Chapter 5

Spray Drying of Xoconostle Juice: Interaction of Microstructure, Function, and Drying Operation Conditions

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Abstract

The xoconostle fruit (*Opuntia matudae*) is rich in polysaccharides, soluble fiber, simple phenols, betalains, and ascorbic acid. However, its consumption is limited due to its high acidity. Spray drying could be a technological option to strengthen the sustainability of xoconostle giving a re-valorization as a possible natural additive for the food industry. The food powders have to be designed considering aspects related to the effect of processing conditions on final quality properties; in this case, the effect of different drying air temperatures was evaluated on moisture content, water activity (Aw), glass transition temperature, microstructure, antioxidant activity, phenolic, and betalain compounds. For all cases, the drying air temperature had a positive effect on physical stability, at low levels of water activity and moisture content, and glass transition temperature (T_g) was increased. The biological functionality (assessed through phenolics, betalain compounds, and antioxidant activity) was also kept constant for all processing conditions investigated. However, the most evident changes were observed at microscopic scale analyzed through morphometric parameters.

Keywords: spray drying, food powders, xoconostle juice, microstructure, betalains, phenolics content, antioxidant activity

1. Introduction

In food, pharmaceutical, and biotechnology industries, spray drying is the most used drying method. In this, a material in liquid state is fed by atomization in the form of fine droplets into a drying chamber, to obtain a solid product (powder). The outlet temperature of the dried product is between the wet bulb temperature and the outlet drying air temperature, remaining below 100°C [1]. Some factors that have shown effect on the final quality characteristics of food powders obtained by spray drying are the type of atomizer (two-fluid nozzle, sonic nozzle, spray nozzle, or rotatory disc), the physical properties of fed materials, heat and mass transfer phenomena, the average size of the atomized drops and their distribution, particle trajectory, size, and speed of particles [2, 3].

The food powders with high sugar content can be classified into two groups: sticky and non-sticky. The non-sticky products such as milk products and egg powders are obtained by spray drying in simple operating conditions and are characterized by free-flowing powders [4]. The powders obtained from fruit juices, honey, and lactose are examples of sticky products. These products are difficult to dry in a spray dryer due to their high sugar content (sucrose, glucose, lactose, and fructose), organic acids (citric, malic, and tartaric acid) [5], and hygroscopic characteristics. To improve the efficiency of drying process of sticky materials, agents of high molecular weight such as maltodextrins are used because of their high solubility, low viscosity, and high glass transition temperature [4, 6].

2. Drying

Drying is defined as the elimination of water in relatively small quantities of certain materials, under controlled conditions. Drying or dehydration of biological materials, in particular food products, is used as a conservation method [7]. The microorganisms that cause food decomposition cannot grow and multiply in the absence of water, and many enzymes that cause biochemical changes in the food and other biological materials may be inactive [8]. Microorganisms are inactive when the moisture content is below 10%. However, it is usually necessary to reduce the moisture content below 5% of weight in food to preserve its flavor and nutritional value [9].

3. Spray drying

Spray drying, as unitary operation, is used to transform liquid into solid particles (powder), eliminating moisture. In food industry, spray drying is widely used as an encapsulation method for food ingredients such as flavors, vitamins, minerals, dyes, waxes, and oils, in order to protect them from environmental stress and prolong the storage stability [10, 11]. Spray drying is commonly used in the food industry since, in comparison with other encapsulation techniques, its cost of production is low, has high availability of equipment, and also produces particles of good quality [12]. The fed materials (solution, emulsion, suspension) are atomized within the drying chamber in the form of very small drops (between 20 and 250 μm). This increases the total contact surface, improving the evaporation of the solvent (usually water).

When the product is encapsulated by spray drying, the nucleus exists as microparticles or droplets distributed within the dry solid capsule. According to the size of the particle, the encapsulation is classified as macro (>5000 μm), micro (0.2–5000 μm), and nano (<0.2 μm). Some authors point out that spray drying can be considered a sustainable process for the consolidation of nanoparticles within a spherical particle of micron size, but with properties in nano scale [13, 14].

Spray-drying equipment components are heating system and circulation of air, atomizer (nozzle or rotary disk), and powder recovery system. The most important stage of spray drying is atomization, because this affects the size, distribution of the droplets and, consequently, the final particle size. The most used atomizers are the rotary disc and pressure nozzle. Two-fluid pneumatic nozzles are used in special applications or in low-capacity dryers in pilot plants. Surface tension, viscosity, and density are the main characteristics of liquid that influence atomization [15]. Processing parameters such as configuration of dryer and operating conditions exert a significant influence on the properties of the final product [16, 17].

3.1. Drying stages

The main stages of a dryer spray are atomization of the fed product, contact air-drop, evaporation of moisture from the product, and recovery of the dry product [15].

a. Atomizing—this stage generates fine drops, increasing the relationship surface-mass, and it is the key parameter to determine the size of the particle. In all cases, the atomization occurs when the magnitude of the disruptive force exceeds the size of the surface tension [15].

b. Contact of drying air droplet—at this stage, the atomized droplets fall into a hot air flow inside the drying chamber. The contact can be on different arrays of liquid flow with the drying air flow (co-current, countercurrent, or mixed flow) [18].

c. Evaporation—the process of elimination of solvent contained in the droplet is divided into two phases. In the first stage, the droplet is heated when contacting with hot air, achieving a value closer to the wet bulb temperature, which corresponds to the drying air condition; in the second stage, a shell is formed, and diffusional process of water and soluble solids are detected [19].

d. Solids recovery—in the last stage, the powder is collected in a cyclone, which consists of a cylinder with a tangential opening through which the gas flows with particles generated in the dryer [20].

During evaporation stage, in the first step, the drying rate is almost constant, at which the surface of the drop remains saturated by the migration of water from the inside to the outside of the particle, and at the same time the solutes are concentrated, and the droplet diameter can be reduced. On the contrary, the drying air temperature decreases, and the particle temperature is increased [21]. In the second step, two regions of the particle are distinguished: a dried shell and a wet core. Drying of the particle speed is controlled by the moisture diffusion from the center to the surface of the shell. As a result of drying, the thickness of the crust increases, and its temperature rises to the set by the end of the process. Once the moisture content drops to a minimum value, the product is considered as a dry particle [22, 23].

3.2. Spray drying of high-sugar products

Food products dried by this process can be classified into sticky and non-sticky products. This categorization is relative, since some non-sticky products behave in an opposite manner, depending on the process conditions and hygroscopicity [24].

The non-sticky products can be treated through a simple dryer, and the powders obtained have a low hygroscopicity and can be classified as free-flow powders. The non-sticky products are dairy powders, micro-encapsulated powders, and powdered white egg. These powders can be dried in single operating conditions and are characterized by dust that flows free [5, 25].

In sticky products such as juicy fruits and vegetables, the spray drying treatment is usually difficult [24]. Sticky products hinder spray drying in normal conditions, such as powders obtained from the juice of fruits and vegetables, honey powder, and lactose powder. This is mainly due to the high content of sugars (sucrose, glucose, lactose, and fructose) and organic acids (citric, malic, and tartaric acid). During drying a product with a high content of sugar, the viscosity of the drops increases until it reaches a critical value (10^7 Pa s), making it a rubbery product prior to obtaining an amorphous structure. This kind of gumminess is considered as a sticky structure that is linked to the water activity (Aw) and temperature, which is between the glass transition temperature and the temperature at which the product is sticky (10–30°C) [26]. When the surface of the droplet reaches a sticky state, it raises the coalition with any area of the surface of another particle. This accumulation or agglomeration depends on the speed, strength, angle, and contact time. The result is considered negative for the case in which the particles adhere to the internal walls of the dryer chamber, causing a loss of the product. However, there are positive benefits when the particles are linked among them, contributing to the drying and changing its structure, forming clumps [27]. The moisture content in sticky products such as juice powder fruit must be between 2 and 4% [25]. Controlling the moisture content is very important, since it has a direct effect on the powder quality.

4. General characteristics of powders

Spray drying of liquid foods or biological materials produces amorphous particles mainly due to the rapid evaporation and the short time for crystallization [28]. Since morphological features are created by the loss of moisture, the rigidity of the surface of amorphous particles depends on the temperature and moisture content [28, 29].

The formation of microparticles takes place when the sprayed droplet enters in contact with the gas (hotter than 100°C). While the moisture is removed, the shell particle is formed, creating structures with different shapes. The vapor produced inside particles expands and collapses the particle structure; therefore, such vapor is primarily responsible for the powder development [30, 31].

When the initial solution is a homogeneous product, it is transformed into two layers; one of them is dense, and the other reveals a porous surface. However, the development of particle microstructure is complex due to the interaction of drying operation conditions and physicochemical liquid-fed composition [31, 32]. When using maltodextrin, changes in the morphological structure of particles are related to the moisture content of material and drying air temperatures. At low temperatures, particles with rough surface were observed; at high temperatures, a smooth surface (but with a greater number of particles broken or fragmented) was developed [31].

4.1. Morphology and microstructure

The particle morphology is described in terms of particle size, shape, surface properties, and internal structure [33]. It is determined by the physical and chemical properties of the particle shell, which at the same time depend on the composition and concentration of power, viscosity, and drying air conditions [34].

Optical microscopy, confocal laser scanning microscopy, and electron microscopy scanning are different techniques used to characterize powder (primary and secondary or agglomerates). Additionally, through digital image analysis, quantitative information is obtained [30, 35]. The scanning electron microscopy (SEM) is one of the most appropriate techniques for the characterization of the morphology and microstructure of food. It is based on the scanning of the sample surface, and it produces results in the form of digital images obtained directly from the microscope. The images can be translated into numeric data for subsequent statistical analysis, and its quantification at any resolution scale may indicate structural changes due to processing [36].

In this sense, digital image analysis is a useful tool that allows using an image to quantitatively describe the different morphometric, colorimetric, and statistical characteristics. Quantification and classification of images via dimensional descriptors such as shape, texture, diameter, and area allow the identification of structural changes at any resolution level. Digital image analysis can be used to evaluate, compare, and characterize microencapsulation under different processing conditions [37, 38].

5. Case study

A fruit of special interest is the xoconostle (*Opuntia matudae*). It is an acid fruit containing a significant amount of polysaccharides, soluble fiber, simple phenols, betalains, and ascorbic acid (which confers antioxidant capacity) [39], among other compounds such as proteins and ashes. In order to improve the sustainable development in rural agricultural areas, the processing of xonocostle could be done by the spray drying of the juice, and the resulting product could be used as a food additive. However, due to its high sugar content, the juice has to be added with a drying adjuvant such as maltodextrin in order to improve the drying process.

5.1. *Opuntia xoconostle* fruit

The xoconostle fruit is a pyriform berry with an apical depression or receptacle. It is composed of the epicarp (skin or shell), mesocarp (pulp), and the endocarp where the seeds are tightly bound in a mucilaginous structure [40] (**Figure 1**). In the genus Opuntia, a wide variety of species that produce xoconostle fruit have been reported, including *Opuntia joconostle* and *Opuntia matudae* (*Xoconostle cuaresmeño*). Xoconostle is an acid fruit that has been underused and considered as an agricultural waste product, although it contains a significant amount of fiber, minerals, phenolic compounds, betalains, organic acids, and waxes [39] with an important nutritional contribution, which makes it an attractive product.

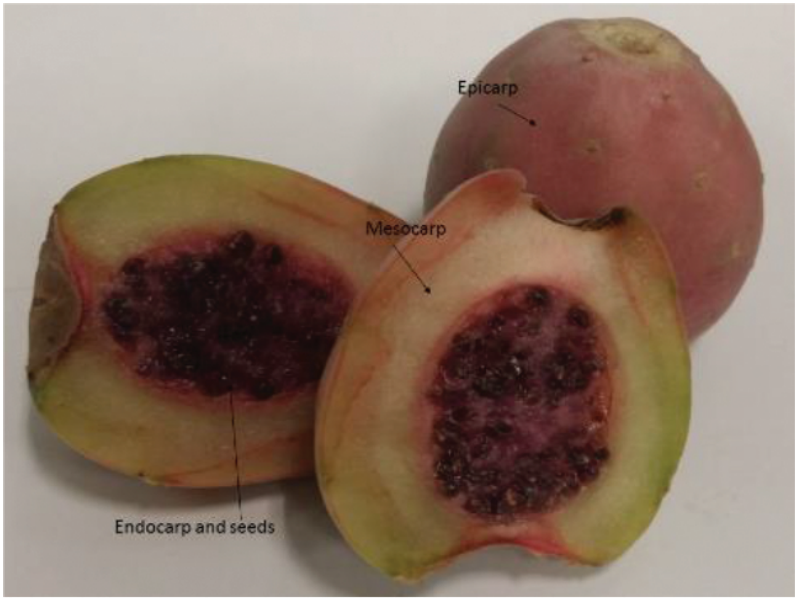

Figure 1. Image of whole and half of *Xoconostle cuaresmeño* fruit. The arrows show the epicarp, mesocarp, endocarp, and seeds.

5.2. Production of xoconostle juice and powder

Xoconostle cuaresmeño (*Opuntia matudae*) of the municipality of San Martín de las Pirámides, Mexico State, Mexico, was collected. The xoconostle batch was put in wooden boxes with kraft paper covering the fruit. Then, it was stored under refrigeration conditions until processing.

Juice extraction was performed by subjecting the fruit clean, after cut into slices, to water bath using the 2:1 ratio (fruit-water) at 70°C for 20 min. Then it was crushed in a pulper machine. The fruit juice was extracted including compounds of the mucilage and seed. Maltodextrin 20DE was added in the ratio 1:1 w/w (with respect to xoconostle total solids). Maltodextrin was used as adjuvant for spray drying juice. After obtaining the blend, an aliquot was separated for analysis, and the rest was spray dried.

A two-fluid nozzle laboratory co-current spray dryer (Mobile Minor 2000, GEA Niro, Denmark) was used, equipped with a peristaltic pump (Model 520, Watson Marlow, USA) for liquid flow control. The spray drying was conducted under 110/60, 155/70, and 200°C/80°C, as inlet/outlet drying air temperature.

5.3. Characterization of xoconostle juice and powder

5.3.1. Degrees Brix (°Bx)

The degrees Brix refers to the amount of dissolved solids extracted from fruit or sugary liquids. The method is based on the change of direction of the light because of the separation of two media in which the velocity of propagation is different. Degrees Brix reading was carried out with a refractometer (Master T, Atago, Japan). Measurements were done in triplicate.

5.3.2. Powder moisture content (MC)

One gram of powder sample was poured into an aluminum tray (constant weight) and was placed in a vacuum oven at 60°C for 24 h [41]. Measurements were done in triplicate.

5.3.3. Water activity (Aw)

One gram of xoconostle powder sample was put in a cell and then subjected to scanner computer activity using a water activity analyzer (AquaLAB 4TE, Decagon Devices, USA), at a constant temperature of 24 ± 1°C. Measurements were done in triplicate.

5.3.4. Glass transition temperature (T_g)

Fifteen milligrams of xoconostle juice powder were poured into an aluminum capsule to be analyzed using a calorimeter (DSC Diamond, Perkin Elmer, USA). The temperature interval of the analysis was from −40 to 120°C, at a heating speed of 5°C/min. The resulting thermograms were analyzed to determine the glass transition temperature (T_g) [42].

5.3.5. Scanning electron microscopy (SEM)

To assess the morphology of microparticles, powders were fixed on carbon tape placed on a specimen slide, and the remainders were removed. Samples were observed by means of a double-beamed scanning electron microscope (Dual Beam Nova 200 Nanolab, FEI) operated at 1.00 kV (for sensible materials), a total amplification of 1000×, 3000×, and 5000× [30]. Measurements were done in triplicate.

5.3.6. Particle size and shape by digital image analysis

To assess the distribution and particle size, powder samples obtained after spray drying were analyzed using software ExpertShape, where the acquisition of images was carried out manually using the light microscope (CILAS 1090-ExpertShape-NT 2107380, France), which has a video camera with a peak bandwidth of 23.2 MB/s. A microscope suited with a 10×

objective lens was used. Illumination was provided by a light-field source. A total of 1300 particles were analyzed for each experiment.

For particle measuring, software ExpertShape acquires the image of a particle defined as a group of contiguous nonzero pixels. Particles can be characterized by a relationship of measures depending on their attributes such as location of the particle, area, and form factor. The coordinates are expressed with reference to the origin (0, 0), located at the upper left corner of the image. When extracting a particle in the image, the outline is defined by the projection of the sensor cutting. Contour is defined by scanning the sequence of adjacent pixels for each particle. The outline is drawn using the chain of “Freeman” code (gray-level image, the thresholder image, the shape without holes and smooth contour, and extracted contour with Freeman chain-code algorithm). Some morphological parameters that could be evaluated are area (A); perimeter (P, as the length of the contour of the particle); *mean Feret diameter* (Calipter diameter) defined as the distance between two tangents on opposite sides of the particle, parallel to some fixed directions, touching opposite sides of particle; *maximum Feret diameter*, as the length of particle; *minimum Feret diameter* is the width of particle; *equivalent circular diameter* is calculated as the circumference with the same area as the projected particle; *roundness* is defined as the ratio of the area of a circle (the most compact shape) that has the same perimeter, and the closer the shape of the particle is to a disk, the closer the roundness is to 1; *equivalent ellipse ratio*, as the ratio of the major axis to its minor axis (elliptical shapes exhibit ratio >1) [43].

5.3.7. Total phenols by Folin-Ciocalteu method

Total phenols of xoconostle juice and powder were obtained by the method of Folin-Ciocalteu to determine the thermal damage caused by drying air temperature as an operation condition of spray drying.

For the extraction of total phenols, 1 g of powder was mixed with 5 mL of 80% methanol and shaken for 30 min at 200 rpm at room temperature. The sample was decanted using filter paper of 110 mm (qualitative circles Whatman 3). The supernatant was separated and placed into an amber glass bottle. The sediment was reconstituted, using 8 mL of 80% ethanol, and the procedure was repeated twice. Thereby, three extractions were joined to be analyzed. For the xoconostle juice, a sample of 4 mL was used, following the previous methodology. For the quantification stage, 0.75 μL of Folin-Ciocalteu reagent (1:10) was added to 100 μL of the extract, and the mixture was left to stand for 5 min in the dark. Subsequently, 0.75 mL of $NaHCO_3$ 60 g/L solution was added to neutralize the reaction. The solution was left to stand for 90 min, and the absorbance was determined at 725 nm. The results were reported as mg gallic acid equivalents/100 g (db) [44]. Measurements were done in triplicate.

5.3.8. Extraction and quantification of betalains

One gram of powder sample was mixed with 5 mL of a solution: methanol ((80:20 v/v), stirring (at maximum speed) using a magnetic stirrer for 30 min and 5°C. The solution was filtered using a membrane pore with a size of 110 mm. The obtained extract was analyzed, expressing the result as mg of betacyanin/100 g of sample (db), since it has been reported that betacyanins are the main components of betalains [45, 46].

5.3.9. Antioxidant activity

ABTS (7 mM) radical cation (ABTS•+) solution was prepared by reacting ABTS (2, 2'-azino-bis(3-ethylbenzothiazoline-6-sulphonic acid)) with 2.45 mM potassium persulfate and allowing the mixture to stand in the dark at room temperature for 12–16 h. The ABTS•+ radical was diluted with ethanol to give an absorbance of about 0.700±0.02 at 734 nm. In order to measure the antioxidant capacity, 10 μL of the extract was mixed with 990 μL of radical solution. The absorbance was monitored at 734 nm for 7 m. All experiments were carried out in triplicate. The results were expressed in terms of mM Trolox equivalent/g sample (mM TE/g, db) [44, 47].

5.4. Results and discussion

Spray drying of xoconostle juice was conducted with low content of maltodextrin as carrier substances, improving the drying characteristics, avoiding technical problems such as the presence of sticky powder and the accumulation of wet material on the walls of the drying chamber and the mechanical cyclone. Although xoconostle juice contains a very low solid concentration (3°Brix), this value was the reference to choose the quantity of carrier agents to be mixed with the juice. The relation of juice solids-maltodextrin (1:1) was enough to reach yield levels from 42 to 79%, the best drying condition being at 110°C/60°C, to obtain the highest powder recuperation (**Figure 2**). This fact could be affected by the powder density and mean particle size, by keeping constant the concentration of the feed flow and atomizer pressure (0.6 Bars). Low drying air temperature produces compact and small particles, as reported by Alamilla-Beltrán et al. [31].

Figure 2. Images of the xoconostle juice and the xoconostle juice powder obtained by spray drying at 110°C/60°C as inlet/outlet drying air temperatures.

Moisture content, water activity, and glass transition temperature (T_g) were evaluated for all conditions, obtaining values of 4–4.98% (db), 0.19–0.23, and 34.37–38°C, respectively

(**Table 1**). The results showed that powdered xoconostle juice was highly stable due to reduced values of moisture and water activity, improving storage stability with the incorporation of a carrier of high molecular weight such as maltodextrin by increasing the glass transition temperature, and considering that fructose has lower values of T_g (16–17.6°C).

Parameters	Drying air temperature (inlet/outlet) (°C)		
	110/60	155/70	200/80
Moisture content (% db)	4.99	4.07	4.88
Water activity (Aw)	0.19	0.22	0.23
Glass transition temperature (°C)	34.70	34.75	38.45
Feret diameter mean (μm)	14.76	16.11	16.12
Feret diameter max (μm)	12.12	11.12	13.27
Feret diameter min (μm)	16.87	17.76	18.40
Equivalent elliptical ratio	1.94	1.80	1.85
Form factor (roundness)	0.8055	0.8571	0.8453
Phenolic content (mgGAE/100 g)	1250.10	1365.60	1359.10
Betalain content (mg/100 g)	2.65	2.14	2.42
Antioxidant capacity (mM TE/g)	65.08	64.39	67.26

Table 1. Mean values of different parameters evaluated for xoconostle juice powder obtained by spray drying process at three different inlet/outlet drying air temperatures.

This variety of fruit contains compounds such as oxalates, organic acids, vitamins, and wax. In this study, the effect of these has not been analyzed; however, it could exist as an important interaction between wax and maltodextrin, which may improve the protective effect upon powdered xoconostle juice. Regarding the drying temperature, the highest condition improves the reduction on powder moisture content, although this difference is minimal. No evident difference was found in the water activity values, which means that drying temperatures do not affect this parameter, getting low values in all cases. At all combined values of water activity and moisture content obtained in this work, biochemical reaction and enzymatic activity were inhibited, and bacterial growth could be reduced.

During spray drying, mass and heat transfer mechanisms act inducing the evaporation of water; meanwhile, solids and moisture content diffuse from the inside of droplets to the outside, developing solid particles. This migration of components was induced by the air-drying temperature and diffusion factors, to create powders with microstructures that influence the final quality of powders.

Powder microstructure could be analyzed by using diverse methods. In this work, scanning electron microscopy (SEM), optical microscopy, and digital image analysis provided information about morphometric parameters such as size, perimeter, area, shape, and structure of microparticle (**Table 1**). Under all conditions, the powdered xoconostle juice (analyzed with SEM) revealed spherical individual particles (primary particles) forming weak agglomerates (secondary particles). The shape of primary particles tends to be spherical with slightly deep depressions of surface; meanwhile, agglomerates form an elliptical shape, without a structured order. Some particles exhibit an external surface with highly porous aspect. This fact could be caused by a selective migration of solutes dissolved in the xoconostle juice. Solutes with low molecular weight could diffuse quickly to the surface forming the porous shell, which means that sugars may be structured and form the external surface. These kinds of particles are not desirable due to the high hygroscopicity of sugars. For all drying temperatures, particle microstructure was similar, with differences assessed by morphometric parameters.

Morphometric parameters evaluated by optical microscopy and digital image analysis described differences in particles for all experimental conditions. In this case, knowledge on morphometric parameters is useful to improve the particle description, so identifying any change induced by the processing conditions (drying air temperature, atomizing pressure, and concentration of feed flow) is useful to improve the spray drying process. This means that any change in the processing conditions will be translated into physicochemical properties of powders. In this work, the morphological parameters gave information about dimensions and particle shape.

The highest particles measured by mean Feret diameter (16.12 μm), maximum Feret diameter (18.4 μm), minimum Feret diameter (13.3 μm), and area (209 μm^2) were obtained at 200°C/80°C. These results could explain the lower moisture content and water activity values obtained at 200°C/80°C, and due to particle expansion, the moisture diffusion could be facilitated through thin wall particles. The opposite was observed in products obtained at reduced drying air temperatures.

Although primary particles observed by SEM seem to be spherical and the agglomerates showed an elliptical form, form factor (or roundness) and equivalent ellipse ratio were calculated as a descriptor of bulk powder. Values of roundness were close to 0.85 for all conditions, being lightly higher for particles obtained at 200°C/80°C, so it could be related to particle expansion and less formed agglomerates (1.84 as equivalent elliptical ration). In the case of 110°C/60°C, this parameter was 1.94.

For all drying air temperatures, total phenolics (1250–1366 mg GAE/100 g), betalain content (2.14–2.65 mg/100 g), and antioxidant activity (64–67 mM TE/g) did not exhibit significant differences. This means that any option could be applied to produce a powdered xoconostle juice containing phenolic compounds and betalains; however, microstructural, physicochemical, and flow properties have to be considered when a powder with functional properties is designed.

6. Conclusions

The spray drying process has been widely used as a useful tool to generate functional products in the form of powders. The processing of xoconostle juice by spray drying allows proposing an option to strengthen the sustainable use of this product as a natural additive in the food Industry, reinforcing the valorization and reutilization of valuable components found in the agro-industrial waste. By keeping constant the drying conditions (such as feed concentration, atomization pressure, flow arrangement, and type of atomizer) and changing the drying air temperature, the powdered xoconostle juice was obtained.

For all cases, the drying air temperature had an evident positive effect upon physical stability given by the low values of water activity and moisture content, and the increase in T_g by the addition of maltodextrin. The biological functionality (evaluated by total phenolics, betalains, and antioxidant activity) was also kept in similar values for all drying conditions. The most evident changes were observed at microscopic scale. For designing similar products, it is necessary to consider the interactions between food properties (microstructural, functional, physicochemical, and flow) to take the best decision.

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