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Development, evaluation, and kinetic modeling of blueberry (*Vaccinium corymbosum* L.) freeze-drying considering osmotic pretreatments and freezing methods

Desarrollo, evaluación y modelado cinético de la liofilización de arándano (*Vaccinium corymbosum* L.) considerando pretratamientos osmóticos y métodos de congelación

C. Michel-Cuello[1]*, J.L. Luna-Gallegos[1], N. Aguilar-Rivera[2], C. López-Palacios[1]

[1] *Universidad Autónoma de San Luis Potosí, Facultad de Estudios Profesionales Zona Media, Carretera Rioverde–San Ciro de Acosta Km 4, Ejido Puente del Carmen, 79617 Rioverde, San Luis Potosí, México.*
[2] *Universidad Veracruzana, Facultad de Ciencias Biológicas y Agropecuarias, Región Orizaba–Córdoba, Camino Peñuela–Amatlán s/n, Peñuela, Amatlán de los Reyes, 94945, Veracruz, México.*
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Abstract

Blueberries (*Vaccinium corymbosum* L.) that do not meet export standards offer potential for valorization through advanced dehydration processes. This study developed, evaluated, and kinetically modeled a freeze-drying process for blueberries, incorporating osmotic dehydration (OD) with sucrose solutions at 50, 60, and 70% (w/v), along with conventional freezing and liquid nitrogen ultrafreezing. The effects on water loss, moisture evolution, and drying efficiency were analyzed. OD was performed at 45 °C, followed by freeze-drying at 0.420 mbar and -51 °C. No significant differences were observed among sucrose concentrations during the initial OD stages; however, the 70% solution enhanced water loss at later stages, requiring more time to approach osmotic equilibrium. Water loss evolution during OD and freeze-drying was successfully described using empirical kinetic models, with the Midilli–Küçük model providing the best fit. Final moisture content showed no statistically significant differences among treatments, indicating that freeze-drying is the dominant unit operation governing overall dehydration. Liquid nitrogen ultrafreezing did not offer clear advantages over conventional freezing. These findings demonstrate that simple, low-cost osmotic pretreatments combined with freeze-drying represent a viable strategy for valorizing off-grade blueberries.
Keywords: blueberry, freeze-drying, kinetic modeling, osmotic dehydration, moisture loss.

Resumen

Los arándanos (*Vaccinium corymbosum* L.) que no cumplen con los estándares de exportación representan una oportunidad de valorización mediante procesos de deshidratación. En este estudio se desarrolló, evaluó y modeló cinéticamente un proceso de liofilización, incorporando deshidratación osmótica (DO) con soluciones de sacarosa al 50, 60 y 70% (p/v), así como congelación convencional y ultracongelación con nitrógeno líquido. Se analizaron sus efectos sobre la pérdida de agua, la evolución de la humedad y la eficiencia de secado. La DO se realizó a 45 °C, seguida de liofilización a 0.420 mbar y -51 °C. No se observaron diferencias significativas entre concentraciones de sacarosa en etapas iniciales; sin embargo, la solución al 70% favoreció mayor pérdida de agua en etapas posteriores, requiriendo más tiempo para alcanzar el equilibrio osmótico. La evolución de la pérdida de agua se describió mediante modelos cinéticos empíricos, destacando el modelo de Midilli–Küçük como el de mejor ajuste. El contenido final de humedad no mostró diferencias significativas entre tratamientos, indicando que la liofilización es la operación dominante. La ultracongelación con nitrógeno líquido no presentó ventajas frente a la congelación convencional. Estos resultados muestran que pretratamientos osmóticos simples, combinados con liofilización, son una estrategia viable para valorizar arándanos fuera de norma.
Palabras clave: arándano, liofilización, modelado cinético, deshidratación osmótica, pérdida de humedad.

*Corresponding author. E-mail: christian.michel@uaslp.mx ;
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1 Introduction

Blueberry (*Vaccinium corymbosum* L.) is a high-value fruit due to its nutraceutical profile, particularly its content of anthocyanins and phenolic compounds, as well as its desirable sensory attributes. These characteristics have driven the development of postharvest processing technologies aimed at preserving fruit quality and extending shelf life (Ma *et al*., 2023; Donno *et al*., 2025). However, a considerable portion of the harvest does not meet the dimensional or aesthetic standards required by export markets. As a result, transforming these off-specification fruits through preservation processes represents a viable strategy to reduce losses and generate added value (Ratti, 2001; Uribe *et al*., 2024).

Freeze-drying is one of the most effective technologies for preserving highly perishable fruits, as it removes water by sublimation at low pressure, thereby maintaining structural integrity and promoting the retention of bioactive compounds compared with conventional drying methods (Ciurzyńska and Lenart, 2011; Prawiranto *et al*., 2019; Bhatta *et al*., 2020; Stratta *et al*., 2020; Hu *et al*., 2025). Nevertheless, its industrial application involves long processing times and high energy costs, which has motivated the evaluation of pretreatments aimed at enhancing mass transfer and reducing drying time (Ratti, 2013; Egas-Astudillo *et al*., 2021; Buceta *et al*., 2022; Yao *et al*., 2023).

The freezing step is a critical factor in freeze-drying, as it controls the formation of the product's porous network and the resistance to mass transfer during sublimation; several studies have demonstrated that the freezing rate and method directly influence drying kinetics and process reproducibility (Nowak and Jakubczyk, 2020).

Among these pretreatments, osmotic dehydration (OD) is widely employed because it promotes partial water removal through immersion in concentrated solute solutions. Its efficiency depends on variables such as osmotic agent concentration and temperature, which directly influence the kinetics of water loss and solid gain (Vega-Gálvez *et al*., 2012; Ghellam *et al*., 2021; González-Pérez *et al*., 2021). In berries, high sucrose concentrations may increase initial water loss but can also prolong the time required to reach osmotic equilibrium due to increased medium viscosity and reduced diffusion rates (Kucner *et al*., 2012; Mari *et al*., 2024).

The integration of OD prior to freeze-drying has shown mixed results. While some studies report reduced drying times and improved structural stability, in small fruits such as blueberries, these benefits do not always translate into significant improvements during freeze-drying (García-Noguera *et al*., 2010; Prosapio and Norton, 2017; Yang *et al*., 2021). Additionally, the freezing method used before freeze-drying influences ice crystal formation and, consequently, the resistance to mass transfer during primary drying (Ratti, 2001; Pobiega *et al*., 2023; Nakagawa, 2025). However, in small fruits, the differences between liquid-nitrogen ultrafreezing and conventional freezing tend to be minimal (Ciurzyńska and Lenart, 2010; Cheng *et al*., 2014; Munzenmayer *et al*., 2020; Semenov *et al*., 2021; Faber *et al*., 2025).

Given these considerations, it is essential to evaluate comprehensively the combined effects of osmotic dehydration and different freezing methods on efficiency, particularly when processing fruits that do not meet commercial quality specifications (Prosapio and Norton, 2017; Rajashree *et al*., 2024).

Despite extensive studies on osmotic dehydration and freeze-drying, the combined effects of pretreatment intensity and freezing conditions on mass transfer behavior and overall process performance in small fruits remain not fully understood. In this context, the present study contributes to the understanding of combined dehydration processes by: (i) evaluating the coupled effect of osmotic dehydration and freezing methods on freeze-drying performance in small fruits; (ii) providing a kinetic and mechanistic interpretation of water-loss behavior under different osmotic conditions; and (iii) identifying the limited effectiveness of intensive pretreatments, such as high sucrose concentrations and ultrafast freezing, when freeze-drying acts as the dominant unit operation. These findings provide relevant insights for the design and optimization of dehydration processes for the valorization of off-grade fruits.

Additionally, an interpretation of the process is proposed from a mass transfer perspective, considering the interactions between water loss and solid gain, as well as their influence on overall dehydration kinetics and subsequent freeze-drying performance.

This study aimed to develop and evaluate a freeze-drying process for blueberry (*Vaccinium corymbosum* L.) using osmotic dehydration at different sucrose concentrations (50, 60, and 70% w/v) and distinct freezing methods (conventional freezing and liquid nitrogen ultrafreezing), analyzing their effects on moisture loss and drying efficiency.

2 Materials and methods

2.1 Plant material

Blueberry fruits (*Vaccinium corymbosum* L.), Biloxi variety, were provided by the company Agrizom S.C. de R.L. de C.V., located in the community of

La Reforma, Ciudad Fernández, San Luis Potosí, México. The fruits displayed commercial maturity, characterized by uniform blue color, smooth and firm texture, and an average diameter between 1 and 2 cm-typical attributes reported for fruits intended for fresh consumption and industrial processing (Kucner *et al.*, 2012; Yang *et al.*, 2021). This stage of commercial maturity is considered equivalent to physiological maturity, at which blueberries reach stable physicochemical properties suitable for postharvest processing. Under these conditions, the fruits presented an average soluble solids content of 30.6 °Brix, measured using a digital refractometer (Lab Digital Refractometer - Salinity, Sper Scientific 30003), a pH of 3.95 determined with a digital pH meter (Ohaus Starter 3100), and a moisture content of 75.5%, evaluated by a thermogravimetric method using a moisture analyzer (Ohaus MB 23). Titratable acidity was 0.65%, determined by acid–base titration and expressed as citric acid equivalents, within the reference range reported for fully mature blueberries, confirming their suitability for dehydration and freeze-drying studies.

Although the fruits were suitable for consumption, they did not meet the dimensional standards required for international markets. After harvest, blueberries were transported under refrigeration and stored at 4 °C until conditioning, with the aim of preserving physicochemical quality and minimizing metabolic alterations prior to processing-an approach commonly recommended for high-respiration berries (Ratti, 2001; Ciurzyńska and Lenart, 2011; Kalt *et al.*, 2020).

2.2 Conditioning of plant material

The fruits were washed with chilled distilled water (4 °C) to remove surface impurities. They were then disinfected by immersion in a cold aqueous solution (4 °C) containing a commercial silver-colloid-based antimicrobial agent for 10 minutes, following procedures commonly used to reduce microbial load without affecting tissue integrity (Ciurzyńska and Lenart, 2011).

After disinfection, the fruits were rinsed with distilled water and surface-dried using absorbent paper. Firm blueberries free of mechanical damage were manually selected. Samples were packed in airtight bags in 600 g portions and stored at 0 °C in a refrigerator–freezer unit (model GBMLOGAX, General Electric, Guanajuato, Mexico). All operations were carried out as rapidly as possible to maintain cold-chain continuity, a critical condition for limiting enzymatic and structural changes prior to freezing and freeze-drying (Ratti, 2001; Semenov *et al.*, 2021).

2.3 Experimental conditions

2.3.1 Pretreatments

Before freeze-drying, different pretreatments were applied in order to modify mass transfer properties, considering the associated effects on fruit microstructure as reported in the literature following what has been reported for combined drying systems (Prosapio and Norton, 2017; Mari *et al.*, 2024).

2.3.1.1 Osmotic dehydration

Osmotic dehydration (OD) was performed using sucrose solutions (commercial sugar) at concentrations of 50, 60, and 70% (w/v), levels commonly applied to fruits with high moisture content and semipermeable skin (Vega-Gálvez *et al.*, 2012; González-Pérez *et al.*, 2021). For each treatment, 100 g blueberry samples were immersed in 300 mL of osmotic solutions prepared to final concentrations of 50, 60, and 70 % (w/v), corresponding to 300, 450, and 700 g of sucrose per 300 mL of solution, respectively. The selected concentrations and sample-to-solution ratio fall within ranges reported to promote effective osmotic gradients in fruits with semipermeable skins (Vega-Gálvez *et al.*, 2012; Yadav and Singh, 2014; Silva-Espinoza *et al.*, 2020; Silva-Espinoza *et al.*, 2021).

The process was conducted at 45 °C with constant agitation at 125 rpm for the experimental duration. These conditions enhance osmotic gradients and water diffusion without causing significant thermal damage to the fruit tissue (Kucner *et al.*, 2012; González-Pérez *et al.*, 2021; Mari *et al.*, 2024). The solutions were maintained at a controlled temperature using a magnetic stirring hot plate (Thermo Scientific, model TF40000064, China).

The osmotic dehydration time was limited to 8 h, as water loss tended to stabilize beyond this point for all sucrose concentrations evaluated.

2.3.1.2 Ultrafreezing

For the ultrafreezing pretreatment, approximately 98 g of blueberries were directly immersed in liquid nitrogen (-196 °C) for 2 min using a 4 L insulated container filled with liquid nitrogen. Liquid nitrogen was applied in excess as a cryogenic medium, without establishing a fixed fruit-to-nitrogen mass ratio, in order to ensure rapid heat removal and near-instantaneous freezing of the fruit tissue. This procedure was designed to guarantee a high freezing rate and homogeneous temperature distribution throughout the samples, rather than to control a specific quantitative ratio between the material and the cryogen.

Rapid freezing under these conditions favors the formation of small and uniformly distributed ice crystals, which may influence the resulting pore structure and the resistance to mass transfer during the subsequent sublimation stage. Previous studies have shown that, when liquid nitrogen is used in excess volume, the exact amount of cryogen is not a limiting factor, provided that it ensures complete and uniform freezing of the product (Assegehegn *et al*., 2019; Nowak and Jakubczyk, 2020).

Ultrarapid freezing can generate smaller ice crystals, with potential effects on porosity and diffusional pathways during sublimation, as reported in studies of rapid freezing and freeze-drying of small fruits (Ratti, 2001; Kono *et al*., 2017; Semenov *et al*., 2021). After ultrafreezing, samples were stored at 0 °C until freeze-drying.

2.3.1.3 Conventional freezing

The conventional-freezing pretreatment consisted solely of storing the blueberries at 0 °C without osmotic or ultrafreezing treatments. This method served as the control, as it is commonly used industrially before freeze-drying (Ciurzyńska and Lenart, 2011; Munzenmayer *et al*., 2020; Al-Faruq *et al*., 2025).

2.3.2 Freeze drying

Freeze drying was carried out using a Labconco FreeZone 2.5 laboratory freeze dryer (Labconco, Kansas City, MO, USA) equipped with a vacuum pump model 117 (A65312906, Labconco). Operating conditions were set to a chamber pressure of 0.420 mbar and a condenser temperature of -51 °C, values within the recommended ranges for high-moisture, thermally sensitive fruits (Ratti, 2001; Prosapio and Norton, 2018; Bhatta *et al*., 2020). Under these pressure conditions, ice sublimation during primary drying occurs at an estimated product temperature of approximately -30 °C, according to the vapor pressure–temperature equilibrium of ice, which is consistent with values commonly reported for freeze-drying of high-moisture food materials.

Blueberries previously subjected to the pretreatments were placed in 300 mL drying flasks (Labconco), which were directly mounted onto the freeze-dryer drying chamber to ensure homogeneous heat and mass transfer during ice sublimation.

Tray Design and Fabrication for Freeze-Drying

During freeze-drying, the fruit load tended to shift and overlap, reducing interstitial free volume and increasing mass-transfer resistance-a phenomenon widely documented in drying systems involving spherical particles (Ratti, 2001; Mousavi *et al*., 2007; Nakagawa, 2025). This effect is associated with the small size and nearly spherical geometry of blueberries, as well as the 45° inclination of the drying flasks inside the freeze-dryer chamber.

To improve drying efficiency, an internal tray was designed to maintain uniform separation between fruits. The tray was modeled using Fusion 360 (Autodesk®, San Rafael, CA, USA) and manufactured with a Flashforge Creator Pro 3D printer (ZheJiang, China). The material used was polylactic acid (PLA), a biodegradable polymer derived from corn starch, selected for its stability at low temperatures, safety, and food-contact compatibility (Jamshidian *et al*., 2010). Dimensions were 11.5 cm (length) × 4.9 cm (width) × 2.4 cm (height), allowing ~0.1 cm spacing between fruits. Each level held up to 65 blueberries, for a total of 130 per tray. Reduced fruit-to-fruit contact decreases external vapor-flow resistance and improves drying uniformity, particularly in spherical or irregularly shaped materials (Mujumdar, 2015; Yao *et al*., 2023; Nakagawa, 2025).

2.3.3 Experimental design and statistical analysis

Five treatments were evaluated: three OD pretreatments (50, 60, and 70% w/v sucrose), one ultrafreezing treatment, and one conventional-freezing control. The selection of several osmotic concentrations and freezing methods was based on their influence on mass transfer, plant-tissue microstructure, and drying kinetics in combined dehydration/freeze-drying processes (Prosapio and Norton, 2017; González-Pérez *et al*., 2021). All samples were subsequently freeze-dried under identical conditions to comparatively assess pretreatment effects, following approaches commonly used in unit-operation evaluation studies (Ratti, 2001; Ciurzyńska and Lenart, 2011).

A completely randomized design was used, with five treatments, each performed in duplicate. Data were analyzed via ANOVA, followed by Tukey's multiple comparison test at $p \leq 0.05$, according to standard procedures for experimental design and statistical analysis (Montgomery, 2017). Statistical analyses were conducted using InfoStat software (version 2016e, Córdoba, Argentina), and graphical representations were created using SigmaPlot 12.3 (Systat Software Inc., San José, CA, USA).

Each treatment was performed in two independent biological replicates ($n = 2$). Residuals were evaluated for normality and homoscedasticity prior to ANOVA. The statistical model considered pretreatment as a fixed factor.

The experimental design was structured in a sequential, non-factorial manner, in which osmotic dehydration and freezing methods were evaluated as independent process steps prior to freeze-drying; therefore, interactions between osmotic dehydration and freezing were not considered, as the objective was to assess the isolated effect of each stage on water-loss kinetics and drying performance.

The moisture content of freeze-dried blueberries was determined using a moisture analyzer (MB90, Ohaus Corporation, Parsippany, NJ, USA). Approximately 1 g of dried sample was evenly distributed on an aluminum weighing pan and subjected to drying at 100 °C for 15 min. This temperature was selected according to standard methodologies for dehydrated foods, allowing the removal of free and partially bound water (AOAC, 2019). Water activity (a_w) was measured using an Aqualab Series 3 water activity meter (Decagon Devices, Inc., Pullman, WA, USA). Prior to measurements, the instrument was calibrated using lithium chloride standards at 13.41 mol/kg LiCl in H_2O ($a_w = 0.250$) and 8.57 mol/kg LiCl in H_2O ($a_w = 0.500$). The instrument has a stated measurement uncertainty of ±0.001 a_w.

3 Results and discussion

Drying curves constitute a fundamental tool to describe water removal kinetics and to analyze the efficiency of dehydration processes in plant matrices, as shown by Botello-Álvarez *et al.* (2026) in the dehydration of broccoli residues, a behavior that is comparable to that observed in the present study of freeze-dried blueberries subjected to different pretreatments.

3.1 Osmotic dehydration effects

During osmotic dehydration, water loss (WL) was quantified based on the relative variation of fruit mass as a function of time, according to Equation (1),

$$WL(t) = (m_0 - m_t)/m_0 \tag{1}$$

where m_0 corresponds to the initial mass of the blueberry and m_t to the mass at time t. This dimensionless formulation allowed the evolution of water removal to be described independently of the initial sample size. Based on the experimental WL(t) values calculated using Eq. (1), the water-loss kinetics were evaluated using empirical thin-layer drying models commonly applied to food dehydration processes. Both the Page and Midilli-Küçük models were tested, and the most appropriate model for each pretreatment was selected based on goodness-of-fit criteria. The model is expressed as Equation (2):

$$WL(t) = a\left[1 - \exp\left(-kt^n\right)\right] + bt \tag{2}$$

where *a, k, n*, and *b* are kinetic parameters estimated by non-linear regression. The inclusion of the linear term *b t* enabled a more accurate representation of the final stage of the process, justifying the selection of this model to describe the effect of osmotic concentration on the water-loss kinetics of blueberries.

These results are consistent with previous studies on fruits and plant based products, where the Midilli–Küçük model has demonstrated superior performance compared to simpler models in osmotic dehydration and drying processes (Kucuk *et al.*, 2014; Buzrul, 2022).

Based on the stabilization of water-loss kinetics, an osmotic dehydration time of 8 h was selected as pretreatment for the subsequent freeze-drying experiments. The explicit definition of the osmotic solution preparation and the equations used to calculate water loss enable a clear kinetic interpretation of the pretreatment stage and ensure the reproducibility of the experimental procedure.

The experimental water loss data (WL, $g \cdot g^{-1}$) obtained during the dehydration were fitted by nonlinear least-squares regression to empirical models commonly used to describe food drying kinetics. The Page and Midilli-Küçük models were evaluated in their water-loss formulations, and the most appropriate model was selected based on the coefficient of determination (R^2) and parsimony criteria, following recommendations reported for thin-layer drying models (Kucuk *et al.*, 2014; Buzrul, 2022).

For the treatments with OD at 50, 60, and 70 %, as well as for conventional freezing, the modified Page model Equation (3):

$$WL(t) = a\left[1 - \exp\left(-kt^n\right)\right] \tag{3}$$

provided the best fits, with R^2 values higher than 0.998, indicating excellent predictive capability of the experimental behavior. In contrast, for the liquid nitrogen ultrafreezing pretreatment, the Midilli-Küçük model Equation (4):

$$WL(t) = a\left[1 - \exp\left(-kt^n\right)\right] + bt \tag{4}$$

yielded the highest R^2 value, reflecting the influence of cryogenic pretreatment on mass transfer kinetics. The estimated kinetic parameters, together with the selected model and R^2 values for each pretreatment, are comparatively presented in Table 1. Model fitting was performed using the Python programming language through the curve fit function of the SciPy module, and statistical evaluation was carried out using the scikit-learn library to calculate the coefficient of determination, an approach widely accepted in kinetic modeling studies of agro-industrial processes due to its reproducibility and numerical robustness (Pedregosa *et al.,* 2011; Virtanen *et al*., 2020).

Table 1. Fitting of empirical models describing water-loss kinetics during freeze-drying of blueberries subjected to different pretreatments.

Pretreatment	Selected model	a (±EE) [g·g^{-1}]	k (±EE) [h^{-n}]	n (±EE) [-]	b (±EE) [h^{-1}]	R^2
OD 50 %	Page-WL	0.7938 ± 0.0083	0.20690 ± 0.00593	0.8164 ± 0.0250	-	0.99938
OD 60 %	Page-WL	0.7526 ± 0.0102	0.19502 ± 0.00805	0.8425 ± 0.0347	-	0.99887
OD 70 %	Page-WL	0.7382 ± 0.0035	0.15437 ± 0.00713	1.0462 ± 0.0308	-	0.99929
Liquid nitrogen ultrafreezing	Midilli–Küçük-WL	1.3000 ± 6.2739*	0.07615 ± 0.30078	0.8220 ± 0.22129	-0.002739 ± 0.07881	0.99255
Conventional freezing	Page-WL	0.7338 ± 0.0235	0.06520 ± 0.00272	1.0708 ± 0.0300	-	0.99993

Water loss (*WL*) was used as a proxy variable to describe moisture removal kinetics, as it directly reflects the relative reduction in sample mass associated with water evaporation and sublimation.

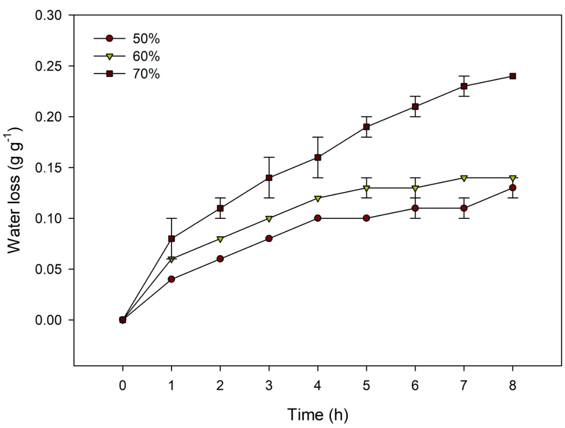

Figure 1. Dehydration kinetics (water loss) of blueberries subjected to osmotic dehydration at 50, 60, and 70 % (w/v) sucrose. Values represent mean ± standard deviation (n = 2). Statistical differences among treatments, when discussed, were evaluated by one-way ANOVA followed by Tukey's multiple comparison test ($p \leq 0.05$).

These results are consistent with previous studies on blueberries and other berry fruits, where the Page and Midilli–Küçük models have demonstrated superior performance compared to simpler models in drying and freeze-drying processes (Pavkov *et al*., 2019; Rurush *et al*., 2022).

Figure 1 shows the dehydration kinetics of blueberries subjected to osmotic dehydration (OD) in sucrose solutions at 50, 60, and 70% (w/v). In all treatments, the curves exhibit a characteristic non-linear behavior, with a rapid initial stage followed by a gradual reduction in the rate of water loss, which is typical of diffusion-controlled processes in plant tissues. During the first 4 h of pretreatment, no statistically significant differences ($p > 0.05$) were observed among treatments, with an average moisture loss of approximately 50%. This behavior agrees with Prosapio and Norton (2017), who indicate that, in the initial stages of OD, mass transfer is governed primarily by internal resistance within plant tissue and by cellular integrity, which limits the influence of the external osmotic gradient. In this stage, the similarity among curves suggests that the permeability of the cellular structure and intracellular diffusion are the dominant factors controlling water removal.

At longer processing times, specifically at 8 h, significant differences ($p \leq 0.05$) were observed: blueberries treated with the 70% sucrose solution exhibited the highest moisture loss, as reflected in Table 2. This is attributed to the increased osmotic pressure gradient, which acts as the driving force for water removal, consistent with observations in berries reported by Vega Gálvez *et al*. (2012) and Ciurzyńska *et al*. (2016). However, the slope of the corresponding curve decreases more gradually compared to lower concentrations, indicating a progressive reduction in the effective mass transfer rate due to increasing diffusional resistance.

The observed differences in water-loss behavior among osmotic treatments, despite the absence of significant changes in solid gain, can be explained in terms of mass transfer mechanisms governing the process. Osmotic dehydration involves simultaneous countercurrent fluxes of water and solutes; however, these fluxes are not necessarily equivalent. In plant tissues, the outward flux of water is typically higher than the inward diffusion of solutes due to differences in molecular size, diffusivity, and membrane selectivity. Water molecules, being smaller and less hindered, diffuse more rapidly through cell membranes and intercellular spaces, whereas sucrose diffusion is more restricted, especially at high concentrations.

Additionally, increasing the concentration of the osmotic solution enhances the chemical potential gradient, promoting water removal, but also increases solution viscosity, which reduces the effective diffusivity of solutes and contributes to the formation of an external resistance layer around the fruit surface. As a result, the process becomes dominated by water loss, while solid gain remains comparatively limited. This selective mass transfer behavior explains why noticeable differences in water loss are observed among treatments (Figure 1 and Table 2), even when solid gain does not present statistically significant variations.

Nevertheless, blueberries treated with the 70% solution did not reach osmotic equilibrium within the same timeframe as those treated with 50% and 60% sucrose.

Table 2. Effect of sucrose concentration during osmotic dehydration on water loss (WL, g g^{-1}) of blueberries after 4 and 8 h, including ANOVA, Tukey's mean comparison ($p \leq 0.05$), and coefficient of variation (CV).

Pretreatment (sucrose concentration, % w/v)	Water loss at 4 h (WL, gg^{-1})	CV at 4 h (%)	Water loss at 8 h (WL, g g^{-1})	CV at 8v h (%)
50 %	10.0 ± 0.0 a	0.00	13.0 ± 1.4 b	10.88
60 %	12.0 ± 0.0 a	0.00	14.0 ± 0.0 b	0.00
70 %	16.0 ± 2.8 a	17.68	24.0 ± 0.0 a	0.00

This behavior-also documented for blueberries and strawberries-has been associated with increased medium viscosity and greater solid gain when highly concentrated sucrose solutions are used (Dermesonlouoglou *et al.*, 2007; Kucner *et al.*, 2012; Yang *et al.*, 2021). From a mass transfer perspective, this behavior can be interpreted as a pseudo-equilibrium condition, where mass fluxes decrease but do not entirely cease, preventing the establishment of a clear plateau in the dehydration curves.

In contrast, blueberries treated with 50% and 60% sucrose solutions exhibited progressive and stable moisture loss during the first 4 h, followed by a tendency toward stabilization consistent with a dynamic equilibrium condition. This behavior aligns with Prosapio and Norton (2018), who reported that intermediate sucrose concentrations promote more controlled dehydration kinetics, preserve fruit structural integrity, and enhance overall process efficiency (Yu *et al.*, 2017).

Table 2 compares the mean water loss (WL, g g^{-1}) of blueberries subjected to osmotic dehydration at different sucrose concentrations after 4 and 8 h of treatment, as determined by one-way ANOVA followed by Tukey's multiple comparison test.

Values represent mean ± standard deviation ($n = 2$). Different letters within the same column indicate statistically significant differences according to Tukey's multiple comparison test following one-way ANOVA ($p \leq 0.05$). The coefficient of variation (CV) was included as an additional descriptor of experimental variability and should be interpreted cautiously due to the limited number of biological replicates.

Mean values were compared using one-way ANOVA, and statistically significant differences among pretreatments were identified using Tukey's multiple comparison test ($p \leq 0.05$). Differences among sucrose concentrations became significant after 8 h of osmotic dehydration.

Figure 2 further illustrates these trends by integrating the effects of two additional freezing methods (liquid nitrogen ultrafreezing and conventional freezing) into the dehydration process. In all cases, the curves maintain a similar overall shape, characterized by a rapid initial decrease in moisture followed by a slower drying phase, consistent with the combined effects of prior dehydration and subsequent freeze-drying. The ultrafrozen treatments tend to exhibit higher water loss during intermediate stages, which may be associated with the formation of smaller and more uniformly distributed ice crystals, resulting in a more porous structure and reduced resistance to mass transfer during sublimation.

However, as the process progresses, the curves corresponding to all treatments converge, indicating that the differences introduced during osmotic dehydration and freezing become less significant over time. As shown in Table 3, these variations are not reflected in statistically significant differences in final moisture content after 23 h of freeze-drying. This convergence supports the interpretation that sublimation during freeze-drying becomes the dominant mass transfer mechanism, effectively homogenizing the system regardless of initial conditions. Therefore, Figure 2 confirms that, under the studied conditions, the impact of osmotic pretreatments and freezing methods is primarily reflected in the transient drying behavior rather than in the final state of the product.

3.2 *Effect of pretreatments on freeze-drying*

ANOVA results corresponding to the freeze-drying stage indicated that none of the evaluated pretreatments-OD at 50, 60, and 70% sucrose, liquid-nitrogen ultrafreezing, or conventional freezing-produced statistically significant differences ($p > 0.05$) in final moisture loss. These findings suggest that, under the conditions studied, freeze-drying operates as a dominant unit operation, capable of homogenizing the drying behavior regardless of the prior pretreatment. In addition to the kinetic analysis, the final moisture content was used as a comparative parameter among treatments. No statistically significant differences were observed, indicating that freeze-drying governs the overall dehydration performance regardless of the pretreatment applied (Ratti, 2001; Ciurzyńska and Lenart, 2011).

This phenomenon has been documented previously by Ratti (2001) and supported by later studies in berries, which report that the sublimation of ice during freeze-drying minimizes structural differences introduced during freezing, especially in small fruits with a high surface-area-to-volume ratio (Ciurzyńska and Lenart, 2011).

Table 3. Effect of osmotic dehydration and freezing pretreatments on final water loss (WL, g g^{-1}) of freeze-dried blueberries after 23 h, including ANOVA, Tukey's mean comparison ($p \leq 0.05$), and coefficient of variation (CV).

Pretreatment	Final water loss (WL, g g^{-1})	CV (%)
Liquid nitrogen ultrafreezing	0.79 ± 0.04 a	5.06
Osmotic dehydration (50% sucrose)	0.74 ± 0.07 a	9.46
Osmotic dehydration (60% sucrose)	0.71 ± 0.00 a	0.00
Osmotic dehydration (70% sucrose)	0.72 ± 0.00 a	0.00
Conventional freezing (control)	0.62 ± 0.04 a	6.45

Values represent mean ± standard deviation (n = 2). Means followed by the same letter are not significantly different according to Tukey's multiple comparison test following one-way ANOVA ($p \leq 0.05$). The coefficient of variation (CV) is shown as an indicator of experimental dispersion.

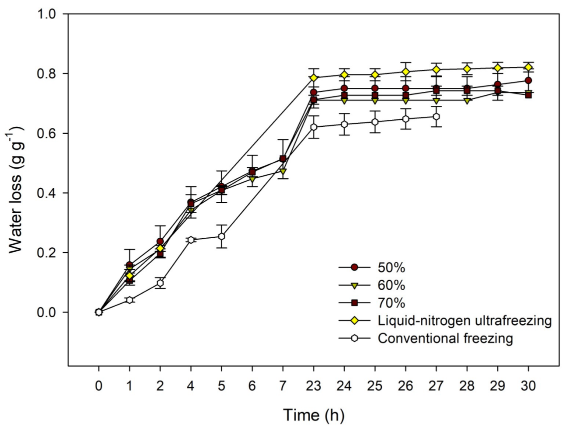

Figure 2. Dehydration kinetics (water loss) of blueberries subjected to osmotic dehydration and different freezing pretreatments. Values represent mean ± standard deviation (n = 2). Statistical analysis was performed using one-way ANOVA and Tukey's test at a significance level of $p \leq 0.05$.

Similarly, the lack of significant differences between liquid-nitrogen ultrafreezing and conventional freezing corroborates the findings of Munzenmayer *et al*. (2020), who observed that, for small fruits, the theoretical advantages of extreme freezing rates do not necessarily translate into improvements in drying kinetics or product quality (Semenov *et al*., 2021). At 23 h, none of the treatments differed significantly from one another, as shown in Table 3.

Mean final water loss values were compared by one-way ANOVA followed by Tukey's test, revealing no statistically significant differences among pretreatments after 23 h of freeze-drying.

These results confirm that, although ultrafreezing may increase moisture loss at intermediate stages, it does not provide a final advantage in freeze-drying performance. This aligns with findings reported for small fruits, where the impact of freezing rate becomes negligible once primary drying dominates mass transfer (Munzenmayer *et al*., 2020; Semenov *et al*., 2021).

Beyond drying kinetics, the evaluation of final product quality is critical to assess the practical and regulatory relevance of freeze-dried blueberries. In order to strengthen the practical implications of the freeze-drying process, additional quality attributes of the final product were evaluated, including residual moisture content and water activity. The moisture content of freeze-dried blueberries ranged from 5.64 to 8.03%, depending on the osmotic pretreatment and the freezing method applied. The lowest moisture content was observed in samples subjected to ultra-fast freezing using liquid nitrogen (5.64%), whereas the highest value corresponded to conventionally frozen samples (8.03%), highlighting the decisive role of freezing rate on water removal efficiency during freeze-drying. With respect to water activity, all treatments exhibited low a_w values (0.419–0.439), well below the 0.60 threshold widely recognized in international technical and regulatory documents, such as those of the Codex Alimentarius, for dehydrated food products, below which microbial growth is effectively inhibited. Furthermore, according to criteria established by the Food and Drug Administration (FDA) and the European Food Safety Authority (EFSA), foods with low a_w values are considered microbiologically stable and unlikely to support the growth of pathogenic microorganisms. In this context, the best-performing treatments evaluated in this study meet the general stability and safety requirements accepted for freeze-dried products, supporting their suitability for long-term storage and commercialization of freeze-dried blueberries.

3.3 Process engineering implications

From a technological perspective, the results indicate that highly intensive pretreatments-such as liquid-nitrogen ultrafreezing-do not offer significant advantages regarding final moisture loss after freeze-drying. This observation supports recommendations by Ratti (2013) and Prosapio and Norton (2017), who argue that pretreatment selection should be based not only on technical performance but also on economic feasibility.

Consequently, conventional freezing prior to freeze-drying emerges as an appropriate and cost-effective option for blueberry processing, provided that the cold chain is maintained immediately after harvest. This approach helps reduce energy costs without compromising drying efficiency or final product quality-an especially relevant factor when processing off-specification fruits intended for value-added applications (Nakagawa, 2025).

From a process engineering standpoint, the results indicate that freeze-drying performance is driven mainly by sublimation rate, making prior OD and freezing steps secondary when operated under the conditions studied.

It should be noted that the experiments were conducted using two independent biological replicates per treatment, each corresponding to a complete execution of the process. While this number is common in laboratory–pilot scale process development studies, future work should consider a larger number of biological replicates to strengthen statistical inference and result extrapolation.

Anthocyanin and total phenolic contents were not determined in this study; therefore, possible changes in these compounds resulting from osmotic pretreatments or freezing methods cannot be assessed. The scope of this work was focused on freeze-drying kinetics and process performance, rather than on the preservation of specific bioactive compounds.

Conclusions

In this study, a freeze-drying process for blueberry (*Vaccinium corymbosum* L.) was evaluated using osmotic dehydration and different freezing methods, with the purpose of analyzing their effects on moisture removal and drying efficiency. The evolution of water loss during osmotic dehydration was quantified and modeled, allowing a kinetic interpretation of the pretreatment stage.

During the initial stages of osmotic dehydration, no significant differences were observed among the sucrose concentrations evaluated, indicating that mass transfer was primarily governed by internal resistances within the plant tissue. At later stages, the 70% (w/v) sucrose solution promoted a higher water-loss rate; however, this effect was accompanied by a longer time required to approach osmotic equilibrium, which limited its overall contribution to process efficiency. The kinetics of water loss during osmotic dehydration were satisfactorily described using empirical models, with the Midilli–Küçük model providing the best representation of the experimental data.

The freeze-drying results indicated that the evaluated osmotic pretreatments and freezing methods did not produce statistically significant differences in the final moisture content, which was used as a comparative parameter among treatments. These findings demonstrate that, under the studied conditions, freeze-drying acts as the dominant unit operation governing the overall dehydration performance, regardless of the pretreatment applied.

Liquid nitrogen ultrafreezing did not provide relevant advantages compared with conventional freezing, suggesting that conventional freezing represents the most appropriate option from a technical and economic standpoint. Overall, the implementation of simple and low-cost osmotic pretreatments, combined with conventional freeze-drying, constitutes a viable strategy for the valorization of blueberries that do not meet commercial quality specifications.

Regarding osmotic dehydration, the obtained results demonstrate that operating conditions, particularly solution concentration and process time, significantly influence the interaction between water loss and solid gain. In this study, although stabilization of the water-loss curves was observed, it cannot be concluded that strict thermodynamic equilibrium was achieved under all the evaluated conditions, especially in the treatment with 70% sucrose. This behavior suggests the presence of a dynamic or pseudo-equilibrium state, in which mass fluxes decrease without being completely eliminated. From a mass balance perspective, it is recognized that the simultaneous evaluation of water loss and solid gain would allow a more accurate estimation of the system's equilibrium conditions. Therefore, future studies should incorporate more detailed quantitative analyses that consider both variables, as well as their dependence on operating parameters, in order to establish more robust criteria for the design and optimization of osmotic dehydration processes.

Overall, the findings of this study contribute to a better understanding of the relative importance of pretreatment stages in freeze-drying processes, highlighting that under certain conditions, process simplification may be a more efficient strategy than increasing pretreatment intensity. This has direct implications for the design of cost-effective and scalable dehydration systems for small fruits.

From a process engineering perspective, the obtained results demonstrate that freeze-drying acts as the dominant unit operation, capable of homogenizing drying behavior regardless of the applied pretreatments. In this regard, the study provides evidence that the use of more intensive osmotic conditions or high-rate freezing methods does not necessarily translate into significant improvements in overall process performance, particularly in small-sized matrices such as blueberries. These findings have direct implications for the selection and design of pretreatment stages prior to freeze-drying, suggesting that lower-cost, less complex strategies may be equally effective, thereby contributing to the development of more efficient processes for the valorization of agroindustrial products that fall outside commercial specifications.

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