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Use of freeze-drying and convection as drying methods of the xoconostle by-product and the effect on its antioxidant properties

Uso de la liofilización y la convección como métodos de secado del subproducto del xoconostle y el efecto sobre sus propiedades antioxidantes

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Abstract
In this study, the influence of the drying method (lyophilization and convection) on the antioxidant properties of the by-product of xoconostle (*Opuntia matudae*) was evaluated. The analysis of convection drying results indicated that the drying took place during the period of decreasing speed, besides the drying speed was influenced by the rise in temperature, reaching the target moisture content (0.06 g_{water} /$g_{dry\ sample}$) in shorter processing times as the temperature increased (60 °C-195 min; 70 °C-165 min; 80 °C-120 min). The effective diffusivity coefficients (D_{ef}) ranged between 4.788 E^{-10}-8.109 E^{-10} m^2/s for the evaluated temperatures. Drying by lyophilization and by convection at 60 °C were ideal for the preservation of antioxidant capacity, however, considering the cost-benefit ratio, convective drying at 60 °C is more favorable for its use on the xoconostle by-products since they showed to maintain phenolic compounds (72.56 ± 0.06 mg GAE/100 g) and antioxidant capacity determined by ABTS and DPPH (7.63 ± 0.96 TEAC/g_{sample}, 88.07 ± 0.38 % DPPH inhibition). Therefore, powdered by-products can be incorporated as functional additives.

Keywords: betalain, total phenols, antioxidant capacity, drying kinetics, flow properties.

Resumen
En el presente estudio se evaluó la influencia del método de secado (liofilización y convección) sobre las propiedades antioxidantes del subproducto de xoconostle cv. Cuaresmeño (*Opuntia matudae*). El análisis del secado por convección indicó que el secado tuvo lugar en el período de velocidad decreciente, además, la velocidad de secado se vio influenciada por el aumento de la temperatura alcanzando el contenido de humedad objetivo (0.06 g_{agua}/ $g_{muestra\ seca}$) en menores tiempos de procesamiento con el aumento en la temperatura (60 °C-195 min; 70 °C-165 min; 80 °C-120 min). Los coeficientes de difusividad efectiva (D_{ef}) oscilaron entre 4.788 E^{-10}-8.109 E^{-10} m^2/s para las temperaturas evaluadas. El secado por liofilización y el tratamiento a 60 °C resultaron ideales para la conservación de la capacidad antioxidante, sin embargo, teniendo en cuenta el costo-beneficio resulta más favorable el secado convectivo a 60 °C para el aprovechamiento de los subproductos de xoconostle, dado que presentaron una importante retención de compuestos fenólicos (72.56 ± 0.06 mg GAE/100 g) y capacidad antioxidante ABTS y DPPH (7.63 ± 0.96 TEAC/$g_{muestra}$, 88.07 ± 0.38 % inhibición DPPH). Por lo anterior, los subproductos en polvo pueden ser incorporados como aditivos funcionales.

Palabras clave: betalaínas, fenoles totales, capacidad antioxidante, cinética de secado, propiedades de flujo.

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1 Introduction

The xoconostle cv. Cuaresmeño (*Opuntia matudae*) is one of the ten species of the genus *Opuntia* found in Mexico, and it is the most important and commercialized xoconostle species in Mexico (Guzmán-Maldonado *et al*., 2010; Morales *et al*., 2015). It belongs to one of the more than 1000 cactaceans of the genus *Opuntia* distributed around the world (Bensadón *et al*., 2010; Morales *et al*., 2012).

The xoconostle cv. Cuaresmeño (*Opuntia matudae*) is used as a raw material for the preparation of food products, such as jams and sauces; however, it is mainly used for juice extraction (Guzmán-Maldonado *et al*., 2010; Morales *et al*., 2015). In addition, it is also used in traditional Mexican medicine for the treatment of diseases, such as diabetes (type 2) and hyperglycemia. Hypolipidemic effects have also been reported (Morales *et al*., 2012; Osorio-Esquivel *et al*., 2011) along with preventing the development of chronic and respiratory diseases (Fernández-Luqueño *et al*., 2021). These beneficial health effects have been attributed to the bioactive compounds contained in the xoconostle fruit; among these, phenolic compounds and betalains stand out, and these compounds are also responsible for the red coloring of the fruits (Feugang *et al*., 2006; Guzmán-Maldonado *et al*., 2010; Morales *et al*., 2015).

Due to the functional properties of the xoconostle fruit, many investigations have focused on the extraction of its bioactive compounds and their preservation for application in food systems (Fernández-Luqueño *et al*., 2021). An example of this is the study carried out by Pérez-Alonso *et al*. (2015), where they accomplished the microencapsulation of the bioactive compounds of the xoconostle fruit (*Opuntia Oligacantha*) with spray drying to stabilize phenolic compounds. Similarly, Aksoylu *et al*. (2021) performed microencapsulation of the bioactive compounds in xoconostle (*Opuntia spp*) to incorporate them into food products, such as yogurt, edible films and chewy candies. Espino-Manzano *et al*. (2020) studied the application of nanoemulsions (w/o) of xoconostle extract (O. *Oligacantha*) /orange oil in gelatin films, which resulted in an increase of phenolic compounds and a reduction in microbiological contamination of the product.

However, these investigations are based only on the study and use of xoconostle juice and do not consider the generated by-products (epicarp and endocarp) that represent up to 45 % of the fruit (Bensadón *et al*., 2010; Morales *et al*., 2015); These residues generate both economic and environmental problems (Aymen and Benvenuti, 2020). In Mexico, approximately 10,000 tons of xoconostle cv. Cuaresmeño (*Opuntia matudae*) are produced yearly (Fernández-Luqueño *et al*., 2021), which implies the generation of at least 4500 tons of by-product (epicarp and endocarp) of xoconostle cv. Cuaresmeño. These by-products retain a significant amount of compounds of interest that can be used to produce food additives with functional potential (Guzmán-Maldonado *et al*., 2010).

To exploit the above-mentioned by-products, it is necessary to reduce their water content to preserve them. Drying technologies provide alternatives for the preservation of food products, increasing their shelf-life and reducing the potential development of pathogenic microorganisms and undesirable chemical spoilage reactions (Kabuo *et al*., 2014; Salcedo-Mendoza *et al*., 2016; Figueroa-Garcia *et al*., 2021).

Convective drying is based on reducing the water activity of a product by eliminating the humidity under controlled conditions; it is the most effective method for the preservation of biological products if the thermic treatment is not aggressive for the product (Zárate-Castillo *et al*., 2018).

Another option for drying is the use of the freeze-drying process, which is based on water sublimation and reduces the depletion of volatile or heat-sensitive components. This drying process has the advantage of minimizing nutritional or functional losses and facilitates rehydration of the obtained powders (Ratti, 2013; Shukla, 2011).

In this study, the effect of the drying method (convection and freeze-drying) on the flow properties and antioxidant capacity of the by-products (epicarp and endocarp) recovered from the xoconostle cv. Cuaresmeño (*Opuntia matudae*) juice extraction process was evaluated to obtain an additive for food products with antioxidant properties. An additional goal was to contribute to the use of an agro-industrial waste, thus mitigating the environmental impact of industrial processes.

2 Materials and methods

2.1 Materials

Xoconostle cv Cuaresmeño (*Opuntia matudae*) fruits were acquired in the municipality of San Martin de las Pirámides, Mexico State (19°41’49.5”N latitude, 98° 49’58.9”W longitude). The fruits were acquired in a state of commercial maturity, discarding those that presented any visible mechanical damage. The fruits were washed and disinfected with a sodium hypochlorite solution (0.05 %) for 10 minutes, then dried and weighed (200 kg) and stored in a refrigerator (6-8 °C) until later use. The 2,2-diphenyl-1-picrylhydrazyl (DPPH) and 2,2’-Azino-bis (3-ethylbenzothiazoline-6-sulfonic acid) diammonium salt (ABTS) used during the antioxidant activity assay were purchased from Sigma-Aldrich (St Louis MO, USA).

2.2 Methods

2.2.1 Xoconostle by-product recovery

The by-product from the pulping process of Xoconostle (BPX) were recovered as follows. The clean and chopped fruits were subjected to a water bath at 70 °C for 20 minutes at a 2:1 ratio (fruit: water). After the elapsed time, the fruits were pulped by using a semi-industrial pulper (Polinox D-7). The juice and by-product made up of the mucilage of the epicarp and of the endocarp were stored at -20 °C until use.

2.2.2 Drying of the xoconostle by-product

2.2.2.1 Freeze-drying

The BPX was dried using the freeze-drying method as follows. The previously frozen samples with liquid nitrogen were dried for 96 h in a freeze dryer (LABCONCO Freezone Plus 4.5 L-Mod 7386020, USA) using a vacuum pressure of 0.040 mbar and a temperature of -80 °C. The resulting material was named FDBX.

2.2.2.2 Convective drying

The drying process was also conducted in a convective dryer (Intertecnica S.A. de C.V, Mexico). Three different temperatures were evaluated (60, 70 and 80 °C) with a constant drying air speed of 1.25 m/s. The drying time for each condition was established by performing the drying kinetics when the resulting values of moisture were under 10 %. The samples obtained from convective drying were named BPX60 (60 °C), BPX70 (70 °C) and BPX80 (80 °C).

2.2.3 Drying speed

The drying kinetics for each processing temperature (60, 70 and 80 °C) were adjusted to a polynomial (degree = 3), which were stablished from the coefficient R^2. The obtained polynomials were derived with respect to time and plotted against the moisture content on a dry basis. The drying rate was obtained through the slope (Jiménez-Guzmán, 2011).

2.2.4 Determination of the effective diffusion coefficient

The drying kinetics for each drying temperature were analyzed to evaluate the effective diffusivity from the Fick model shown by Eq. 1 (Mota *et al*., 2010):

$$X^* = \frac{X^* - We}{W_0 - We} = \frac{8}{\pi^2}\sum_{n=0}^{\infty}\exp\left[\frac{-(2n+1)^2 n^2 D_{ef}^t}{4H^2}\right] \quad (1)$$

where X^* is the unfulfilled moisture (g/g), We is the equilibrium moisture (g/g), W_0 is the initial moisture (g/g), H is the thickness of the by-product (m), D_{ef} is the effective diffusivity (m^2/s) and t is the time (s).

The analysis was made considering an infinite flat plate, disregarding the by-product shrinkage and the absence of resistance to mass transport. The effective diffusivity was constant. The D_{ef} was determined from the slope method for which the unfulfilled moisture was plotted against time, resulting in a straight line with a slope ($\ln X^*$). Equation 1 was simplified into a straight-line equation (Eq. 2) and was solved to obtain the D_{ef}.

$$\ln X^* = \left(\frac{-\pi^2 D_{ef}}{4H^2}t\right) \quad (2)$$

2.2.5 Particle size

The size reduction of the dried by-products was using a food processor (Nutribullet 600w). The size distribution of the particles (SDP) was determined by the sieving method, this method is the most used in the industry due to its simplicity, low cost, and ease of analysis. It was carried out according the ASABE S319.4 (ASABE, 2008) and AACC 55-60.01 (AACC, 2011) standards with slight modifications; 50 g of the samples were passed through the following sieves: No. 20 (841 μm), 30 (595 μm), 60 (250 μm), 80 (177 μm) and bottom (<177) using a sieve separator (W.S. Tyler Rx-812 RX-812) with stirring for 15 minutes. The mass of the four separated fractions was weighed on an analytical balance (Velab model VE-204). From these data, the percentage of retained mass on each sieve in relation to the total mass was calculated to determine the particle size distribution.

2.2.6 Physicochemical characterization of the xoconostle by-product powder

2.2.6.1 Proximate analysis and water activity

Analyses of moisture (Method 925.09) and ash (Method 930.05) were carried out by the official methods described by the Association of Official Analytical Chemists (Horwitz and Latimer, 2005). The water activity (aw) was evaluated using a Labmaster-aw equipment (Novasina Labswift) at 25 °C for 1g of each sample for each drying condition.

2.2.6.2 Total carbohydrates

This test was conducted with the phenol-sulfuric method according to López-Legarda *et al*. (2017) as follows. A quantity of 0.0020 g of each sample was placed in a beaker, and distilled water was added until a volume of 100 mL was reached. Then, 2 mL of this solution was placed in a test tube, 1 mL of phenolic solution (5%) was added, and 5 mL of concentrated sulfuric acid was added. The test tubes were allowed to stand for 10 min. Finally, the measurement was conducted in a spectrophotometer (Thermo Scientific^{TM} GENESYS^{TM} UV-Vis) at 490 nm. A standard curve for glucose (10-100 mg/L) was previously measured to make the quantification.

2.2.6.3 Colorimetry

The color was determined with a colorimeter (Konica Minolta CR-400) that provided values for (L), a * and b *. With the obtained data, the chroma (C) and tone (H) of the samples were calculated according to Eqs. 3 and 4 (Darniadi *et al*., 2007):

$$C = \sqrt{a^2 + b^2} \tag{3}$$

$$H = \arctan\left(\frac{b}{a}\right) \tag{4}$$

2.2.6.4 Solubility index

The water solubility index (WSI) was determined according to the method described by Martínez-Jiménez *et al*. (2015) with slight modifications. To begin, 0.3 g of each sample was weighed in a centrifuge tube, and then, 30 mL of distilled water was added. The tubes were then stirred on a vortex (Cole-Parmer^{TM} Vortex Mixer) for 30 seconds and placed in a water bath at 30 °C for 30 minutes. Subsequently, the suspensions were centrifuged (Centrificient IV CRM Globe) for 10 min at 3500 g. The supernatants were transferred to porcelain capsules with a constant weight and were dried at 105 °C for 24 h in a drying oven. The WSI was calculated according to Eq 5:

$$WSI = \frac{\text{weight of dissolved solids supernatant (g) x (V)}}{\text{Sample weight (g)}} \times 100 \tag{5}$$

2.2.7 Antioxidant evaluation of powders

2.2.7.1 DPPH antioxidant activity

Methanolic extracts were obtained from each sample according to the methodology proposed by González-Jiménez *et al*. (2018) with slight modifications. 1 g of each sample was mixed with 10 mL of aqueous methanol (50 %) for 1 h. The mixture was then centrifuged at 2500 g for 10 minutes, and then, the supernatant was separated and stored at -20 °C in amber flasks. The antioxidant activity was evaluated by adding 1.9 mL of DPPH in methanol (0.025 g/L) to react with 0.05 mL of the extracts of each sample. The tubes were incubated at 25 °C and protected from light for 30 min. Finally, the absorbance was measured at 515 nm in a spectrophotometer (Thermo Scientific^{TM} GENESYS^{TM} UV-Vis), and the percentage of inhibition of DPPH was calculated according to Eq. 6:

$$\%DPPH\ Inhibition = \frac{(|DPPH|) - (|DPPH| + Sample)}{|DPPH|} \times 100 \tag{6}$$

2.2.7.2 ABTS antioxidant activity

The extracts were obtained from each sample according to the methodology proposed by González-Jiménez *et al*. (2018). The determination of antioxidant activity using ABTS was carried out according to the methodology developed by Re *et al*. (1999) with slight modifications. The procedure was as follows: ABTS 7 mM was prepared, and the radical was activated by mixing it with a 2.45 mM potassium persulphate solution in a 2:1 ratio by incubating it for 16 hours at room temperature and in the absence of light. Trolox^{TM} (dissolved in 70 % ethanol) was used as a reference, it was used to prepare a 1500 μM stock solution, dilutions were made to obtain concentrations of 300, 600, 9000, 1200 and 1500 μM. finally, 100 μL of each dilution were mixed with 900 μL of ABTS (absorbance was adjusted to 0.700 ± 0.02 at 734 nm). The data obtained were plotted (% inhibition vs. Trolox^{TM} concentration) and the result obtained from each sample was expressed as the Trolox^{TM} equivalent antioxidant capacity per g of sample (TEAC/g$_{sample}$).

2.2.7.3 Total Phenolic Content

This assay was conducted using the Folin-Ciocalteau reagent as follows. To start, 0.1 mL of each sample extract was mixed with 1 mL of Folin-Ciocalteau reagent (diluted in water 1:10), and then, 0.8 mL of sodium carbonate (7.5 %) and 0.1 mL of distilled water were added. The mixture was incubated for 30 minutes in the dark, finally the absorbance was measured at 765 nm. The results are expressed in mg of gallic acid equivalents (GAE/100 g$_{sample}$) (González-Jiménez *et al*., 2018).

2.2.7.4 Quantification of Betalains

To perform the assay, 100 mg of each sample were mixed with 10 mL of aqueous methanol (50 % v/v). The mixture was stirred for 60 minutes and then centrifuged at 5000 g for 15 min. The supernatant was recovered, and the solid residue was treated again with aqueous methanol until the absence of color was noted. The resulting extracts for each sample were then combined and their absorbances at 476, 538 and 600 nm were recorded (Hernández-Fuentes *et al*., 2015a).

The betalain content was calculated according to the equation 7:

$$Betalain = \left(\frac{a}{1129}\right) \times DF \times 100 \tag{7}$$

where a = 1.095 (A538 - A600) and DF is the dilution factor. The betalain content is reported in mg/100 g$_{sample}$.

2.2.8 Techno-functional properties analysis

2.2.8.1 Apparent and packed density

The apparent density was evaluated by placing 2 g of each sample (in powder form) in a 10 mL graduated cylinder. The registered volume occupied by the sample was used to calculate the apparent density, which is reported in g/mL (Pereyra-Castro *et al*., 2018). The packed density was assessed by tapping the cylinder that contained the powdered sample on a flat surface to achieve a constant volume.

2.2.8.2 Carr's Index (CI) and Hausner's Ratio (HR)

The apparent density and the packed density values were used to calculate the Carr's Index (CI) and Hausner's ratio (HR) according to Eqs. 8 and 9 (Martínez-Jiménez *et al*., 2015), respectively:

$$CI = \frac{\rho packed - \rho apparent}{\rho apparent} \times 100 \tag{8}$$

$$HR = \frac{\rho packed}{\rho apparent} \tag{9}$$

2.2.9 Hygroscopicity

The hygroscopicity was determined by placing 2 g of sample in a container with saturated NaCl solution (relative humidity of 75.9 %) at 25 °C. Seven days later, the samples were weighed. The hygroscopicity is expressed as the weight of water adsorbed per 100 g of dry weight of the sample (g/100 g) (Souza *et al*., 2018). Eq. 10 was used to calculate the hygroscopicity:

$$\%Higroscopicity = \frac{D_S - S_{ME}}{D_S} \times 100 \tag{10}$$

where D_S is the dry sample and S_{ME} is the sample with the moisture in equilibrium.

2.2.10 Statistical analysis

The results were processed with Minitab® software (version 16.0). All determinations were made in triplicate. The analysis of variance and a comparison of the means were conducted using the Tukey test with a significance level of 5% and a confidence level of 95%.

3 Results and discussion

3.1 Process yield

Derived from the pulping process of the xoconostle fruit, a yield of 41.34 ± 3.47 % of BPX (epicarp and endocarp mucilages) was obtained, thus showing how important it is to make use of it due to the amount of generated by-product and the functional compounds it has been reported to contain (Guzmán-Maldonado *et al*., 2010). Regarding the BPX drying process, yields of 18.23 ± 1.11, 18.07 ± 0.56 and 18.02 ± 0.01 % were obtained for BPX60, BPX70 and BPX80 respectively, while freeze drying had a yield of 16.11 ± 1.04 %. The lower yield found in freeze drying compared to convection drying was also reported by Gopinathan *et al*. (2020), in the evaluation of different drying methods on the antioxidant capacity of cempedak powder (*Artocarpus integer*) and by Shuen *et al*. (2021), on the effect of different drying methods on the antioxidant capacity of Kuini (*Mangifera odorata*).

Table 1. Equilibrium moisture content, slope value and D_{ef} for each drying temperature.

Temperature (°C)	We ($g_{water}/g_{dry\ sample}$)	Slope value	D_{ef} (m^2/s)
60	0.04 ± 0.000	7.81×10^{-4}	$4.788\times10^{-10} \pm 7.156\times10^{-12b}$
70	0.03 ± 0.001	9.42×10^{-4}	$4.961\times10^{-10} \pm 5.250\times10^{-12b}$
80	0.02 ± 0.000	1.06×10^{-3}	$8.109\times10^{-10} \pm 1.731\times10^{-11a}$

We: Equilibrium moisture content. Identical letters in the same column indicate that there were no significant differences (significance level p≤0.05).

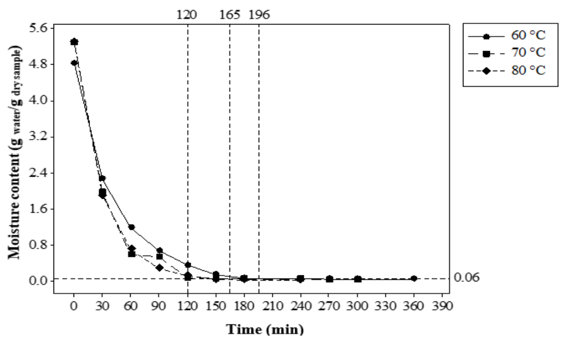

Figure 1. Drying kinetic for obtaining equilibrium moisture content and time to reach target moisture content.

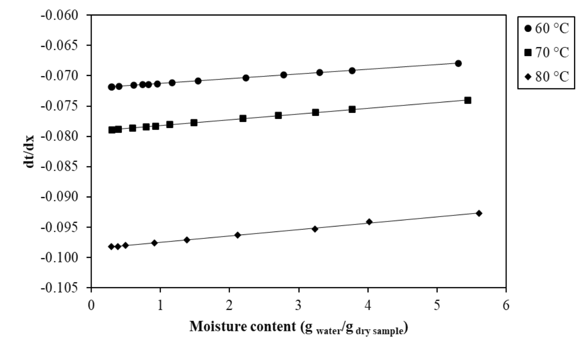

Figure 2. Drying rate curves at 60, 70 and 80 °C.

3.2 *Freeze-drying*

The total drying time represents a very important variable for the freeze-drying processes due its direct influence on production costs (Ramírez *et al.*, 2019). This can be observed during the drying of FDBX, whose drying time was 96 hours, which is approximately 29 times longer than the time required for the dehydration of BPX60. This behavior is attributed to the variables that influence during the drying process, such as the relative humidity of the air, the process temperature and air speed, so that the process times for freeze drying are consequently longer than those required for convection drying (Izli and Polat, 2019).

3.3 *Convective drying*

Figure 1 shows the drying curves of the xoconostle by-products at 60, 70 and 80 °C. For the three drying temperatures, the loss of moisture developed in a non-linear way. The decreasing speed period dominated the drying process as shown in the drying curves; therefore, the mass transfer from the system to the environment was controlled by diffusion (Elhussein and Şahin, 2018; Prachayawarakorn *et al.*, 2008). The target final moisture content was established at 0.06 on a dry basis for the above-mentioned drying temperatures; this moisture was reached in a drying time of 196 min for 60 °C, 165 min for 70 °C and 120 min for 80 °C according to the drying kinetics (Figure 1). On the other hand, the equilibrium moisture content (Table 1) was 0.04, 0.03 and 0.02 on dry basis for temperatures of 60, 70 and 80°C, respectively, presenting an inversely proportional relationship with the drying temperature, this behavior corresponds with that reported by Jiménez-Guzmán, (2011) and Roberts *et al.* (2008) for the simultaneous drying and husking of parchment coffee and grape seed drying, respectively where they also indicate that this parameter is only in function of temperature, that is, it is unique for each drying temperature.

Figure 2 shows the drying rate curves for each processing temperature. Based on the slopes of these lines (Table 1), it can be observed that the change in the drying rate, with respect to the moisture on a dry basis is greater than 80 °C followed by that of 70 °C and finally 60 °C, indicating that a higher slope value corresponds to a higher drying rate. This fact could be influenced by an acceleration in the movement of water molecules when they are exposed to high temperatures or by the formation of pores in the system that cause a rapid migration of moisture from the system to the environment (Mayor *et al*., 2011; Prachayawarakorn *et al*., 2008; Sagar and Suresh, 2010).

3.3.1 Effective diffusion coefficient

The effective diffusion coefficient (D_{ef}) includes the effects of all the phenomena that can intervene in the migration or loss of humidity through convection and diffusion processes that flow from the interior of the product to its surface, its value is always calculated through the second law of Fick which is generally used for the analysis of the mechanisms of moisture transfer within the sample during the period of decreasing speed (Azaka *et al*., 2019; Virgen-Navarro *et al*., 2016). Effective diffusivity can be understood as the ease with which water is removed from the material (Giraldo-Zuniga *et al*., 2010). The average values of the effective diffusion coefficient for each drying temperature are shown in Table 1. The D_{ef} for drying at 80 °C showed significant differences with the treatments at 60 and 70 °C (p≤0.05). A proportional correlation between the temperature and the D_{ef} was found, where the higher the drying temperature was, the greater the D_{ef}. This result corresponds with those reported in numerous investigations regarding drying processes of food systems, where it is pointed out that an increase in the temperature causes an increase in the activity of water molecules and in the contraction of the system. This caused a decrease in the diffusion area and consequently in the disposition of the water to migrate from the system (Azaka *et al*., 2019; Doymaz, 2005; Kumar *et al*., 2015). Thus, the effective diffusion coefficients for the different BPX drying temperatures (60, 70 and 80 °C) are within the proposed range for food systems (Doymaz, 2005; Vega *et al*., 2007), which indicates that the Fick model satisfactorily fits the experimental data for the drying of BPX.

3.4 Particle size

The evaluation of the particle size and distribution of food powders is of paramount importance to determine their usefulness and application in food matrices, processing, and packaging (Patwa *et al*., 2014; Chen, 2009). According to the particle size distribution analysis (Table 2) performed on the four samples (BPX60, BPX70, BPX80 and FDBX), four fractions of xoconostle powder were obtained. FDBX presented the highest coarse dust fraction (10 %) with a particle size of 595 μm on the other hand, BPX70 had the finest dust fraction (81%) with a particle size < 177 μm, followed by BPX60 (80 %), BPX80 (78 %) and FDBX (70.33 %). The powders obtained from convective drying (BPX60, BPX70 and BPX80) did not show significant differences in particle size (p ≤ 0.05), however, the highest mass retention occurs in FDBX (30 %), which indicates that the powders have particle sizes ≥ 177 μm. This could be associated with its water absorption capacity that gives rise to the formation of agglomerates that prevent the granules from passing through the mesh. This coincides with the hygroscopic capacity found in FDBX, which was higher than that found in BPX60, BPX70 and BPX80. Moreover, the four samples can be classified as fine flours, since 90 % of the particles had a particle size of less than 595 μm (Prachayawarakorn *et al*., 2008). Therefore, both drying methods allow the obtention of powders with an appropriate particle size. Thus, the powders from the different treatments could be considered as alternative flours with food applications due to their granulometry (Dussán-Sarria *et al*., 2019).

3.5 Analysis of physicochemical properties

3.5.1 Proximate analysis and water activity

In this study, the convection dried BPX (BPX60, BPX70, BPX80) was brought to a target moisture content, therefore, no significant differences in the moisture content were found, values were in a range of 0.07 to 0.08 $g_{water}/g_{dry\ sample}$ (Table 3). However, the moisture content found for FDBX (0.09 $g_{water}/g_{dry\ sample}$) presented significant differences with respect to convection dried samples and was higher than that reported by Arias-Rico *et al*., (2020), in xoconostle flour (*Opuntia spp*.) obtained by lyophilization (0.07 $g_{water}/g_{dry\ sample}$). Furthermore, the aw values for BPX60, BPX70, BPX80 ranged from 0.45 a 0.53 and significant differences were found (p ≤ 0.05) they also presented an inversely proportional relationship with the drying temperature, that is, the higher drying temperature, the lower aw, this behavior generally occurs during fruit drying (Bhandari and Adhikari, 2009) it was also reported by Michalska *et al*. (2019) in the assessment of heat-induced changes in by-products of Prunus domestica L. and by Macedo *et al*. (2020) in the evaluation of drying temperature on the physico-chemical properties of bananas.

Table 2. Granulometric analysis of powdered xoconostle by-products for different drying conditions.

Sample	Sieve number	% Retained	% Passing
BPX60	20	0	100
	30	4 ± 0.2022^{c}	96.03
	60	14 ± 0.2048^{c}	82.05
	80	2 ± 0.1902^{b}	79.89
BPX70	20	0	100
	30	4 ± 0.1365^{c}	96
	60	13 ± 0.1036^{d}	84
	80	2 ± 0.1231^{b}	81
BPX80	20	0	100
	30	5 ± 0.2600^{b}	95.45
	60	15 ± 0.1729^{b}	80.76
	80	2 ± 0.1711^{b}	78.37
FDBX	20	0	100
	30	10 ± 0.4226^{a}	90.4
	60	17 ± 0.1471^{a}	73.83
	80	3 ± 0.4908^{a}	70.33

Identical letters in the same column indicate that there were no significant differences (significance level p≤0.05). BPX60: sample dried at 60 °C; BPX70: sample dried at 70 °C; BPX80: sample dried at 80 °C; and FDBX: freeze dried sample.

On the other hand, FDBX presented the highest aw value (0.54 ± 0.00) compared to the other drying treatments, as it is observed in Table 3, aw depended significantly on the moisture content of the samples, displaying a proportional relationship between these values, that is, the higher moisture content, the higher aw, this behavior was also reported by Michalska *et al*. (2017) in blackcurrant pomace drying, however, both drying methods resulted in aw values and moisture contents below the recommended limits to avoid the development of pathogenic microorganisms, and thus contributing to a low risk of physicochemical decay and an increase of shelf life of the obtained powders (Brito *et al*., 2020; Costa *et al*., 2017).

The ash contents of the samples (BPX60, BPX70, BPX80 and FDBX) are shown in Table 3 and range from 10.24 ± 0.18 % to 10.77 ± 0.26 %. The mineral content of the powders (BPX60, BPX70, BPX80 and FDBX) in this study is lower than that reported by Guzmán-Maldonado *et al*. (2010) in their physicochemical analysis of xoconostle cuaresmeño but higher than that reported by Hernández-Fuentes *et al*. (2015a) in the analysis of the physicochemical variability between different xoconostle species. The differences in mineral content can be attributed to different factors, such as the maturity, variety, type of soil and cultivation zone of the fruit (Miranda *et al*., 2009). The ash content found in the powders (BPX60, BPX70, BPX80 and FDBX) represents an important source of minerals (Guzmán-Maldonado *et al*., 2010; Hernández-Fuentes *et al*., 2015a).

3.5.2 Total carbohydrates

The total carbohydrate content is shown in Table 3, where an inversely proportional correlation was observed between the drying temperature and the total carbohydrate content for the powders exposed to convective drying (BPX60, BPX70, and BPX80). That is, the lower the drying temperature was, the higher the total carbohydrate content. On the other hand, the total carbohydrate content of FDBX was higher than that found in BPX60, BPX70 and BPX80. In this sense, it can be established that the total carbohydrate content was significantly affected by the drying process, where its concentration was reduced by more than 50 % with convective drying. This is related to the application of high temperatures for the dehydration of BPX (Guiné *et al*., 2011). Hernández-Fuentes *et al*. (2015b) reported total carbohydrate contents of xoconostle lower than those found in this research, establishing that the total carbohydrate content may vary according to the species, maturity, and ecosystem in which the plant develops.

Table 3. Physicochemical analysis of powdered xoconostle by-products.

Sample	Aw	Hbs ($g_{water}/g_{dry\ sample}$)	Ash %	WSI %	Total carbohydrates (mg/L)
BPX60	0.53 ± 0.00^{b}	0.08 ± 0.01^{b}	10.77 ± 0.26^{a}	21.57 ± 0.54^{b}	11.64 ± 2.39^{b}
BPX70	0.46 ± 0.00^{c}	0.07 ± 0.01^{b}	10.56 ± 0.27^{a}	21.77 ± 1.05^{b}	10.51 ± 0.28^{b}
BPX80	0.45 ± 0.00^{d}	0.07 ± 0.00^{b}	10.24 ± 0.18^{a}	21.37 ± 0.40^{b}	9.78 ± 1.46^{b}
FDBX	0.54 ± 0.00^{a}	0.09 ± 0.01^{a}	10.59 ± 0.24^{a}	25.51 ± 0.10^{a}	25.79 ± 1.46^{a}

Identical letters in the same column indicate that there were no significant differences (significance level $p \leq 0.05$). BPX60: sample dried at 60 °C; BPX70: sample dried at 70 °C; BPX80: sample dried at 80 °C; and FDBX: freeze dried sample.

Table 4. Colorimetric analysis of powdered xoconostle by-products.

Sample	L	a*	b*	C	°H
BPX60	71.46± 0.26^{a}	7.76 ± 0.05^{ab}	17.51 ± 0.06^{ab}	19.15 ± 0.07^{ab}	66.09 ± 0.69^{b}
BPX70	70.28 ± 0.12^{b}	7.54 ± 0.03^{b}	16.93 ± 0.12^{b}	18.54 ± 0.12^{b}	66.01 ± 0.11^{b}
BPX80	68.38 ± 0.28^{c}	7.79 ± 0.13^{ab}	18.74 ± 0.31^{a}	20.29 ± 0.33^{a}	67.42 ± 0.14^{a}
FDBX	72.96 ± 0.59^{d}	7.85 ± 0.14^{a}	15.29 ± 0.92^{c}	17.19 ± 0.88^{c}	62.79 ± 0.99^{c}

Identical letters in the same column indicate that there were no significant differences (significance level $p \leq 0.05$). BPX60: sample dried at 60 °C; BPX70: sample dried at 70 °C; BPX80: sample dried at 80 °C; and FDBX: freeze dried sample.

3.5.3 Water solubility index

No statistically significant difference was found between the water solubility index (WSI) of the powders obtained by convective drying (BPX60, BPX70 and BPX80) at the different processing temperatures. The results ranged from 21.37 ± 0.40 % to 21.77 ± 1.05 % (Table 3); however, the percentage of solubility found for the FDBX powder was higher (25.51 ± 0.54%). The higher solubility for FDBX may be related to the freezing process and application of a vacuum, which generates amorphous products. Cano-Chauca *et al*. (2005) and Ribeiro *et al*. (2016) reported that an amorphous structure has a higher solubility and dissolution speed than crystalline structures, which are formed in dehydrated materials during convection drying (Joardder *et al*., 2017).

3.5.4 Colorimetry

The color is one of the parameters that affects consumer perception of a product (Sant'Anna *et al*., 2013); therefore, it is important to determine the effect of the temperature and drying process on the color of the resulting powders. The color parameters of BPX dried under the different experimental conditions are given in Table 4. As shown in Table 4, there was an inversely proportional behavior between the convective drying temperature and the L* coordinate, indicating a darkening in the color of the samples with the increase in the drying temperature. Furthermore, FDBX had the highest L* coordinate value, which indicates greater whiteness compared to BPX60, BPX70 and BPX80. As for the values obtained in parameter a* for each of the samples (BPX60, BPX70, BPX80 and FDBX), the values obtained were positive thus indicating a tendency towards red, it can also be seen that the drying temperature did not influence the value of this coordinate since, according to the performed analysis of variance, no significant differences were found. The b* coordinate showed its lowest value in FDBX and its highest value in BPX80, presenting positive values for each of the analyzed samples, this indicates a tendency towards yellow. The chroma color parameter indicates the degree of color saturation, where BPX80 had the highest color saturation. The tonality (h*) indicated that the convection dried powders (BPX60, BPX70 and BPX80) exhibited a darker yellow color (brown) than FDBX. A factor for these results is the formation of pigments through the well-known Maillard reaction, which is favored at high temperatures. The parameters that affect the Maillard reaction are mainly the content of sugars and proteins, temperature, and heat treatment length (Corrêa *et al*., 2011; Medina-Torres *et al*., 2021).

Table 5. Antioxidant capacity measured by DPPH and ABTS methods.

Sample	DPPH (% inhibition)	ABTS (TEAC/g_{sample})
BPX60	88.07 ± 0.38^b	7.63 ± 0.96^b
BPX70	47.51 ± 1.65^c	5.01 ± 0.23^c
BPX80	47.26 ± 0.56^c	4.79 ± 0.11^c
FDBX	95.6875 ± 0.15^a	11.62 ± 0.60^a

Identical letters in the same column indicate that there were no significant differences (significance level p≤0.05). BPX60: sample dried at 60 °C; BPX70: sample dried at 70 °C; BPX80: sample dried at 80 °C; and FDBX: freeze dried sample.

3.6 Antioxidant properties analysis

3.6.1 Antioxidant capacity

The percentages of DPPH radical inhibition of the xoconostle by-product powders (BPX60, BPX70, BPX80 and FDBX) are shown in Table 5. The antioxidant capacity found in FDBX (95.69 ± 0.15 %) presented significant differences (p≤0.05) regarding the percentages of inhibition found in BPX60, BPX70 and BPX80 with 88.07 ± 0.38, 47.51 ± 1.65 and 47.26 ± 0.56 % respectively. The BPX70 and BPX80 powders showed the lowest inhibition percentages, which was due to their exposure to high temperatures over longer periods of time (Miranda *et al*., 2010; Morales *et al*., 2012).

There are several methodologies to determine the antioxidant capacity, being DPPH and ABTS the most used due to various advantages such as their stability against changes in pH, economy, and easy implementation (Zulueta *et al*., 2009) However, it is sometimes advisable to make the comparison by two methods because of the complexity of the chemical components in a sample which can absorb light in ranges similar to those of the free radical (DPPH or ABTS). Therefore, in this study, the antioxidant activity was evaluated using DPPH (515 nm) and ABTS (734 nm) to verify that the components present in BPX (mainly betalains which absorb light between 480 and 530 nm) did not generate an underestimation or overestimation of the antioxidant activity. The results obtained with ABTS showed that FDBX had the highest antioxidant capacity 11.62 ± 0.60 (TEAC/g_{sample}), followed by BPX60 7.63 ± 0.96 (TEAC/g_{sample}), BPX70 5.01 ± 0.23 (TEAC/g_{sample}) and finally BPX80 4.79 ± 0.11 (TEAC/g_{sample}) which confirms the influence of drying temperature on the preservation of antioxidant compounds. These results correspond with the tendency obtained with the DPPH method (FDBX>BPX60>BPX70>BPX80) attributing the highest antioxidant activity to FDBX due to the freeze-drying process which contributes to minimizing the loss of thermosensitive antioxidant compounds compared to convection drying, where high processing temperatures are used, which favor the oxidation of these compounds (Ratti, 2013; Shukla, 2011).

The Trolox equivalent antioxidant capacity (TEAC) reported by Guzmán-Maldonado *et al*. (2010) in the xoconostle cv Cuaresmeño shell was 14.5 (TEAC/100 g). On the other hand, the antioxidant capacity in percentage of inhibition of DPPH radical reported by Osorio-Esquivel *et al*. (2011) for the epicarp, endocarp and mesocarp of the fruit of *Opuntia* joconostle was 62.96 ± 0.5, 51.70 ± 0.55 and 42.27 ± 0.5 % being these lower than the percentages of inhibition found in our investigation for FDBX and BPX60. Although FDBX had the highest antioxidant capacity compared to convective drying samples, it is important to mention that, considering the thermal treatment between both methods, the BPX60 treatment is largely more favorable to preserve the antioxidant function and a viable alternative for drying the BPX.

3.6.2 Total phenolic content

The total phenolic contents (Figure 3) of the powders produced by convective drying were influenced by the effect of the temperature. There was a decrease in the content of total phenolic compounds as the temperature increased for a range from 72.02 ± 0.01 to 72.56 ± 0.06 mg GAE/100 g_{sample}. On the other hand, freeze-drying the xoconostle by-product (FDBX) produced the highest content of total phenolic compounds (73.16 ± 0.05 mg GAE/100 g_{sample}), indicating a significant difference between the two drying processes. This outcome could be attributed to the fact that freeze drying is a process that reduces the losses of volatile compounds (Ratti, 2013; Shukla, 2011). Hernández-Fuentes *et al*. (2015b) reported a total phenolic content of 108 ± 2.0 GAE/100 g in fresh pulp of the xoconostle cuaresmeño fruit (*Opuntia matudae*), which is greater than the values found in this study. However, it is worth mentioning that BPX60 came from the by-product of the extraction of xoconostle juice (mucilage of the epicarp and endocarp). Furthermore, the total phenolic content is high considering that the BPX60 was subjected to a heat treatment, and it has a phenolic content higher than that reported by De Oliveira *et al*. (2017) and Sedej *et al*. (2011) in sorghum and wheat flours, respectively, and comparable to that reported

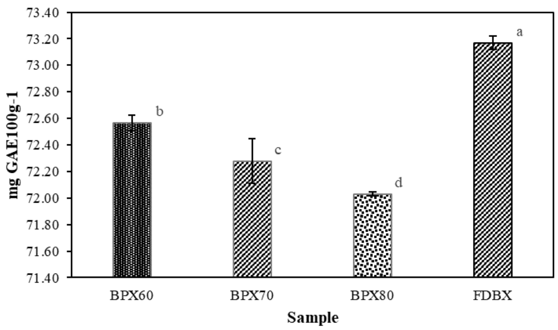

Figure 3. Total phenolic content of xoconostle by-product powders. The results are expressed in mg GAE/100 g (mean ± SD, n = 3). Identical letters above the bars indicate that there were no significant differences (significance level $p \leq 0.05$). BPX60: sample dried at 60 °C; BPX70: sample dried at 70 °C; BPX80: sample dried at 80 °C; and FDBX: freeze dried sample.

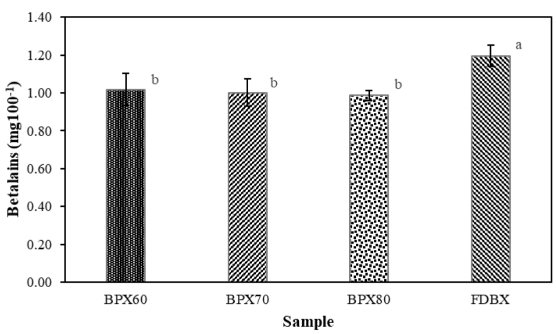

Figure 4. Total betalain content percentage of xoconostle by-product powders. Results are expressed as mg/100 g (mean ± SD, n = 3). Identical letters above the bars indicate that there were no significant differences (significance level $p \leq 0.05$). BPX60: sample dried at 60 °C; BPX70: sample dried at 70 °C; BPX80: sample dried at 80 °C; and FDBX: freeze dried sample.

by Durazzo *et al*. (2014) in a commercial carob flour.

3.6.3 Betalains

The average content of betalains in the BPX60, BPX70 and BPX80 powders (Figure 4) was not significantly different, which suggests that the convective drying operating temperature did not influence the betalain content. Regarding the drying method, there were significant differences in the betalain content, where the FDBX powder had the highest pigment content (1.19 ± 0.05 mg/100 g_{sample}). Hernández-Fuentes *et al*. (2015b) reported a betalain content of 1.70 ± 0.37 mg/100 g in fresh pulp from xoconostle cv Cuaresmeño (*Opuntia matudae*). Therefore, it can be established that the total betalain content found in the BPX60 powder (1.02 ± 0.08 mg/100 g_{sample}) was considerable. Due to the above, the convective drying at 60 °C can be considered as an alternative for the utilization and retention of betalains that confer functional properties in BPX (Ruiz-Gutiérrez *et al*., 2015).

3.7 Flow properties of the powders

The study of the flow properties of powders is important, since these parameters provide a description of the powder behavior, and these parameters impact the design of equipment for handling and transporting of the powders as well as their packaging (Stasiak *et al*., 2013). The flow properties for FDBX, BPX60, BPX70 and BPX80 are shown in Table 6. FDBX presented the lowest apparent density (0.30 g/mL), while BPX80 had the highest apparent density (0.41 g/mL). This was a consequence of the time of exposure to high temperatures that gave rise to structural modifications, such as contraction of the material and consequent cellular collapse, which caused an increase in the apparent density (Calín-Sánchez *et al*., 2015; Veras *et al*., 2012). Moreover, BPX60 and BPX70 did not show significant differences ($p \leq 0.05$).

In relation to the packed density of the samples obtained by convective drying (BPX60, BPX70 and BPX80), the collected values showed a decreasing behavior as the drying temperature increased, while the freeze-dried sample (FDBX) had the lowest packed density (0.50 g/mL) compared to that for the convection-dried samples. According to Calín-Sánchez *et al*. (2015), the final behavior of the packed density of the recovered powders could be influenced by the moisture content of the samples, with a lower packed density at a higher moisture content.

The ease of powder flow can be observed when it is subjected to compression. The Carr's index indicates the compressive capacity of powders, and it has been established that when CI > 32, a powder has a low flow capacity (Dima *et al*., 2016). The flow capacity properties of the resulting powders (BPX60, BPX70, BPX80 and FDBX) are listed in Table 5, it can be observed that BPX80 had the best flow capacity (lesser CI) compared to that for BPX60, BPX70 and FDBX, with statistically significant differences ($p \leq 0.05$).

On the other hand, the HR indicates the cohesion capacity of the particles given by the friction between them (Bian *et al*., 2015; Leturia *et al*., 2014). It was established that when the HR has a value higher than 1.4, powders have a high cohesion and little fluency (Gawalek *et al*., 2017). Based on this, it can be stated that the powders from the different drying conditions (BPX60, BPX70, BPX80 and FDBX) displayed a high cohesion between the particles, thus affecting the flow capacity. The BPX70 and BPX80 powders exhibited the best fluidity with respect to the other analyzed conditions, therefore establishing that an increase in the processing temperature during convective drying caused a decrease in the particle cohesion and CI, giving rise to a better flow capacity. This behavior could be associated with the formation of crystalline bodies that are formed during convection drying (Michalska *et al*., 2019) and that, according to Bhandari, (2013) have greater ease of flow compared to amorphous bodies usually obtained by freeze drying. The low fluency of the powders may also be associated with the sugar content in the obtained samples (Amagliani *et al*., 2016) and as it is shown in Tables 3 and 5, an inversely proportional correlation between IC and total carbohydrate content, having a better flow behavior at a lower carbohydrate content.

3.8 Hygroscopicity

Hygroscopicity is a property from granular materials that depends upon the porosity of a material (Ernesto and Ortiz, 2005). It is defined as the ability to absorb water steam from the atmosphere at a constant temperature with changes in relative humidity. Moisture adsorption can result in physical (caking) and chemical (microbial growth and taste deterioration) changes, which can severely affect sensory qualities (Moondra *et al*., 2018).

A statistically significant difference ($p \leq 0.05$) was observed between the samples dried by convection (BPX60, BPX70, and BPX80) and freeze drying (FDBX) (Table 6). The powder with the highest hygroscopicity was the FDBX powder because drying by this method allowed the formation of amorphous powders that had a higher degree of hygroscopicity (9.29 ± 0.48 g/100 g). Meanwhile, the convection dried samples showed an increase in their hygroscopicity as the drying temperature increased, and statistically significant differences between BPX60 (7.78 g/100 g) and BPX80 (7.01 g/100 g) were found. Ouaabou *et al*. (2021) mention that the powders obtained from freeze-drying have porous microstructures, which are easily rehydrated because they have a high specific free volume in their molecular matrix so they are able to incorporate greater amounts of moisture (Joardder *et al*., 2017; Palzer *et al*., 2012). The above mentioned coincides with what was reported by García-Armenta and Gutiérrez-López, (2022) in the fractal microstructure study of food where according to their research, the application of high processing temperatures results in more homogeneous microstructures, while processing at low temperatures results in irregular microstructures, which could explain the increased hygroscopicity in FDBX.

The hygroscopicity values of the powders displayed values below 10 %, which is why the powders derived from this study can be considered as non-hygroscopic (Gopinathan *et al*., 2020), thus reaffirming their storage stability.

Table 6. Flow properties of powdered xoconostle by-products.

Sample	Apparent density (g/mL)	Packed density (g/mL)	CI	HR	Hygroscopicity (g/100 g)
BPX60	0.39 ± 0.00^{b}	0.65 ± 0.00^{a}	67.72 ± 3.23^{a}	1.68 ± 0.03^{a}	7.01 ± 0.08^{c}
BPX70	0.39 ± 0.01^{b}	0.63 ± 0.00^{b}	60.89 ± 4.69^{a}	1.61 ± 0.05^{a}	7.38 ± 0.07^{bc}
BPX80	0.41 ± 0.00^{a}	0.62 ± 0.00^{c}	50.77 ± 0.00^{b}	1.51 ± 0.00^{b}	7.78 ± 0.13^{b}
FDBX	0.30 ± 0.00^{c}	0.50 ± 0.01^{d}	63.66 ± 2.23^{a}	1.64 ± 0.02^{a}	9.29 ± 0.48^{a}

CI: Carr Index.HR: Hausner's Ratio. Identical letters in the same column indicate that there were no significant differences (significance level $p \leq 0.05$). BPX60: sample dried at 60 °C; BPX70: sample dried at 70 °C; BPX80: sample dried at 80 °C; and FDBX: freeze dried sample.

Conclusions

According to the analysis of drying kinetics of xoconostle byproducts, it can be concluded that the convection drying process was highly influenced by the increase of the drying temperature allowing to reduce the drying time from 196 to 120 minutes with an increase in temperature from 60 to 80 °C. Regarding the modelled drying kinetics in terms of Fick's law of diffusion, the data obtained were successfully adjusted, validating the use of the Fick model for the analysis of the drying of xoconostle by-products. The effective diffusivity of the by-products increased with an increase in the temperature, where the D_{ef} at 60 °C (BPX60) was significantly lower with a value of 8.109E^{-10} ± 1.731E^{-11} m^2/s. The physicochemical analysis carried out on the different powders revealed interesting nutritional characteristics such as the total content of carbohydrates which are important functional materials with vital roles in various biological functions and the ash content which represents an important source of minerals. Although the freeze-drying process was more effective for preserving the functional compounds present in the xoconostle compared to convection drying, the latter drying method is more attractive due to its simplicity, speed and low cost and, according to the results, BPX60 has an important nutritional content, high physicochemical stability and a remarkable ABTS and DPPH antioxidant capacity (88.07 ± 0.38 % DPPH inhibition - 7.63 ± 0.96 TEAC/g$_{sample}$), thus convective drying is as an interesting alternative for the processing and use of BPX for its application as a functional additive, specifically in antioxidant enriched formulations.

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