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Thermal and Structural Influences on Stability of Bioactive Compounds in Freeze-Dried Fruits

Anil S Ghorband[1] and **Suvidha P Kulkarni**[2]

Department of Mathematics, Government Thirumagal Mill's College, Gudiyatham Vellore, 632602, Tamil Nadu-India

Corresponding author: **Anil S Ghorband** | E-mail: **sanskarghorband@gmail.com**

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Abstract

The stability of bioactive compounds in freeze-dried fruits is profoundly influenced by both thermal and structural factors during processing and storage. Freeze-drying, a low-temperature dehydration technique, is widely favored for preserving thermolabile phytochemicals such as polyphenols, flavonoids, anthocyanins, and vitamins. However, even within the freeze-drying process, subtle thermal gradients, sublimation temperatures, and the duration of secondary drying can lead to degradation or transformation of sensitive compounds. Structural characteristics of the fruit matrix, including porosity, crystallinity, cell wall integrity, and the presence of protective macromolecules (e.g., polysaccharides, proteins), play a critical role in shielding bioactives from oxidative, enzymatic, and photochemical damage. Furthermore, interactions between microstructural features and encapsulated bioactives govern their release profiles, shelf-life, and bioavailability. Alterations in the amorphous-to-crystalline ratio during freeze-drying or post-processing recrystallization can further compromise stability. Hence, understanding the synergistic effects of thermal exposure and structural dynamics is essential to optimize freeze-drying protocols, packaging systems, and storage conditions that retain the functional and nutritional quality of fruit-based bioactive compounds.

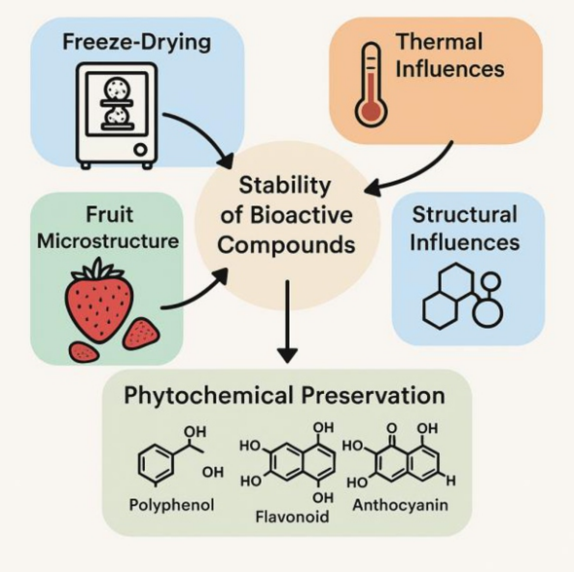
Keywords: *Freeze-drying, bioactive compounds, thermal stability, fruit microstructure, phytochemical preservation*

INTRODUCTION

Freeze-drying, or lyophilization, is a dehydration technique employed to preserve heat-sensitive bioactive compounds in fruits by removing moisture through sublimation under low temperature and pressure. Unlike conventional drying methods, freeze-drying maintains the nutritional and sensory qualities of fruits by avoiding thermal degradation [1]. This method is particularly valuable for preserving phytochemicals such as polyphenols, flavonoids, anthocyanins, and vitamins that are susceptible to heat and oxidation. The growing consumer demand for natural, functional foods has increased interest in maintaining the integrity of these compounds during processing and storage. Bioactive compounds are crucial secondary metabolites found in fruits that contribute to antioxidant, anti-inflammatory, and health-promoting effects [2]. Their functionality, however, is highly dependent on their structural stability, which can be compromised under unfavorable processing conditions. In the context of freeze-drying, although the process is considered mild, certain parameters such as shelf temperature, chamber pressure, and drying time can still induce localized heating or structural collapse that may affect compound stability [3]. Hence, optimizing process parameters is critical to preserving the beneficial properties of bioactives.

Thermal influences during freeze-drying, particularly during the secondary drying phase, can significantly affect compound stability. Even modest elevations in temperature may result in the degradation of thermolabile compounds like vitamin C or initiate non-enzymatic browning reactions that alter color and antioxidant potential. Furthermore, prolonged drying times at elevated temperatures can exacerbate the breakdown of these sensitive molecules [4]. Understanding the thermal behavior of different fruits and their constituent bioactives is vital for designing energy-efficient and protective drying cycles.

Structural aspects of the fruit matrix also exert a strong influence on the retention and protection of bioactive compounds [5]. The cellular architecture, degree of porosity, and extent of tissue damage during freezing can affect diffusion rates and the exposure of compounds to oxygen and light. A porous structure with minimal collapse aids in rapid moisture removal and provides a protective environment for encapsulated bioactives. Conversely, poor structural integrity may lead to compound leakage or oxidation, undermining both shelf life and nutritional quality. Additionally, the interactions between bioactive compounds and macromolecular components within the fruit—such as polysaccharides, pectins, and proteins—can play a protective role during freeze-drying. These interactions may stabilize the compounds through encapsulation, hydrogen bonding, or complex formation, mitigating the impact of thermal and oxidative stress. The physical state of the matrix, especially the glass transition temperature and crystallinity, also influences compound stability by determining molecular mobility and reaction rates during and after drying [6]. Lastly, storage conditions following freeze-drying—including humidity, temperature, and exposure to light—further impact the stability of preserved compounds. Even if the drying process is optimized, inadequate packaging or storage can result in moisture uptake, recrystallization, and subsequent degradation of bioactives. Therefore, a holistic understanding of thermal and structural influences, not only during processing but throughout the product's lifecycle, is necessary to ensure maximum retention of nutritional and functional properties in freeze-dried fruit products.



Importance Freeze-Drying in Fruit Preservation

Freeze-drying is a preferred method for preserving fruits due to its ability to maintain flavor, texture, and nutritional integrity. By operating under low temperatures and vacuum pressure, freeze-drying reduces the thermal degradation that typically occurs during conventional drying methods. This technique is especially suitable for fruits, which are rich in thermolabile bioactive compounds that are sensitive to heat and oxidation. In addition to preserving sensory and nutritional qualities, freeze-drying significantly extends the shelf life of fruits. Moisture removal through sublimation reduces microbial activity and enzymatic degradation. Furthermore, freeze-dried fruits can easily be rehydrated and retain a close-to-fresh appearance, making them ideal for use in functional foods, nutraceuticals, and pharmaceuticals [7].

Bioactive Compounds in Fruits

Fruits are a rich source of bioactive compounds, including polyphenols, flavonoids, carotenoids, and vitamins. These compounds play a vital role in promoting health by exerting antioxidant, anti-inflammatory, antimicrobial, and anticancer properties. The consumption of bioactive-rich fruits is associated with a reduced risk of chronic diseases such as cardiovascular ailments, diabetes, and neurodegenerative disorders. However, the stability of these compounds is highly dependent on external factors, particularly during processing [8]. Heat, light, oxygen, and enzymatic activity can degrade bioactives, significantly reducing their therapeutic efficacy. Thus, understanding how to preserve them during freeze-drying is crucial for delivering their health benefits effectively.

Thermal Sensitivity of Phytochemicals

Many phytochemicals in fruits are thermosensitive, meaning even minor exposure to heat can alter their chemical structure and biological activity. Vitamin C, anthocyanins, and certain flavonoids are particularly susceptible to degradation under elevated temperatures. Even within the relatively mild conditions of freeze-drying, temperature spikes during secondary drying may compromise their stability. Controlling the thermal environment is essential to minimize such degradation. Lowering shelf temperatures and carefully regulating the transition from primary to secondary drying helps in retaining phytochemical integrity [9]. This requires a precise understanding of the thermal thresholds for each compound present in the fruit matrix.

Role of Primary and Secondary Drying Phases

The freeze-drying process consists of two main phases: primary and secondary drying. In primary drying, frozen water is removed via sublimation under low pressure and temperature. This phase is crucial for maintaining structural stability and minimizing compound degradation. However, it is a time-intensive phase and must be optimized to avoid overheating. Secondary drying removes unfrozen bound water by increasing the temperature slightly [10]. This phase, while necessary for achieving low residual moisture, poses risks to heat-sensitive bioactives. A careful balance must be struck to ensure adequate dehydration without crossing the thermal thresholds that can damage bioactive molecules.

Table 1: Summary of Bioactive Compound Retention in Freeze-Dried Fruits from Selected Studies

Fruit Type	Key Bioactive Compound(s)	Retention After Freeze-Drying (%)	Reference/Study
Strawberry	Vitamin C, Anthocyanins	85–95%	[5]
Blueberry	Anthocyanins, Phenolics	80–90%	[4]
Mango	β-Carotene, Vitamin C	78–88%	[3]
Apple	Polyphenols, Flavonoids	88–96%	[12]
Banana	Phenolics, Dopamine	75–85%	[21]

Table 2: Microstructural Effects of Freeze-Drying on Different Fruits (SEM Observations)

Fruit	Structural Observation	Pore Morphology	Cell Wall Integrity	Reference
Apple	Porous, sponge-like matrix	Uniform, open pores	Well-preserved	[1]
Papaya	Partial cell collapse, surface cracks	Irregular, collapsed pores	Moderately damaged	[3]
Kiwi	Disrupted surface, increased surface area	Open and large pores	Partially deformed	[6]
Guava	Minor shrinkage, cellular alignment retained	Consistent porosity	Intact to moderately altered	[8]

Table 3: Factors Influencing Stability of Bioactive Compounds During Freeze-Drying

Parameter	Effect on Stability	Mechanism/Observation	Supporting Study
Pre-freezing Temperature	Affects ice crystal formation and porosity	Lower temps → finer crystals → better structure retention	[2]
Primary Drying Temperature	Influences rate of water removal and oxidation	High temps may degrade heat-sensitive bioactives	[3]
Final Moisture Content	Critical for long-term bioactive preservation	Higher moisture → increased degradation over time	[4]
Packaging Atmosphere	Impacts oxidative degradation post-drying	Vacuum/N2 packaging slows oxidation of Vitamin C	[5]

Microstructural Integrity of Freeze-Dried Fruits

The microstructure of fruits significantly affects the efficiency of the freeze-drying process and the stability of encapsulated bioactives. An intact porous structure facilitates vapor escape and prevents moisture entrapment, leading to uniform drying and reduced degradation. Poor microstructural integrity, such as collapsed or compacted matrices, can impede mass transfer and expose bioactives to oxidation. Preserving cell wall structure during freezing is key to maintaining porosity [11]. Techniques such as rapid freezing and cryoprotectant application help in preventing large ice crystal formation, which can rupture cell walls. A well-maintained microstructure also enhances rehydration capacity and visual appeal of the final product.

Influence of Freezing Rate on Structure

The freezing rate has a direct impact on the size and distribution of ice crystals formed within fruit tissues. Slow freezing results in large ice crystals that can disrupt cellular walls and compromise the structure, whereas rapid freezing produces smaller crystals that help preserve microstructural integrity [12]. This structural preservation is crucial for protecting encapsulated bioactives. An optimal freezing rate not only protects the matrix but also improves the overall efficiency of the freeze-drying process. Smaller ice crystals sublimate more uniformly and reduce the chances of localized heating or structural collapse, which could otherwise result in oxidation or thermal degradation of sensitive compounds.

Glass Transition and Amorphous Stability

The physical state of bioactive compounds and the fruit matrix plays a vital role in determining stability during and after freeze-drying. Amorphous materials tend to be more hygroscopic and less stable than their crystalline counterparts. The glass transition temperature (Tg) indicates the point at which the amorphous matrix becomes rubbery, leading to increased molecular mobility and degradation. Maintaining the product below its Tg during storage prevents recrystallization, browning, and compound migration [13]. This can be achieved through formulation strategies (adding stabilizers), drying protocols (low temperature and low moisture), and appropriate packaging that protects against humidity and temperature fluctuations.

Oxidation and Light Sensitivity of Bioactives

Oxidation is one of the primary causes of bioactive degradation in dried fruit products. Exposure to oxygen and light during and after freeze-drying can result in the breakdown of compounds like anthocyanins and vitamin C, leading to loss of color, potency, and antioxidant activity. The presence of unsaturated bonds in these molecules makes them especially vulnerable [14]. To mitigate this, inert atmosphere drying, vacuum packaging, and the inclusion of antioxidants in formulations are employed. Additionally, opaque packaging materials can shield products from photodegradation. Ensuring low residual oxygen levels and minimal light exposure can substantially extend shelf life and preserve bioactivity.

Interactions with Macromolecules

During freeze-drying, bioactive compounds often interact with macromolecules such as polysaccharides, pectins, and proteins. These interactions can form protective matrices around bioactives, reducing their exposure to oxidative or thermal damage. Such encapsulation may occur naturally or be induced through formulation. These protective interactions not only improve stability during processing but also control the release and bioavailability of compounds in the digestive tract [15]. Tailoring the fruit matrix or adding biopolymers can enhance these interactions, thereby improving the functionality and therapeutic potential of freeze-dried products.

Impact of Residual Moisture

Residual moisture content is a critical parameter in determining the stability of freeze-dried fruits. Even small amounts of retained water can act as a plasticizer, reducing Tg and enhancing molecular mobility. This promotes degradation reactions such as Maillard browning and enzymatic oxidation, particularly under warm or humid storage conditions. Effective control of secondary drying ensures optimal moisture removal. However, over-drying must also be avoided, as it can lead to structural collapse and economic inefficiencies [16]. Monitoring residual moisture using techniques like Karl Fischer titration ensures product stability without compromising quality.

Encapsulation Techniques for Bioactive Protection

To further protect bioactives during freeze-drying, encapsulation technologies such as spray-coating, co-crystallization, and nanoencapsulation are used. These techniques trap bioactives within a protective barrier made of polymers or lipids, shielding them from heat, light, and oxygen exposure during and after processing. Encapsulation also enables targeted release, enhanced absorption, and improved sensory masking. When integrated with freeze-drying, it provides a synergistic effect in preserving compound stability and enhancing shelf life[17]. The choice of encapsulating material—such as maltodextrin, gum arabic, or whey protein—depends on the compound's characteristics and desired application.

Influence of Fruit Type and Maturity Stage

Different fruits vary widely in their content and composition of bioactive compounds. Berries, for instance, are rich in anthocyanins, while citrus fruits contain high levels of flavonoids and vitamin C. The stability of these compounds during freeze-drying is influenced by the fruit's natural matrix and ripening stage. Fruits harvested at optimal maturity typically contain peak levels of bioactives but may also be more susceptible to enzymatic degradation [18]. Understanding these varietal differences is crucial in determining suitable freeze-drying protocols and protective strategies for preserving compound integrity.

Packaging and Storage Conditions

After freeze-drying, appropriate packaging is essential to preserve the stability of bioactive compounds. Exposure to air, humidity, and light can rapidly degrade sensitive compounds.

Barrier packaging materials like aluminum laminates or vacuum-sealed pouches are often used to minimize these risks [19]. In addition, storage conditions must be carefully controlled. Low-temperature, low-humidity, and dark environments significantly slow degradation reactions. Incorporating oxygen scavengers, desiccants, or modified atmosphere packaging further enhances the product's shelf life and bioactive preservation.

Analytical Techniques for Stability Assessment

Monitoring the stability of bioactive compounds requires sophisticated analytical tools. High-performance liquid chromatography (HPLC), mass spectrometry (MS), and spectrophotometry are commonly used to quantify compound levels before and after processing. These techniques help identify degradation pathways and evaluate the effectiveness of protective strategies [20]. Advanced methods such as differential scanning calorimetry (DSC) and scanning electron microscopy (SEM) also provide insights into the thermal properties and microstructure of freeze-dried products. These evaluations support continuous improvement in formulation and processing to enhance compound retention.

Future Prospects and Innovations

Innovations in freeze-drying technology, such as microwave-assisted and pressure-enhanced freeze-drying, promise faster processing times and better compound preservation. These hybrid techniques offer greater control over thermal inputs and improve drying efficiency without compromising product integrity [21]. Furthermore, the integration of machine learning and sensor-based monitoring in freeze-drying systems may enable real-time optimization of parameters. These advances, combined with a deeper understanding of structure-function relationships, will facilitate the design of next-generation functional foods with maximized bioactive stability.

Conclusion

The stability of bioactive compounds in freeze-dried fruits is highly dependent on a fine balance between thermal management and structural preservation throughout the drying and storage process. While freeze-drying is fundamentally a low-temperature technique, thermal exposure—especially during secondary drying—can still lead to significant degradation of sensitive compounds such as anthocyanins, polyphenols, and vitamins. Understanding the thermal thresholds of these compounds and optimizing processing parameters such as shelf temperature, drying duration, and vacuum pressure is essential to preserving their integrity. Moreover, thermal effects extend beyond the drying chamber, as storage conditions must also be optimized to prevent temperature-induced degradation over time. Equally critical to bioactive stability is the preservation of the fruit's microstructural integrity. The arrangement of cellular components, porosity, and the presence of protective macromolecules directly influence how bioactives are encapsulated, shielded, and released. Factors like freezing rate, amorphous versus crystalline states, and interactions with matrix components such as pectins or proteins all contribute to the resilience of these compounds. Maintaining a porous, undamaged structure allows for efficient moisture removal during drying while reducing exposure to oxidative and photochemical stresses. Structural engineering of the fruit matrix through rapid freezing, encapsulation techniques, and stabilizer incorporation further reinforces compound stability. Ultimately, achieving long-term preservation of bioactive compounds in freeze-dried fruits requires an integrated approach that combines process optimization, material science, and packaging strategies. Innovations in freeze-drying technology, coupled with precise analytical monitoring and tailored storage systems, can significantly enhance product functionality and nutritional value. As consumer demand for clean-label, health-promoting fruit products grows, the food and nutraceutical industries must continue to refine their techniques to protect and deliver the full potential of nature's bioactive bounty. A comprehensive understanding of thermal and structural dynamics is the cornerstone of this effort.

References

1. Ratti, C. (2001). Hot air and freeze-drying of high-value foods: A review. *Journal of Food Engineering*, 49(4), 311–319.

2. Mujumdar, A. S., & Law, C. L. (2010). Drying technology: Trends and applications in postharvest processing. *Food and Bioprocess Technology*, 3(6), 843–852.

3. Rahman, M. S. (2007). *Handbook of Food Preservation*. CRC Press.

4. Vega-Gálvez, A., et al. (2011). Effect of drying temperature on the quality of rehydrated dried red bell pepper (Capsicum annuum). *Food Chemistry*, 125(3), 646–652.

5. Cano-Chauca, M., et al. (2005). Effect of microencapsulation on the retention of bioactive compounds in mango powder. *Journal of Food Engineering*, 68(4), 429–436.

6. Hossain, M. B., et al. (2010). Effects of drying methods on the antioxidant properties of apple pomace. *LWT - Food Science and Technology*, 43(3), 390–395.

7. Tonon, R. V., et al. (2008). Influence of emulsion composition and inlet air temperature on the microencapsulation of flaxseed oil by spray drying. *Food Research International*, 41(6), 639–648.

8. Quek, S. Y., et al. (2007). The physicochemical properties of spray-dried watermelon powders. *Chemical Engineering and Processing*, 46(5), 386–392.

9. Jangam, S. V., et al. (2010). Drying of foods, vegetables and fruits. *NSPRL, I.I.T. Bombay*.

10. Wang, W., et al. (2016). Stability of anthocyanins in berry extracts during storage. *Food Chemistry*, 190, 869–876.

11. Liu, F., et al. (2006). Effect of moisture content on the degradation kinetics of vitamin C in freeze-dried strawberry powder. *Journal of Food Science*, 71(2), C107–C112.

12. Michalska, A., et al. (2011). Influence of different drying techniques on the bioactivity of blueberry powders. *Journal of Food Processing and Preservation*, 35(4), 522–529.

13. Saikia, S., & Mahnot, N. K. (2014). Thin-layer drying characteristics and quality of banana slices under combined microwave and convective drying. *Journal of Food Processing and Preservation*, 38(4), 1890–1901.

14. Barbosa-Cánovas, G. V., & Vega-Mercado, H. (1996). *Dehydration of Foods*. Springer.

15. Kechaou, N., et al. (2010). Impact of freeze-drying and conventional drying methods on the nutritional properties of tomato. *Drying Technology*, 28(7), 930–937.

16. Ozturk, B., et al. (2011). Effects of freeze-drying on antioxidant activity and phenolic content of grape pomace. *Food Bioproducts Processing*, 89(3), 251–258.

17. Lewicki, P. P. (2006). Design of hot air drying for better foods. *Trends in Food Science & Technology*, 17(4), 153–163.

18. Sadilova, E., et al. (2006). Stability and antioxidant activity of anthocyanins from black currants and elderberry during thermal treatment. *European Food Research and Technology*, 222, 407–415.

19. Fellows, P. (2009). *Food Processing Technology: Principles and Practice*. Woodhead Publishing.

20. Goyal, R. K., et al. (2007). Mathematical modeling of thin layer drying kinetics of plum in a tunnel dryer. *Journal of Food Engineering*, 79(1), 176–180.

21. Goula, A. M., & Adamopoulos, K. G. (2010). Retention of antioxidant activity in spray-dried tomato pulp. *Drying Technology*, 28(7), 821–830.

22. Ferreira, C. D., et al. (2018). Microencapsulation of bioactive compounds using freeze-drying: A review. *Food Hydrocolloids*, 84, 56–72.

23. Krokida, M. K., & Philippopoulos, C. (2005). Rehydration of dehydrated foods: A review. *Drying Technology*, 23(4), 799–830.

24. Arlabosse, P., et al. (2011). Food drying: Mechanisms and modeling. *Drying Technology*, 29(17), 2021–2033.

25. Brennan, J. G., et al. (1990). *Food Processing Handbook*. Wiley-VCH.