, porosity, mechanical properties, and water content of the sample [80]. In the research of *R. Pisano et. al.*, the pressure was used to determine the endpoint of the primary drying, which is an important process of the FD before the secondary drying [81]. The article explained how to find the most effective primary drying time with pressure to obtain the maximum product quality. Effective control of the drying time on the FD, especially, decreased the structure collapse and degradation of the bioactive properties of the sample. The pressure and time were determined for the stop system and applied to the primary drying step of the FD to facilitate the manufacture of a high-quality final product. This study concluded that pressure and time, which are parameters of the FD processes, must determined for effective usage of the FD to obtain maximum product quality.

4. Impact on Bioactive Properties

The nutritional value of samples and their shelf life can be preserved and increased by the FD technique [82,83]. It generally yields a high-quality product that retains several bioactive properties crucial for human health [84–86]. FD is an effective technique that uses low temperatures and

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especially low pressure, which varies among drying processes [38]. Compared to other techniques, lyophilization generally minimizes the degradation of bioactive components in the sample [87–89].

Milk and dairy products are the most widely consumed animal-based food category and are rich in nutrients [90–92] The various functions such as antimicrobial, antioxidant, neuroprotective, and other effects of products based on their bioactive components, including lactoferrin, growth factors, lactoperoksidase, and liposomes, etc. [93–95]. Additionally, the bioactive components can protect against various diseases [96–98]. This category appeals to all age groups, from childhood to old age, as well as athletes with special needs [99–101] Products including milk, cheese, yogurt, and colostrum are used as different foods or supplements processed by FD [102–104]. The effects of different techniques on milk and colostrum drying regarding content and digestibility are noteworthy issues [102].

Colostrum is a functional food known as the first milk produced by the mother after birth. This milk is produced between 0 and 7 days after birth [105]. Since the components in its content change every day, it is both remarkable and needs to be preserved very well [106]. At this point, drying techniques such as spray drying and FD are considered ideal techniques for colostrum samples. In a recent study conducted by *Fatima* et al., these two techniques were compared in terms of preserving the content of colostrum [107]. When the results were evaluated, it was determined that FD preserved the protein content, IgG and IgA content of colostrum better than spray drying.

In addition, new products are being created by combining milk and dairy products with various freeze-dried materials to create a wide range of products [108]. In particular, the combination of dried fruits and vegetables with widely consumed product was used to obtain health-supporting or health-enhancing properties in the production of functional foods [22,109].

Fruits are consumed outside of their seasons is an increasingly widespread manner. People use different production and preservation techniques to ensure this [110,111]. For this purpose, the preservation of the content, taste, and rheological properties of products is maintained by drying techniques applied according to samples [112,113]. At this stage, FD was applied to various species of fruits, and the results explained the efficiency of the FD compared with several drying techniques [114]. For example, FD and spray drying were applied to Cornelian cherry in a study to compare these drying techniques in terms of product quality [115]. Consequently, a greater preservation ability than that of spray drying regarding the total phenolic contents, antioxidant capacity, and anthocyanins of the sample was provided by FD. In another study on fruits, white mulberries were dried by FD and oven drying to preserve the fruits' quality after drying in terms of their bioactive ingredients and physical properties [7]. When compared with fresh samples, air drying produced better redness, and FD resulted in better lightness and yellowness as physical properties. Also, the L-ascorbic acid of the sample degraded minimally, and the highest total phenolics and antioxidant capacity as other bioactive properties were obtained by the application of FD. Similarly, maoberry fruits *(Antidesma bunius L.*) were used to determine the effects of FD and convective hot air drying in order to choose the best technique for the maximum quality of the final product when this was looked from divergent perspectives [8]. FD that used a lower temperature than convective hot air drying, effectively preserved ascorbic acid, total phenolics, total flavonoids, antioxidant activity, and total anthocyanins of the fruits, more effectively than convective hot air drying. In an experiment with orange puree dried only using the FD technique to assess the impact of the FD on the bioactive and physical properties of the orange puree [80]. A clear, yellowish, darker, and more saturated color was obtained and high porosity was maintained after FD of orange puree, while a slight decrease in the amounts of vitamin C, carotenoids, and phenols was obtained in the freeze-dried orange puree.

In the study by *J. Kowalska* et al., FD was used to obtain a high-quality final product, and it caused a slight degradation in vitamin C and polyphenolic content as bioactive properties and in the flavor, and texture values as physical properties of the fruits [116]. Not only any color alternative was not obtained at the end of the study, but also the high porosity of strawberries was observed. Moreover, FD was used with different drying techniques including hot-air drying, vacuum drying, ultrasound-assisted vacuum drying, and ultrasound-pretreated FD on goji berries in the study to separately determine their effects on the berries in terms of their bioactive properties [117]. For each drying

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technique, the determination of the total phenolic content, phenolic profile, antioxidant capacity, carotenoid profile, and color change of the sample was conducted to contrast the drying techniques for maximum product quality. The study showed that the best technique for obtaining a maximum-quality product in regardto the bioactive properties was hot air drying including the carotenoid profile, and antioxidant activity of the sample. Although better product quality was also provided by FD and ultrasound-assisted vacuum drying, the high cost and energy consumption of FD generally take second place in this technique.

Some cases show that FD is not the most effective technique to preserve the product quality, among other drying techniques. For example, *Qingyang* et al., determined that walnut kernels as a stone fruit provided a useful basis to compare FD with other drying techniques, including gradient hot drying and constant hot drying, and to determine the degradation of bioactive properties of the walnut kernels after FD [118]. As a result, gradient hot drying was chosen as the most effective technique to preserve the total phenols and antioxidant activity of walnut kernels.

Similar to fruits, drying techniques are also applied to vegetables to extend their shelf life and preserve their sensory and rheological properties [119–121]. As an example, in the research of *R.* Macura et al. both FD and air drying techniques were used to dry purple carrots to obtain a high-quality product [73]. At the end of the study, while FD did not cause any change in the carotenoid and anthocyanin content, air drying reduced the amount of carotenoids, anthocyanins, and polyphenols in purple carrots. However, the total phenolic compounds of purple carrots were reduced by FD. Also, in the research of *Naomi N. Mbondo* et al., African eggplant was dried with FD, oven drying, vacuum drying, and solar drying to determine the best technique to obtain maximum product quality [122]. The results demonstrated that African eggplant was most effectively preserved while retaining its bioactive properties by FD. Several results were also obtained aside from FD. These results explained that oven drying at 70 °C significantly degraded the beta-carotene of the sample and indicated high temperatures which that caused the degradation of beta-carotene, while vacuum drying at 70 °C preserved more beta-carotene of the sample compared to oven drying at the same temperature, and solar drying caused the highest degradation in total phenolic compounds of the sample due to the activation of polyphenol oxidase enzymes. Although no lycopene was detected in fresh samples, interestingly small quantities of lycopene were determined in all dried samples.

In another study by *H. Wang* et al., the bitterness, hygroscopicity, and antioxidant properties of soybeans were detected by FD and spray drying, and their results were compared with each other to obtain the best drying technique [123]. Even though FD reduced the degradation of the bioactive properties of the sample, spray drying was determined to be the most usable technique to preserve these properties. In a study, two different varieties of FD, vacuum and atmospheric FD, were used to explain the effect of the FD varieties on the broccoli quality after drying [124]. The bioactive properties of broccoli, including total selenium content, total polyphenol content, and anti-radical power, were determined for each FD variety. As an outcome, while atmospheric FD was chosen as the best technique for the retention of the antioxidant properties of broccoli, the best retention of the total phenolic content of the sample was obtained by vacuum FD.

Also, other plant-based materials, including flowers and leaves, can be evaluated as food components for various industries and can be dried by FD to obtain high-quality products [125,126]. The fresh and dried forms of these components can be used in various industries. With this perspective, different drying techniques are being developed and invented to utilize these components for years to obtain the dried sample, which is rich in terms of nutritional value. For example, natural drying, microwave drying, hot air drying, infrared drying, vacuum drying, and FD were applied to *Bletilla striata* (Thunb.) Reichb.f. flowers [127]. FD was chosen as the most effective technique due to its high retention capacity for total phenols, total anthocyanins, and antioxidant properties, while also preserving the flower's characteristics and color with minimal browning. Other techniques yielded notable results as well; microwave drying had a low drying time; hot air drying exhibited a high browning capacity and long drying time but also had a high retention ability for total flavonoids and total polysaccharides, higher than FD; natural drying resulted in almost complete degradation of anthocyanins.

Also, the impacts of FD on basil leaves (*Ocimum basilicum*) were investigated in another study conducted by *de* Carvalho et al., [72]. Higher concentrations of phenolic compounds, carotenoids, and flavonoids in the sample were obtained with FD compared to fresh basil leaves. However, the vitamin C, chlorophyll a, chlorophyll b, and antioxidant activity of the leaves decreased slightly, resulting in a minimum degree of color and physical changes.

Seafood draws the attention of consumers with its nutritional and functional properties [128,129]. This food group is included in the food and supplement categories, and in both categories, production and storage conditions are evaluated to protect the content and extend long shelf life [130]. This category includes fish, shellfish, mollusks, marine plants, and other seafood. With the development of the natural and healthy nutrition trend in the last decade, the consumption of algae, one of the foods with proven health benefits, has also increased [131]. Algae offer valuable bioactive content in addition to protein, fat, carbohydrates, vitamin, and mineral content [131]. At this point, different techniques have been used to preserve its content [132]. The FD technique is used to increase the retention ability of the bioactive ingredients of the kinds of seafood after the drying processes [133]. Vacuum FD and vacuum spray drying were applied to myofibrillar proteins from silver carp in the article of *J. Niu* et al.*;* vacuum FD protected the protein of the sample more effectively than vacuum spray drying [134].

Also, this drying technique, which is highly protective of foods, was applied to the mushroom species. In a study, vacuum FD, oven drying, and air drying were used to dry *Cordyceps sinensis* [135]. The research showed morphological and bioactive compound differences among the techniques. FD preserved the antioxidant activity of the sample better and resulted in better color and fullness compared to other drying techniques. Additionally, in another study on FD of the mushrooms, some bioactive properties were detected in the dried samples but not in the fresh ones [136]. Hot air drying and FD were used to process shiitake mushrooms and preserve their bioactivity. Both bioactive and physicochemical properties were assessed. Interestingly, cysteine was found in the dried samples but not in the fresh ones. These results may be attributed to the temperatures used in the drying techniques.

In addition to these studies, several studies on the effect of FD on the bioactive properties, physical properties, and flavor of various materials, including potatoes, cabbage, garlic, eggplant, and tomatoes as vegetables; berry species, apples, hawthorn fruit, strawberries, and pears as fruits; other plants, honey, milk, seafood, and mushrooms, were examined (**Table 1**) [15,84,137–139].

Table 1. Effects of freeze-drying on nutritional and physical properties of various products.

Samples	Percentage of Removed Water	Outcomes	References
Gastrodia Elata	*	-Exhibit the retention of active ingredients and original color. -Exhibit the retention of the internal structure and main components. -Exhibit the preservation of pungent odors as physical and sensory properties	[74]
Basil leaves (*Ocimum basilicum*)	Approximately 94.1%	-Exhibit the no net change in pH. -Exhibit the decrease in the	[72]

		chlorophyll content and vitamin C. -Exhibit the increase in total polyphenols, flavonoids, antioxidant activity, and total carotenoids. -Exhibit the darkening as color changes.	
Pleurotus eryngii	*	-Exhibit the preservation of the color and texture features. -Exhibit the decrease of the hardness. -Exhibit the high retention capacity of total phenolic contents. -Exhibit the decrease in the amount of polysaccharide content, ascorbic acid content, and antioxidant capacity for ABTS was high but low for DPPH total volatile constituents, eight-carbon volatiles, essential amino acids, and free amino acids.	[140]
White mulberry fruits (*Morus alba* L.)	*	-Exhibit the high lightness and yellowness color. -Exhibit the high retention of the amount of L-ascorbic acid, total phenolics, and antioxidant activity as bioactive properties.	[7]
Brown rice (Oryza sativa) porridge	*	-Exhibit the high retention of the amounts of potassium, sodium, iron, zinc, and vitamin B6.	[82]
Potato tubers	-The moisture value changed with exposure time by freeze-drying. For example, absorbed moisture was 39.80 + 0.2 g	-Exhibit the increase in the total content of alcohol-soluble flavonoids in terms of quercetin, phenolic compounds, and low	[137]

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	for 5 minutes, while for 600 minutes, 47.2 + 0.2 g.	molecular weight antioxidants according to mg/g dry matter compared with a fresh sample.	
Bovine skim colostrum	*	-Exhibit the retention of the bioactive proteins and immunoglobulins. -Exhibit the no net change on the secondary structure of proteins.	[104]
Black Currant and Sea Buckthorn Berries	83.73% for black currant berries and 82.8% for sea buckthorn berries.	-Exhibit the high retention of the vitamins, organic acids, and carbohydrates.	[110]
Wild guava leaves	96–92%	-Exhibit the preservation of the total phenolic content, total flavonoid content, and antioxidant capacity as bioactive properties. -Exhibit the preservation of the taste and physical properties.	[84]
Chilean murta (*Ugni molinae* Turcz) shrub	*	-Exhibit the retention of the antioxidant activity, anti-inflammatory activity, anti-tumoral activity, and total phenolic compounds, including catechin, pyrogallol, tyrosol, and gallic acid.	[12]
Maoberry fruits	Approximately 85%	-Exhibit the reduction on the spoilage of ascorbic acid, total phenolics, total flavonoids, and total anthocyanins.	[8]
Vacuum impregnation celery	*	-Exhibit the protection on the amount of the volatile organic compounds, total phenolic content, and antioxidant properties. -Exhibit the not significant	[10]

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		color change.	
Broad beans	*	-Exhibit the protection of total phenol content, antioxidants, and gallic acid. -Exhibit the preservation of starch and amino acids. -Exhibit the preservation of the physical properties, including color and shape.	[6]
Cynomorium songaricum	*	-Exhibit the retention of the cellular structure, color, textural properties, antioxidant activities, and flavor.	[15]
Prunus domestica	97.60%	-Exhibit the decrease in the quercetin content. -Exhibit the increase in the cyanidin content. -Exhibit the alternation of the phytochemical and antioxidant properties.	[23]
Coconut Drink	*	-Exhibit no net change in mineral content, amino acid profile, or fatty acid profile. -Exhibit the decrease of the fermentation aroma. -Maintain the flavor, except for a reduction in sweetness as a sensory property.	[141]
Wild Blueberry (*Sideroxylon mascatense*)	Approximately 86.46%	-Exhibit the protection of the total polyphenol, fat, and flavonoid contents.	[142]
Cabbage (*Brassica oleracea var. capitata f. alba*)	*	-Exhibit the retention of the contents of catechin hydrate, chlorogenic acid, 4-hydroxybenzoic acid, and rutin hydrate.	[17]

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		-Exhibit the decrease in the contents of isothiocyanates, gallic acid, epicatechin, p-coumaric acid, sinapic acid, and myricetin.	
Asian pear	80% (kg of water/kg of dry matter)	-Exhibit the high preservation of antioxidant and total phenolic contents, while no significant change in chlorogenic acid. -Exhibit the retention of the porous structure and high rehydration capacity.	[11]
Bilimbi (*Averrhoa bilimbi*)	Approximately 83,3%	-Exhibit the protection of the ascorbic acid content. -Exhibit the increase in the oxalic acid content, total sugar content, and antioxidant activity.	[143]
Pomegranate arils	Approximately 80%	-Exhibit the high retention of the bioactive compounds, including antioxidants and total phenolic contents. -Exhibit the no net change in physical properties.	[14]
Centella asiatica L. leaves	97.6%	-Exhibit the alternatives in physical properties including color and also increased lightness. -Exhibit the high retention ability of the antioxidant activity, phenolic content, and triterpene saponins.	[32]
Edamame	97%	-Exhibit the high protection on the total phenolic content, total chlorophyll content, ascorbic acid content, and antioxidant activity. -Exhibit some physical	[139]

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		alterations in color and shape.	
Rosehip	*	-Exhibit the retention of the total bioactive compounds, including β-carotene, lycopene, phenolic composition, and antioxidant activty. -Exhibit the color alternation.	[34]
Durum wheat (*Trıtıcum Durum*) sprouts	90-85%	-Exhibit the preservation of nutritional properties and color. -Exhibit the decrease in the water absorption capacity.	[70]
White and purple garlic	More than 90%	-Exhibit the high whiteness. -Exhibit the retention of the allicin content, total phenolics, and antioxidant activity.	[76]
Sweet potato	92.58% for the purple sample and 94.49% for the yellow sample	-Exhibit the preservation effect on the antioxidant capacity, hydrolyzable polyphenols, and condensed tannins of the purple and yellow samples.	[77]
Chia (*Salvia hispanica* L.) Herb	*	-Exhibit the preservation of bioactive properties, particularly total carotenoids, total polyphenols, and antioxidant activity.	[86]
Red-fleshed apples	*	-Exhibit the degradation of phenolics and anthocyanins. -Exhibit the alternation on physical properties, including color, and sensory properties, including hardness.	[120]

Sea buckthorn (*Hippophae rhamnoides* L.) berries	*	Exhibit the high retention of the total phenolic content, total flavonoid content, and antioxidant capacity of the sample as bioactive properties.	[129]
Stingless bee (Heterotrigona itama) honey	95%	Exhibit the preservation of the antioxidant activity, total phenolic content, and flavonoids.	[138]
Plums	*	-Exhibit the high retention of the anthocyanin pigment content, total polyphenol content, phenolic acid, and chlorogenic acid.	[109]
Ginger (*Zingiber officinale* Roscoe)	*	-Exhibit the protection on the total phenolic content, total flavonoid content, gingerol content, and volatile compounds.	[65]
Garlic	Approximately 91.57%	-Exhibit the minimal shrinkage, reduced hardness, and the low rehydration capacity. -Exhibit the superior protection of the bioactive properties.	[9]
Persimmon	*	-Exhibit the reduction of the degradation of total phenolic content, total flavonoid content, soluble tannin, arbutin, proanthocyanidin, β-carotene contents, and antioxidant activity.	[16]
Hawthorn fruit	99.8%	-Exhibit the decrease in the total phenolic content, antioxidant activity, and total flavonoid content. -Exhibit the preservation of	[53]

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		the color and reduction in shrinkage. -Exhibit the rehydration capacity development.	
Okara	More than 96%	-Exhibit high retention ability on the bioactive properties, including total phenols, soybean isoflavones, and antioxidant activity. -Exhibit the protection on the physical and physicochemical properties, such as water retention capacity, swelling capacity, oil retention capacity, solubility, and color.	[1]
Courgette slices	*	-Exhibit the changes in quercetin, lutein and zeaxanthin, carotenoids, and chlorophylls as bioactive properties, as well as color as a physical property.	[119]
Ginger (*Zingiber officinale* Rhizome)	*	-Exhibit the change on the total phenolic contents, total flavonoid contents, and antioxidant activities. -Exhibit the alternation of the color, taste, odor, and turbidity.	[19]
Bletilla striata (Thunb.) Reichb.f. flower	*	-Exhibit the minimal color change and browning. -Exhibit the preservation of the total phenols, total anthocyanins, and antioxidant activity.	[127]
Ginkgo biloba L. seed	*	-Exhibit the preservation of the total sugar, reducing sugar, soluble solids, ascorbic acid, phenolics, and tannins. -Exhibit the change in	[4]

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		physical property including shrinkage.	
Tunisian eggplant *(Solanum melongena* L.)	*	-Exhibit the decrease and increase in the anthocyanins, ascorbic acid, total phenols, flavonoids, and antioxidant activities as bioactive properties, also on color change and enzymatic browning index as physical properties.	[24]
Ginkgo biloba L. seed	Approximately 94%	-Exhibit no net changes in the amount of the physicochemical properties, total phenolic content, phytochemical contents, and antioxidant activity. -Exhibit the preservation of the physical properties including rehydration capacity, and taste and enzymatic activity as bioactive properties.	[83]
Orange puree	Approximately 95.8%	-Maintain a clear, yellowish, darker, and saturated color with high porosity. -Exhibit the reduction of vitamin C, carotenoids, and phenols.	[80]
Organic strawberry powders	*	-Exhibit the stability of the values of vitamin C and polyphenols. -Exhibit the preservation of antioxidant activity.	[112]
Moringa stenopetala leaf extract	Approximately 98%	-Exhibit the preservation of the total phenolic content, total flavonoid content, and antioxidant activity.	[125]

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Oyster mushrooms (*Pleurotus Ostreatus*)	Approximately 96.15%	Exhibit protection of the color and conformation. Exhibit the retention of the total phenolic content, total flavonoid content, and antioxidant activity.	[144]
Brown alga (*Durvillaea antarctica*)	Approximately 92.65%	-Exhibit the protection on the bioactive compounds except the fatty acid and amino acid profiles. -Exhibit the red tones.	[133]
Red seaweed (*Pyropia orbicularis*)	Approximately 97.21%	-Exhibit the protection of the product quality, including physical and bioactive properties (especially phycoerythrin, phycocyanin, and total phenolic contents).	[132]
Red-fleshed apples	97.01%	-Exhibit the preservation of the phenolic content, antioxidant activity, and physical properties including color and shape.	[111]
Hawthorn fruit powders	Approximately 95%	-Exhibit the high preservation of the total phenolic content and natural color.	[68]
Edible rose flower (*Rosa rugosa* flower)	Approximately 95.80%	-Exhibit the stability of the amount of volatile compounds and the taste. -Exhibit the retention of flavonoids, anthocyanins, and vitamin C.	[126]
Mango (Mangifera indica) leather	83.69%	Exhibit the retention of the total phenolic, total flavonoid, and total carotenoid contents. -Exhibit the low change on the color and shape.	[114]
Ginger (*Zingiber officinale*) rhizome	Approximately 91.66%	-Exhibit the high retention of the total phenolic content,	[71]

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		antioxidant activity, total carotenoids and phenolic compounds.	
Feijoa pulp *(Acca sellowiana)*	*	-Exhibit the preservation of the total phenolic content, total flavonoid content, and especially the ascorbic acid and antioxidant activity.	[5]
Purple carrot	-A small amount of the water content provides the moisture, however, there is no net information about the percentage of the water.	-Exhibit the no net change in anthocyanin content. -Exhibit the decrease of the total phenolic content. -Exhibit the increase of the carotenoid content.	[73]
Fish	*	-Exhibit the increase of the free sulfhydryl content and surface hydrophobicity. -Exhibit the decrease of the solubility of fish myofibrillar proteins, water retention capacity, and biochemical properties, including Ca^{2+}-ATPase activity.	[134]
Cardaba banana flour	Higher than 90%	Exhibit the retention of the protein value, indigestible carbohydrate, total phenolic content, flavonoids, and antioxidant activity.	[87]
Chinese yam flour	*	Exhibit positively the affect the bioactive properties including total phenolic content.	[88]
Bitter gourd *(Momordica charantia L.)* slices	94%	Exhibit the increase in the product quality by preserving the bioactive properties and structure.	[33]
Lemon myrtle *(Backhousia citriodora)*	Approximately 96.5%	-Exhibit the retention of the total phenolic content, total	[89]

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		flavonoids, proanthocyanidins, gallic acid, hesperetin, and antioxidant activity	
Cranberries	*	-Exhibit the preservation of the contents of total phenolics, total flavonoids, total monomeric anthocyanins, and antioxidant activity. -Exhibit the physical alterations.	[145]
Strawberries	*	-Exhibit no changes in color. -Exhibit the degradation of vitamin C content, polyphenolic content, flavor, porosity, and texture values of the sample.	[116]
African eggplant	~90%	-Exhibit the reduction of beta-carotene and total phenolics. -Exhibit degradation of the antioxidant capacity. -Exhibit the increase in lycopene content.	[122]
Ashitaba leaves *(Angelica keiskei Koidzumi)*	*	-Exhibit the high retention of the chlorophyll and flavonoids. -Exhibit the enhancement of the green color and flavor.	[146]
Cirsium setidens	*	-Exhibit the retention of the flavonoid contents and antiproliferative activities.	[85]
Australian maroon bush (*Scaevola Spinescens)*	*	-Exhibit the prevention of the decomposition of antioxidant activity, total phenolic content, total flavonoid content, and saponins.	[13]

Edible Centaurea (*Centaurea cyanus*) petals	*	-Exhibit the increase in antioxidant activity and flavonoids. -Exhibit the decrease in the carotenoids and some physicochemical properties. -Exhibit the darker, smaller, and more shriveled shape.	[147]
Button mushroom	74.2%	-Exhibit the decrease of the degrees of luminosity. -Exhibit the higher red tones. -Exhibit the no change in rehydration rates of the sample.	[75]
Chokeberries	*	-Exhibit the high preservation of total phenolics and anthocyanins as bioactive compounds, as well as color as a physical property.	[148]
Guava (*Psidium guajava Linn.*)	*	-Exhibit the decrease in the total polyphenols, flavonoids, and antioxidant activity.	[69]
Yellow passion fruit residues *(Passiflora edulis f. flavicarpa)*	*	-Exhibit the increase in the total phenolics and total flavonoids. -Exhibit the decrease in the citric acid and ascorbic acid.	[18]
Chinese ginger (*Zingiber officinale* Roscoe)	*	-Exhibit the high ability to preserve gingerols, total phenolic content, total flavonoid content, and antioxidant activity.	[121]
Raspberry	86.6% (g /g dry basis * 100)	-Exhibit the high protection of the original color. -Exhibit the retention of the total polyphenol content, total flavonoid content, and antioxidant activity.	[149]

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Apple	78.8%	-Exhibit easily shrank and turns to yellow, followed by browning.	[49]
Persimmon	Higher than 75%	-Exhibit the high lightness and yellowness. -Exhibit the high retention of total phenolics, total flavonoids, condensed tannin, total hydrolyzable tannin contents, antiradical activity, and antidiabetic activity.	[150]
Saskatoon berries	Approximately 88%	-Exhibit the protection of the anthocyanin and antioxidant activity.	[113]
Blueberries	*	-Exhibit the reduction in ascorbic acid content. -Exhibit the increase in total phenolic content. -Exhibit the no net change in antioxidant efficiency of the sample.	[151]

*Not identified.

5. Impact on Physical Properties and Flavor

The drying process affects the physical properties of the dried products as well as their bioactive properties. Sensory or rheological properties of the products, such as color, smell, and taste, can change after drying. For instance, according to the research of *G. Rajkumar* et al., FD increases the flavor retention of tomatoes by affecting their volatile compounds [152]. FD provided better results in flavor protection, with spray drying results being close to FD. However, the hot air drying technique had the lowest flavor retention capacity for tomatoes.

In another study on *Gastrodia elata*, FD was the most effective technique in terms of appearance and color maintenance compared to hot air drying, vacuum drying, microwave drying, and microwave vacuum drying, despite having the longest drying time [74]. However, microwave vacuum drying was the most practical technique for *Gastrodia elata*, as it preserved the original shape, color, and active components while reducing the drying time. In another study on *Dendrobium officinale* flower tea, also natural air drying, and oven drying except the FD were applied, and the study determined that vacuum FD was the most effective technique for the preservation of the shape, color, taste, and antioxidant activity of the sample compared to other techniques [153]. However, vacuum FD showed the least protective effect on the aromatic compounds of the sample. When the results were obtained are evaluated, natural air drying was determined to be the best technique because of its preservation ability on the bioactive and physical properties of the fresh flower scent.

In another study, microwave drying, vacuum microwave drying, sun drying, vacuum drying, hot air drying, and vacuum FD were used to improve the preservation and long-term storage of the kiwifruit pomace, and these drying methods were compared [154]. As a result of the study, while

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vacuum FD caused the longest drying time and the highest energy consumption with better retention capacity, vacuum microwave drying showed a shorter drying time and lower energy consumption. Vacuum microwave drying was considered the most suitable drying technique for the pomace because of its retention ability on the bioactive contents and vitamin C. Also, vacuum FD was good at the protection of the original structure of the pomace by preserving its physical properties, nutrient values, antioxidant activity, and microstructure.

In another article on vacuum FD, air impingement drying, hot air drying, pulsed vacuum drying, and medium- and short-wave infrared radiation drying were applied to garlic slices to determine the effects of these drying techniques on the slices [155]. As a result of study, while vacuum FD caused the longest drying time and the highest energy consumption with better retention capacity, vacuum microwave drying showed a shorter drying time and lower energy consumption. Vacuum microwave drying qualified as the most suitable drying technique for the pomace because of its retention ability on the bioactive contents and vitamin C. Also, vacuum FD was good at the protection of the original structure of the pomace by preserving its physical properties, nutrient values, antioxidant activity, and microstructure.

In another article about vacuum FD, air impingement drying, hot air drying, pulsed vacuum drying, and medium- and short-wave infrared radiation drying were applied to garlic slices to determine the effects of these drying techniques on the slices [146]. A high amount of chlorophyll and flavonoid contents were retained by this drying technique.

In some cases, FD may not be the best drying technique to preserve the bioactive and physical properties of the samples compared with other drying techniques. For example, coffee pulp tea was dried by applying the FD to preserve the original color, total phenolics, trigonelline, total soluble sugars, chlorogenic acid, and protocatechuic acid of the tea [156]. As a result of the study, a long drying time and the lowest taste were obtained by applying the FD compared with sun drying, hot air drying, microwave drying, and vacuum drying. Also, the most significant degradations of bioactive components of the sample were acquired by spray drying and hot air drying. In contrast, microwave drying highly increased the retention of the bioactive components and preserved the color, taste, and aroma of the tea while reducing the drying time. Due to these outcomes, microwave drying was considered as the best drying technique to preserve the tea.

On the other hand, FD can be applied to protect the quality of mushrooms due to the high retention capacity of the FD on the components of the sample [144]. In a study, vacuum FD, hot air drying, microwave rolling-bed drying, hot-air microwave rolling-bed drying, and microwave vacuum rolling-bed drying techniques were applied to Pleurotus eryngii mushrooms, and these drying techniques were compared with each other according to their drying suitability for the preservation of flavor and physicochemical properties of the sample [140]. When the results were examined, FD time was effectively reduced by microwave rolling-bed drying and hot-air microwave rolling-bed drying. However, the least drying time was obtained, with color and flavor protection of the mushrooms achieved by microwave vacuum rolling-bed drying compared with other drying techniques. As a result, the study explained that microwave vacuum rolling-bed drying and vacuum FD gave similar outcomes regarding the bioactive properties, color, and taste of *Pleurotus eryngii*. However, microwave vacuum rolling-bed drying was chosen as the best drying technique among other techniques instead of vacuum FD due to its short drying time with a high-quality final product.

Additionally, FD is used to improve the shelf life of various industrial products, while decreasing the degradation of their bioactive properties and physical properties especially flavor [143]**.** For example, in the case of coconut drinks, FD maintained the Mg and Fe content but lowered the Na content [141]. Furthermore, FD preserved the sensory properties, flavor, and taste, though some minor degradation was observed in the sweetness and fermented aroma of the coconut drink. However, these degradations were minimal. When comparing the freeze-dried coconut drink with the fresh coconut drink, no significant changes were observed in several minerals, amino acids, and fatty acids in the freeze dried sample. As a result of this experiment, the shelf life of the sample was successfully improved. More examples of the effect of ì FD on the samples, including fruits, vegetables, mushrooms, and flowers, were examined [145–150].

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6. Conversion of Waste and By-Products

Waste is a global problem that every person must consider in terms of several reasons, including the cause of the environmental pollution, especially, and governmentally caused the cost of degrading it [157–159]. Waste and by-product conversion has been a significant topic in recent years for several industries and researchers [160–162]. Waste product transformation into valuable material is conducted according to the zero waste policy [163]. Communities are solving the problem of waste, which is produced in various ways [164].

The waste products, including peels, pomaces, and seeds, that are obtained from foods have a high potential to improve the bioactivity of other industrial production [165–168]. Instead of using new source, the bioactive contents in the waste products, which are dried and protected by FD, can be used by manufacturers based on a zero-waste policy, which is important in all fields [169–172].

Valorization of waste products is enhanced by using the FD techniques in various industrial areas [22,173–175]. The utilization of bioactive content derived from bread waste as a product for various industries is possible [176]. In this study, bioactive peptides were obtained, microencapsulated, and protected from environmental conditions such as oxygen and temperature. Maltodextrin was used in the microencapsulation method to protect the bioactive peptides in the sample, and it was also found to facilitate freeze-drying processes.

The bioaccessibility of bioactive compounds increased in the waste and by-products enhanced by the FD process. In a study conducted by *J.F. Aldana-Heredia* et al., waste tomato peels, which contain a high amount of total carotenoids, were encapsulated by applying the FD to preserve the total carotenoids for reuse in industrial applications [177]. Another study investigated the conversion of the waste cocoa bean shells valorized by the FD [178]. In this study, the ingredients of the shells supported the nutritional value of cookies to show the importance of waste product conversion. As a result of the study, the shells were dried using FD and spray drying techniques to preserve the ingredients of the shells, especially the protein, antioxidant activity, and polyphenol contents. Also, while the powder produced by applying FD had the highest protein content, the powder obtained by spray drying had the highest antioxidant activity and total phenolic content in the cookies.

In another study on carotenoids, FD was used to obtain this pigments, which are the bioactive components of most foods [179]. These were obtained from waste carrot products with the application of the encapsulation which provides better preservation of the sample ingredients. In the study by *V. Seregelj* et al., carotenoids were obtained by spray drying and FD from carrots by applying various wall materials, including whey protein, maltodextrin, and inulin. Also, this study demonstrated which drying technique is most effective for the preservation of the carotenoids. Spray drying was successful due to the lowest water activity, moisture content, and particle size of the samples that were achieved by encapsulation, while encapsulation by FD produced the freeze-dried sample which has the highest hygroscopicity, oxidative stability, and original color properties. These results may be due to the application of high temperatures that significantly impact the bioactive properties and morphology of the samples, during the spray drying processes. In addition to these, a study examined the conversion of berry pomace and oregano processing wastes by FD [180] Freeze-dried waste materials were used as the bioactive ingredient of various products instead of new sources. These studies explained why and how FD was chosen to transform waste products into nutritionally rich products by improving bioactive properties, also its potential was highlighted in sustainable food processing (**Table 2**) [15,181–188].

Table 2. Impact of freeze-drying process on conversion of waste and by-products.

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Waste sample	**Obtained material result of drying**	**What content and bioactive properties are used to increase bioactivity?**	**Valorization areas or improved product by bioactive properties**	**References**
Elderberry pomace	Freeze-dried elderberry pomace	- Phenolic content - Antioxidant capacity - Monomeric anthocyanin content	Food/nutraceutical products	[181]
Raspberry pomace	Freeze-dried raspberry pomace	- Fat and ash content - Carbohydrate content - Antioxidant properties	Gluten-Free Bread	[182]
Bignay [Antidesma bunius (L.) spreng.] pomace	Freeze-dried bignay pomace	- Total phenolic and anthocyanin changed but were low. - Flavonoids and tannins were not affected. - High antioxidant properties.	Dietary products	[183]
Blueberry pomace	Blueberry pomace powder	- Antioxidant activity - Anthocyanin - Total phenol - Total sugar	Industrial products	[189]
Grape pomace	Grape pomace flour	- Carbohydrates - Vitamin C - Minerals - Phenolic compounds	Gluten-free filled cookies.	[184]
Royal gala apple pomace	Freeze-dried royal gala apple pomace	- Total phenol content - Antioxidant properties - Phenol profile - Thickening capacity	Food, cosmetics, and nutraceuticals.	[185]
Watermelon rind	Freeze-dried watermelon rind	- Phenolic compounds - Flavonoid compounds - Antioxidant capacity - Polyphenolic compound - Ascorbic acid	Food supply, sustainability, health, and environmental studies	[162]
Banana and watermelon peel	Banana and watermelon peel powder	- Total phenolic content - Antioxidant activity - Antimicrobial activity	*	[173]

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País grape (*Vitis vinifera* L.) marc	Freeze-dried grape marc	- Phenolic compounds - Proanthocyanidins - Dietary fiber contents	Animal feed production and dietary ingredient	[171]
Apple pomace and pomegranate peel	Apple pomace powder and pomegranate peel powder	- Total phenolic content - Total flavonoid content - Antioxidant activity	Functional yogurt	[22]
Turmeric (Curcuma longa L.) rhizome	Freeze-dried turmeric rhizome	- Ascorbic acid - Curcumin - Total phenols - Total flavonoids	*	[170]
Pineapple pomace	Pineapple pomace powder	- Carbohydrate contents - Fat content - Protein contents	Set-type yogurt	[25]
Mango (*Mangifera indica* L.) and Rambutan (*nephelium lappaceum* L.)	Exotic Fruit Seed Powders	- Tannins - Total polyphenols - Antioxidant activity	Dietary and functional food production	[26]
Chokeberry pomace extracts	Chokeberry pomace powder	- Phenolic acids - Anthocyanins - Flavonoids - Content of hydroxymethyl-L-furfural	Juice	[27]
Raspberry pomace	Freeze-dried raspberry pomace	- Total phenolic compounds - Phenolic acids - Flavonoids - Anthocyanins - Antioxidant properties	Fruit and vegetable industry	[161]
Tomatoes peels	Tomato peel powder	- Lycopene - Carotenoids - Antioxidant activity	Industrial applications	[174]
Olive leaves	Freeze dried olive leaves	- Total phenolic compounds - Antioxidant capacity	Food, pharmaceutical or cosmetic industries	[172]
Olive	Phytocompounds	- α-tocopherol - Carotenoids	Pharmaceutical products	[158]

pomace		- Chlorophylls - Polyphenols - β-sitosterol		
Wine pomace	Freeze-dried wine pomace	- Antioxidant capacity - Total phenolic content	Food supplement industry	[166]
Grape pomace	Microencapsulation of powder with freeze-drying	- Phenolic compounds retention - Total monomeric anthocyanins - Antioxidant activity	*	[167]
Black chokeberry pomace extracts	Dispersible powder of black chokeberry pomace	- Polyphenolic contents - Antioxidant activity	Dairy products, food suitable for people with dysphagia, and yoghurt-based products	[29]
Berry pomace	Freeze-dried berry pomace	- Total phenolics - Flavonols - Anthocyanins - Antioxidant activity	Food industry and other industries	[30]
"BRS magna" grape skin residues	Freeze-dried grape skin residues	- Antioxidant activity - Total phenolics - Total flavonoids - Anthocyanins - Procyanidins	*	[186]
Pitted olive pomace	Freeze-dried pitted olive pomace	- Dietary fiber contents - Low antioxidant capacity - Phenolic contents.	Healthful food ingredient	[187]
Kinnow (*Citrus reticulata*) peel	Freeze-dried kinnow peel	- Total phenols - Flavonoids - Antioxidant activity -Beta-carotene -Ascorbic acid	Food sector	[28]
'Ataulfo' mango by-products	Freeze-dried mango peel and paste	- Carotenoids - Total phenolic concentration	Functional foods or beverages	[159]
Grape (*Vitis labrusca* L.)	Freeze-dried grape pomace	- Total phenolics - Antioxidant activity	Yoghurt production	[108]

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pomace				
Lemon (*Citrus limon) pomace*	Freeze-dried lemon pomace	- Polyphenols - Antioxidant activity - Neohesperidin content - Total flavonoid content - p-coumaric acid content - Gallic acid content - Rutin	*	[188]
Blackcurrant pomace	Blackcurrant pomace powders	- Total polyphenolic content - Antioxidant capacity	Colorization of instant foods	[36]
Banana (*Musa cavendish*) peels	Freeze-dried banana peel	- Antioxidant capacity - Total phenolic content - Total flavonoid content - Proanthocyanidin content	Nutraceutical industry	[35]
Murta(*ugni molinae* turcz) berries	Freeze-dried murta berries	- Total flavonoids - Anthocyanin	Functional foods	[165]
Watermelon (Citrullus lanatus) rind	Freeze-dried watermelon rind	- Ash content - Fat content - Carbohydrate content - Protein content - Alkaloid content - Tannin content - Vitamin C - Antioxidant activity	Food processing industry	[168]
Grapefruit peels (*Citrus paradisi* macf.)	Freeze-dried grapefruit peels	- Flavonoids - Antioxidant activity - Phenolic compounds	Biomedical usage	[175]

*Not identified.

7. Conclusions

Freezing, primary drying, and secondary drying are the main processes of the FD. FD is used to preserve bioactive properties and physical characteristics containing flavor and texture of samples with enhanced shelf life. In some studies, FD is the best drying technique compared with several

 doi:10.20944/preprints202409.0950.v1

other drying techniques, including hot air drying, spray drying, and microwave drying to obtain the highly preserved waste product after drying.

In summary, FD is particularly effective for drying samples with a short shelf life. However, it is an expensive technique, which restricts its widespread use. Due to the high cost of FD, there is a need to explore other techniques that yield results comparable to FD. Furthermore, if FD is necessary, it can be enhanced by combining it with other techniques or using devices such as microwaves to assist the process. These devices can shorten the drying time, thereby reducing the overall cost and energy consumption while maintaining the effective preservation of the bioactive properties of the samples.

To further reduce costs and improve product quality, various pretreatments, including high hydrostatic pressure and ultrasound, can be applied prior to FD. The article also exemplifies several parameters, including pretreatment, size, temperature, sample, time, and pressure of the FD process, which directly affect the physical and chemical properties of end products. With an enhanced perspective, FD was used just for protection of the bioactive property, physical property, and flavor of the sample; it was used to utilize the waste by-products that are produced after industrial manufacture. The result of these manufacturing, generally contentful powders were produced to add to other products, including bread, yogurt, and dietary products, as bioactive content sources. FD is a novel and valuable technique for various industries, and ongoing research aims to improve its efficiency and effectiveness for various samples.

Author Contributions: Conceptualization, S.K., S.S., and N.C.; writing—original draft preparation, N.C., S.S.; writing—review and editing, S.K., S.S., M.B., Y.J. and N.C.; visualization, N.C., S.S., and S.K. All authors have read and agreed to the published version of the manuscript.

Funding: This review received no external funding.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Not applicable.

Conflicts of Interest: The authors declare no conflicts of interest.

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