A REVEW OF FREEZE DRYING AND ITS IMPACT ON FRUITS AND VEGETABLES

A Thesis

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ABSTRACT

This review gives an overview of the influences of freeze drying on the nutritional, chemical, and physical characteristics of fruits and vegetables. Advantages and drawbacks associated with lyophilization are discussed and approaches to mitigate the disadvantages proposed. Freeze drying maximizes the retention of total phenolic content, carotenoid, ascorbic acid, color, and microstructure of a majority of fruits compared to conventional drying methods. Higher drying temperature negatively affects these properties, but the influences are also dependent on factors inherent to the fruits. Various research suggests that the long processing time and high energy consumption of freeze drying can be addressed by using combined drying technology.

BIOGRAPHICAL SKETCH

Shijun completed her undergraduate study in University of Massachusetts Amherst majoring in Food Science. She graduated from Cornell University in 2022 as a Master of Food Science. Her plan after graduation will be working in the food industry with a focus on quality assurance.

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I dedicate this thesis to my parents, without their love and support, the completion of my degree would not have been possible.

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LIST OF ABBREVIATIONS

ABTS – 2, 20-Azinobis (3-ethyl- benzothiazoline-6 sulfonic acid) radical scavenging property

Acy – monomeric anthocyanin

AD – air drying

AD-MVD – combined drying consisting of air drying and microwave vacuum drying

AEAC – ascorbic acid equivalent antioxidant capacity

CD/MVD – convective drying/microwave-vacuum drying

DB – dry weight basis

DPPH – 2-diphenyl-1-picrylhydrazyl (DPPH) radical-scavenging ability

FD – freeze-dried

FD-MVD – combined drying consisting of freeze drying and microwave vacuum drying

FRAP – ferric reducing antioxidant activity

MC – moisture content

MVD – microwave vacuum drying

TFC – total phenolic content

Tg – glass transition temperature

TPC – total flavonoid content

UT – ultrasonic treatment

1. BACKGROUND

According to USDA's Food Guide Pyramid, a healthy diet should be composed of a variety of food groups – bread, cereal, rice, and pasta group; vegetable group; fruit group; meat, poultry, fish, dry beans, eggs, and nut group; milk, yogurt, and cheese group; and fats, oils, and sweets group (Harper 1981). Consumption of 2 to 4 servings of fruits daily is recommended for a wholesome diet (GAO 2003). Fruits do not only have extraordinary tastes, flavors, and odors, but are also abundant in vitamins, minerals, fiber, and various polyphenols with antioxidant qualities (Zotarelli et al. 2012). Research suggests that adequate intake of fruits and vegetables effectively reduces the risks of heart disease, stroke, diabetes, among other chronic diseases (GAO 2003). However, most fruits around the world are only seasonally available. For instance, summer is the peak season of berries but not of orange, which has the maximum production during winter. Therefore, it is critical to find a way to preserve fruits not only to provide a steady availability throughout the whole year, but also in a manner that can retain the physical and chemical properties of fruits to the largest extent (Liliana et al. 2015). Dehydration is one of the oldest and the most common practices used to preserve, extend shelf life, and provide year-round supply of fruits and other food products. Drying elongates the product shelf life by lowering water activity of the fruits which inhibits or slows down the enzymatic, chemical, and microbiological reactions happening within the fruits, thereby ensuring the microbiological stability (Zotarelli et al. 2012).

Traditional dehydration methods, such as sun-drying and hot air drying, usually have a low drying rate and compromised product qualities due to the high processing temperature (Sarkar et al. 2020). Freeze drying, also called lyophilization or cryodesiccation, is a low temperature dehydration processing method of placing completely frozen products in a vacuum and removing the liquid content of the product through sublimation (Li et al. 2014). The technology was first used on a large-scale during World War II. Blood plasma and penicillin were processed through freeze-drying to preserve stability and viability when refrigeration was not available (Bhatta et al. 2020). The technique is broadly favored because of its ability to preserve bioactive compounds. In recent years, freeze-drying has gained increasing attention in food and (bio)pharmaceutical industries, including the production of foods, vaccines, and proteins (Assegehegn et al. 2020). However, freeze drying is also a costly, energy-intensive, and time-consuming process when compared to conventional dehydration techniques (Jiang et al. 2017). Therefore, most foods that undergo this process are high-value products, such as seasonal fruits and vegetables, space foods, coffee, and insects (Bourdoux et al. 2016).

There are two main types of freeze dryers currently available on the market — home use freeze dryer and laboratory/production-scale freeze dryer. At the beginning, freeze-dried foods were produced mainly for military, astronauts, or hikers due to their various benefits and distinguished characteristics, including light-weighted, easily rehydrated, shelf stable, and high preservation of structure and nutrients (Liliana et al. 2015). In recent years, cryodesiccation technology started to gain more attention as it has been used to develop novel vegetable and fruit-based products available to the public, including freeze dried snacks, toppings for bakeries, and powders used as natural food colorants (Bhatta et al. 2020). According to Mordor Intelligence, the global freeze-dried food market is expanding at a rate of 7.4% a year, among which freeze-dried fruits occupy the largest portion of the FD food market with a share of 32% (McHugh 2018). Consumers developed an appetite for freeze dried products for several reasons, one of which would be the increasing demand for clean label products – products containing as few ingredients as possible. Freeze dried products are known to have prolonged shelf lives even without the addition of any preservative which corresponds with modern consumers' pursuit of healthy and convenient diets, driving the growth of the market (Licensors et al. 2020). Furthermore, reduced degradation of product quality, high retention of nutritional value, and the unique crispy texture are also the reasons why freeze drying is considered a promising process for the dehydration and preservation of fruits and vegetables. Market research have shown that there is an increasing acceptance and liking for crispy dried vegetable and fruit snacks, indicating a growing consumption trend for freeze-dried products (Mintel). The growing intake is likely due to the dynamic contrast between the texture and mouthfeel of FD and fresh fruits, heightening the pleasure associated with FD products (Hyde and Witherly 1993).

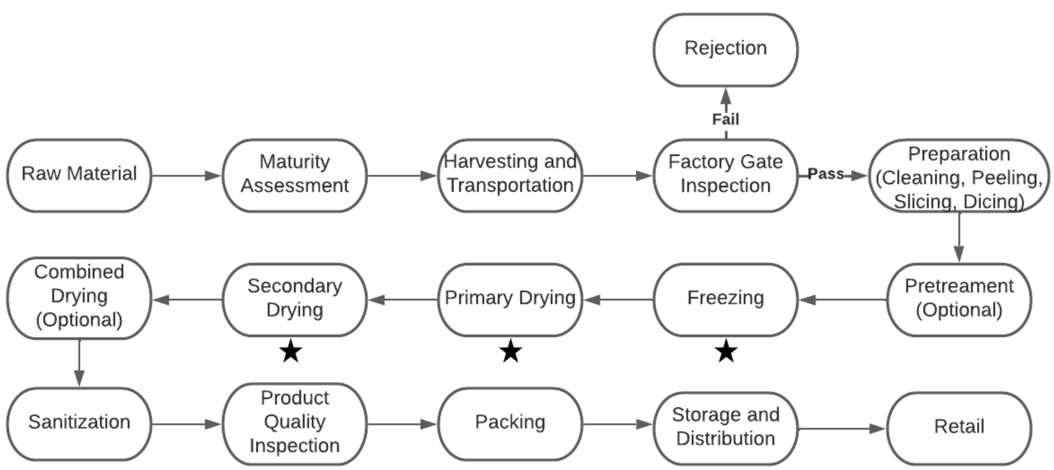

Figure 1. – General flow diagram of freeze-dried fruits manufacturing process from raw materials to packaged goods in retail. Three primary steps of freeze drying are highlighted with stars underneath. Pretreatment steps or combined drying methods can be applied to improve the product quality and drying efficiency.

2. REVIEW OF LITERATURE

2.1 Effects of freeze drying on nutritional profile

2.1.1 Total phenolic content

Phenolic compounds are a large group of antioxidants found in fruits, vegetables, seeds, and a variety of plants. Phenolics are considered beneficial for health due to their therapeutic and preventive roles against several health conditions, including aging, inflammation, and certain cancers (Shofian et al. 2011). Both intrinsic factors such as varieties of fruits and ripeness, and extrinsic factors such as climate and post-harvest processing conditions have influences on the measured total phenolic compound content (Asami et al. 2004; Liliana et al. 2015).

Freeze drying causes the loss of total phenolic compounds because the crystals formed during the freezing step damages cell walls and releases oxidative and hydrolytic enzymes, degrading the phenolic compounds (Liliana et al. 2015). Work done on freeze-dried starfruit, mango, papaya, and watermelon showed a decrease in TPC of 24.08%, 23.19%, 39.72%, and 48.23%, respectively (Shofian et al. 2011). However, comparing with other dehydration processes such as sun drying, hot air drying, microwave drying and so on, freeze-drying preserves the total phenolic compound to the largest extent. Freeze-dried mango leathers contained total polyphenolic compounds of 7.2 ± 0.06 mg/g db, whereas sun-dried mango leathers had a significant lower TPC value (4.1 ± 0.03 mg/g db) (Sarkar et al. 2020). Freeze-dried black berry powder demonstrated 73% TPC retention, which is twice the value obtained on spray-dried black berry powders (Franceschinis et al. 2014). Lachowicz et al. (2019) demonstrated that freeze-dried saskatoon berries contained the highest level of measured polyphenolic compounds compared to samples dehydrated using microwave drying, convective drying, and combined drying (CD/MVD).

On the other hand, research on blueberries processed by freeze-drying suggests a higher total phenolic content compared to fresh blueberries. It is likely caused by the enhanced extraction of polyphenols instead of an actual increase in the TPC concentration (Reyes et al. 2011).

2.1.2 Carotenoid

Carotenoids are yellow, orange, red organic pigments widely distributed in fruits and vegetables (Mangels et al. 1993). Similar to phenolic compounds and ascorbic acids, carotenoids exhibit great antioxidant activity which makes it valuable compounds to be included in day-to-day diet (Shofian et al. 2011). However, like other antioxidants, carotenoids are unstable in nature, rendering them highly susceptible to oxidation and heat-induced degradation (Bhatta et al. 2020).

Freeze drying better preserves carotenoid content compared to other thermal processing techniques. A study conducted on the drying of Seabuckthorn berry illustrated that freeze drying preserved 93% more carotenoids compared to air drying. Higher drying temperature was also associated with higher carotenoid retention which is likely due to the reduced drying time and consequently reduced oxygen exposure (Araya-Farias et al. 2011). In another study on effects of different drying technology on the bioactive compounds of mango leather, Sarkar et al. (2020) stated that FD sample contained the highest total carotenoid content (205.10 ± 9.43 μg/100 g db) compared with samples dehydrated by sun drying (169.18 ± 7.55 μg/100 g), microwave drying (163.52 ± 9.06 μg/100 g), and hot air drying (102.50 ± 6.70 μg/100 g). Freeze drying preserved 69.9% carotenoids with respect to fresh mangos, which had a carotenoid concentration of 293.4 ± 5.98 μg/g db (Sarkar et al. 2020). The results were in line with other findings from the literature. Freeze dried mango showed a 71.4% carotenoid retention in relation to fresh fruits. While mangos dehydrated by air drying, vacuum drying, and conductive multi-flash drying had lower retention percentages of 35.1%, 50.1%, and 44.7%, respectively (Link et al. 2017).

2.1.3 Ascorbic acid

Ascorbic acid, also known as vitamin C, is a bioactive compound with antioxidant capacity commonly found in citrus and other fruits and vegetables. The loss of vitamin C is largely affected by temperature and moisture content during dehydration process (Araya-Farias et al. 2011). In general, high temperature treatments are associated with decreased ascorbic acid content due to degradation and deterioration (Liliana et al. 2015). Freeze drying, as a low temperature processing technique, has been demonstrated to maximize ascorbic acid preservation by several studies.

Tylewicz et al. (2020) compared the vitamin C content of kiwi leathers and snack bars processed by hot air drying and freeze-drying and found that freeze-dried samples demonstrated greater retention of vitamin C. A study on the effects of freeze-drying on antioxidant compounds of five tropical fruits, namely starfruit, mango, papaya, muskmelon, and watermelon, concluded that there was no significant ($p > 0.05$) difference in the ascorbic acid content of the fresh and freeze-dried fruits (Shofian et al. 2011). The vitamin C content of FD alpine strawberries (51 mg/ 100 g) had no significant difference from the vitamin C content of fresh samples (50.7 mg/ 100 g) (Yurdugül 2008). Guava processed by freeze-drying demonstrated 63% vitamin C retention which is more desirable compared to 25% retention achieved by heat pump dryer (Bhatta et al. 2020). Freeze-dried guanabanas showed 50% and 66.4% ascorbic acid retention under rapid and slow freezing conditions, respectively. Yet, vitamin C content of freeze-dried blueberries was significantly reduced up to 60.43% compared to fresh blueberries, regardless of different freeze drying operating conditions (Reyes et al. 2011). It is important to note that the measured values of vitamins are also dependent on other factors including fruit maturity and different extraction methods used (Shofian et al. 2011).

2.2 Effect of freeze drying on physical properties

2.2.1 Color

Color is a critical factor to consider in dehydrated foods since it is viewed as an indicator of product quality (Francis 1995). Color variation in fruits can be caused by the Maillard reaction, color pigment degradation, enzymatic browning, or vitamin C oxidation. Many studies have proven that drying method has great influence on the color parameters of dried fruits (Liliana et al. 2015; Rudy et al. 2015).

Freeze-dried cranberries showed significantly increased brightness and redness, and yellowness is stable or slightly increased with respect to fresh samples. The study also stated a positive correlation between drying temperature and the lightness, redness, and color intensity of cranberries (Rudy et al. 2015). Similar study on the effect of freeze drying on the color of osmotically dehydrated cranberries proved that there was no significant difference between the freeze-dried and fresh (frozen-thawed) samples (Beaudry et al. 2004). Lightness (L*), red/green chroma (a*), and yellow/blue chroma (b*) are color parameters used to evaluate color variations in most studies. Values of these parameters of freeze-dried alpine strawberries ($L^* = 58$, $a^* = 23$, $b^* = 26$) were not significantly different from the values of fresh fruits ($L^* = 57$, $a^* = 24$, $b^* = 26$), indicating maximum color preservation by freeze drying (Yurdugül 2008). Study done on the color change of freeze-dried strawberry, apple, and pear discovered that pear was the only fruit to experience severe discoloration (Khalloufi and Ratti 2003). However, kinetic color analysis of freeze-dried pumpkin showed decreased chroma, but the hue angle was well preserved when compared with fresh counterparts (Guiné and Barroca 2012).

Compared to conventional drying methods, freeze drying also supports less color deterioration on fruits (Duan et al. 2016). Freeze-dried hawthorn fruit powder demonstrated a higher red (a*) value compared to hot-air-dried sample and was more similar to the color of fresh hawthorn fruit (Liu et al. 2020). The color change of freeze-dried green pepper was little compared to samples dried under high temperature hot air drying (Guiné and Barroca 2012). Nevertheless, the state of the product and its detailed composition should also be taken into account when considering the effects of drying on color variations (Khalloufi and Ratti 2003).

2.2.2 Structure

Collapse is an undesirable structural change happens when the viscosity of a product decreases beyond the glass transition temperature (Tg). When the temperature of a porous product is increased above the glass transition temperature, the viscosity of the solid material can no longer support its own structure, causing collapse, contraction, and eventually the loss of microscopic structure. Products with collapsed structure are described as harder, have reduced aroma, and have compromised rehydration capacity. Collapse can also be defined as a decrease in volume or increase in apparent specific density (Harnkarnsujarit and Charoenrein 2011; Liliana et al. 2015).

Low temperature does not produce obvious shrinkage on freeze-dried materials. High drying temperature can cause negative effects on the humidity and viscosity of the product, leading to fast structural loss or collapse (Krokida et al. 1998). Freeze-dried mango showed a collapse phenomenon when frozen at -35 °C and dried at -15 °C, while mangoes frozen at the same temperature and dried at a lower temperature of -40 °C did not show sign of collapse (Harnkarnsujarit and Charoenrein 2011). Research on freeze-drying characteristics of pineapple, Barbados cherry, guava, papaya, and mango proved that minimum collapse was observed on the above mentioned FD tropical fruits

(Marques et al. 2006). Internal factors including composition and viscosity of different materials greatly affect the structural changes that happen during dehydration (Krokida et al. 1998).

Nonetheless, the capacity of freeze drying to retain the original structure of fruits is superior compared with conventional drying techniques. Freeze-dried mangoes demonstrated a high porosity value of 65.9 ± 3.4 and a low bulk density value of 0.42 ± 0.04, giving the fruits a crispy texture. While air drying, vacuum drying, and conductive multi-flash drying produced fruits with lower porosity values and higher bulk density values, indicating denser structures (Link et al. 2017). Freeze drying fresh jujube slices demonstrated smallest volume change and lowest apparent density with respect to samples dried using hot air drying and pressure-differential puffing drying, maximizing the retention of the microstructure of fresh fruits (Wang et al. 2021). Similar results were obtained from freeze-dried *Amomum villosum* fruits, which showed minimum tissue structural change compared to fruits dried by other techniques (Ai et al. 2022).

2.3 Environmental impact of freeze drying

Still, there are several disadvantages associated with freeze-drying, including high cost, long processing time, extensive energy consumption, and potential high level of residual microbes. Among the limitations, high energy consumption is the most concerning as it poses negative impact to the environment. Applications of pre-treatment and combination drying methods have been proposed to mitigate the disadvantages of freeze-drying (Li et al. 2014; Cao et al. 2018). Barley grass that underwent ultrasonic pre-treatment required less manufacturing time and energy to be freeze-dried compared to the untreated counterpart. The reduced drying time was likely due to the ruptured cell walls, promoting pore formation and subsequent faster sublimation. Use of UT in freeze drying decreased the energy consumption from 6.275 × 106 J/g to as low as 5.175 × 106

J/g (Cao et al. 2018). Combination of freeze-drying and microwave vacuum drying of okra showed reduced drying time, lower specific energy consumption, and improved drying rated with respect to freeze-drying alone. FD-MVD also promoted the retention of bioactive compounds, color, and texture of the dehydrated okra (Jiang et al. 2017). The results were supported by the conclusion from another study of freeze-dried apple slices. Samples treated with microwave after freeze-drying required 85-95% less processing time compared to the untreated samples (Li et al. 2014). Infrared-assisted freeze drying also reduced energy use by increasing drying rate and reducing drying time, and preserved the structural qualities of banana (Khampakool et al. 2019).

Table 1. – Drying parameters, chemical and nutritional content, antioxidant activities, structural and physical changes in freeze-dried fruits.

Fruit Product	**Drying Parameters**	**Chemical and Nutritional Content**	**Antioxidant Activities**	**Structural and Physical Changes**	**Reference**
Black berry powder	- 84 °C 0.04 mBar 48 hr	Acy - 162±5 TPC - 657±23	Antiradical activity - 78±7%	A_{w} - 0.19 ± 0.04 Bulk density - 0.45 ± 0.02 g/mL	(Franceschinis et al. 2014)
Mango leather	- 20 °C 0.1 mBar 20 hr	TPC - 7.2±0.06 mg/g db. TFC 3.27±0.22 - mg/g db. Carotenoid content - 205.1±9.43 μg/100 g db.	DPPH - 7.32±0.8% FRAP 8.02±0.69 - mg/100g db. ABTS - 81.03±3.92%.	Hardness - 3.03±0.09 N. MC - 14.31±1.17% ΔE = 4.97 ± 0.08	(Sarkar et al. 2020)
Okra	- 50 °C 20 Pa	TPC - 11.59±0.02 mg GAE/g d.w. TFC - 17.46±0.23	AEAC - 7.67 ± 0.02 mgVc/g d.w. FRAP - 16.76 ± 0.55 mgVc/g d.w.	Shrinkage - 24.76 ± 4.36% Hardness - 14.95 ± 1.05 N Crispness - 3.80 ± 0.59 N Smallest color difference.	(Jiang et al. 2017)

		mg Rutin/g d.w.	ABTS - 29.71 ± 0.69 mgVc/g d.w.		
Strawberries	- 80 °C 24 hr	Vitamin C - 51mg/100g Acy - 50.22mg/100g		Firmness - 0.56 N Color L* - 58 Color a* - 23 Color b* - 26	(Yurdugül 2008)
Watermelon	- 20 ±1 °C 36 hr	TPC - 15.18±2.95 Vitamin C - 2.38±0.11 β-carotene - 165.21±5.89	Lipid peroxidation inhibition - 41.2%	MC - 92.47±0.12%	(Shofian et al. 2011)

2.4 Future study

As implied in the previous sections, the preservation of bioactive compounds in fruits and vegetables does not only depend on the drying methods, but also is influenced by the composition of the product itself. Future studies can focus on determining the optimum processing parameters corresponding to different types of fruits to amplify the retention of these compounds. Advanced research can be done on the effects of freeze drying on different cultivars of the same type of fruit to determine the breed that is most well-preserved and suitable for producing functional snacks.

3. CONCLUSIONS

Freeze drying produces influences on the nutritional, physical, and chemical properties of fruits and vegetables. The specific effects depend on both factors inherent to the product and the external processing parameters. In general, freeze drying preserves the total phenolic content, vitamin c content, structure, color, and other qualities of fruits and vegetables to the largest extent as opposed to traditional thermal dehydration methods. The limitations of freeze drying, including long manufacturing time and high energy consumption, can be relieved by the application of pretreatment or combination drying methods. The information gathered can be useful for culinary use, especially in the bakery industry. Include a sentence or two regarding research opportunities that were informed by your review. Future research can focus on establishing a system of processing parameters corresponds to different types of fruits and vegetables that can maximize the retention of nutritional and physiochemical properties.

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