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Impact of Spray Drying on the Properties of Grape Pomace Extract Powder

Betina Louise Angioletti Decker [1], Emilio de Castro Miguel [2], Thatyane Vidal Fonteles [3], Fabiano A. N. Fernandes [1] and Sueli Rodrigues [3,*]

[1] Chemical Engineering Department, Federal University of Ceara, Fortaleza 60440-900, Brazil; blangioletti@gmail.com (B.L.A.D.); fabiano@ufc.br (F.A.N.F.)
[2] Metallurgical and Materials Engineering Department, Federal University of Ceara, Fortaleza 60440-554, Brazil; emiliomiguel@ufc.br
[3] Food Engineering Department, Federal University of Ceara, Fortaleza 60440-900, Brazil; thatyanevidalfonteles@ufc.br
* Correspondence: sueli@ufc.br

Abstract: Incorporating anthocyanins, valuable natural pigments, into a powder can improve their stability, but exposure to high temperatures during processing can cause them to degrade. The purpose of this study was to investigate how the inlet air temperature during spray drying affects the physical and chemical characteristics as well as the flowability of a grape pomace anthocyanin powder obtained through ultrasound-assisted extraction using acidified water as the solvent. An anthocyanin solution containing 13% (w/v) maltodextrin was subjected to spray drying at temperatures ranging from 120 to 170 °C. Tukey's test was applied to compare the means of the samples. The samples dried at temperatures between 130 and 170 °C were adequate, with a moisture content < 5% and a water activity < 0.3, indicating that the powder was stable. The highest anthocyanin retention (91.94 ± 1.59%) and process yield (50.00 ± 3.06%) were achieved at 140 °C, while higher temperatures resulted in anthocyanin degradation. Furthermore, the powder exhibited poor flowability, indicating cohesive behavior (Hausner ratio > 42.29% and Carr index > 1.73), which is an industrial parameter rarely considered in spray-drying studies. The acidification process was found to promote high anthocyanin retention following high-temperature processing. However, powders obtained from food matrices with low pH and high sugar content may exhibit increased cohesion due to interaction forces. These findings highlight the potential of utilizing grape pomace and green solvents to produce bioactive-rich powders for industrial applications.

Keywords: encapsulation; maltodextrin; anthocyanin stability; bioactive compounds; malvidin; green solvents; powder cohesiveness

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1. Introduction

The growing appreciation of functional foods is driving the demand for the development of food ingredients with improved nutritional quality. In this context, much attention has been given to natural compounds and their bioactive potential [1]. Anthocyanins are water-soluble phenolic pigments that present colorimetric and biological properties. The benefits associated with anthocyanins have increased the interest in studying their extraction from various food matrices [2,3].

Grape pomace stands out as an anthocyanin-rich residue from the beverage industry due to the incomplete pigment extraction during the winemaking process. Moreover, reusing grape pomace is an alternative to reduce the negative impact of its direct disposal in nature [4,5]. Six anthocyanin types are predominant in fruits and vegetables: cyanidin, delphinidin, pelargonidin, peonidin, petunidin, and malvidin [6]. From these, glycosylated malvidins are the most abundant in grape pomace, with malvidin diglucoside being the anthocyanin most abundant in Vitis labrusca varieties [7].

Several studies have focused on extracting anthocyanins from grapes using innovative technologies. Extraction is a valuable unit operation for recovering desirable compounds. However, the extract's high water activity (a_w) and the low stability of anthocyanins under several environmental conditions make further processing, such as drying, necessary to enhance stability. Drying not only ensures extract stability but also reduces transportation and storage costs [8,9].

Among the available technologies, spray drying is the most widely used process for drying and encapsulating anthocyanins [2]. In this process, a liquid medium is transformed into a powder by spraying the liquid feed into a hot gas over a very short contact time. Therefore, this technique enables the drying of heat-sensitive compounds like anthocyanins, which are also encapsulated and protected by the carrier agent used [10].

Contrastingly, spray drying may pose challenges like stickiness and low yields due to droplet adhesion to the dryer chamber walls. The sticky behavior of sugar-rich extracts may be attributed to the presence of low-molecular-weight sugars and their low glass transition temperature (Tg). To address this challenge and raise the Tg, carrier agents can be added to the solution. Maltodextrin is particularly effective because of its affordability, high solubility, and low viscosity [3,11,12].

The inlet air temperature (drying temperature) is one of the key factors that affects the spray-drying process, playing a crucial role in the powder's physical and chemical properties. This temperature can lead to chemical changes in compounds, especially in heat-sensitive ones like anthocyanins. Additionally, the temperature directly affects the removal of water during the process, which in turn impacts important physical properties of the powder such as moisture content, a_w, and flowability [13].

Various studies in the literature have explored the ideal temperature for encapsulating extracts rich in anthocyanins. Chuwattanakula et al. [14] suggested drying Karanda juice at 174 °C (temperature range studied: 160–200 °C) using a combination of maltodextrin and whey protein. Meanwhile, Rosales-Chimal et al. [15] examined the drying process of an anthocyanin extract from *Solanum tuberosum* using taro starch and suggested 125 °C as the ideal drying temperature. The results for the drying of Bordô grape extract using maltodextrin indicated that the ideal drying temperature depended on the content of the carrier agent, with the highest anthocyanin retention achieved with 30% maltodextrin at 170 °C [16]. However, most of the studies utilized ethanolic or non-acidified aqueous extracts, which could impact the retention of anthocyanins during spray drying. It is widely recognized that anthocyanins are more stable at low pH values (1–2). As a result, the use of acidified water could be a beneficial and environmentally friendly option for enhancing anthocyanin retention during the spray-drying process [17].

Additionally, these studies primarily focused on assessing the physicochemical properties of the powders. However, given that the powders are used as ingredients for various purposes in the food industry, evaluating their flow performance is crucial. Powder flowability is often disregarded during product development but can be a significant obstacle [18]. Flowability is crucial for finding the best conditions for handling and storing the product [18,19]. Thus, this study aimed to investigate how the air temperature used in spray drying affects the physical and chemical properties and the flowability of a grape pomace anthocyanin powder obtained through ultrasound-assisted extraction (UAE) using acidified water as a solvent. The evaluation included the process yield, anthocyanin retention, moisture content, water activity, and powder flowability.

2. Materials and Methods

2.1. Plant Material and Grape Pomace Drying

Grano D'Oro grape pomace (*Vitis labrusca*) was provided by wineries from Nova Trento (Santa Catarina, Brazil). After manually removing pomace seeds, it was dried in a convective air oven (TE-394/1, Tecnal, Piracicaba, Brazil) for 5 h at 60 °C. Then, the dried pomace was ground in a laboratory mill (A10, I.K.A., Campinas, Brazil), vacuum-packaged in polyethylene bags, and frozen at −20 °C until extraction. The frozen pomace was thawed at 4 °C before the experiments.

2.2. Ultrasound-Assisted Extraction (UAE) of Grape Pomace Anthocyanins

Anthocyanins were extracted from dried grape pomace (moisture content = 4.38% and a_w = 0.249) through UAE, previously optimized (ultrasonic power = 500 W, extraction time = 5 min). The UAE of anthocyanins was optimized in preliminary tests, and the ultrasonic power and time that resulted in the highest anthocyanin content were chosen. UAE was carried out with 10 g of dried bagasse mixed with 200 mL of water acidified with hydrochloric acid, pH = 1.5, placed in a jacketed glass reactor with a capacity of 250 mL coupled to a thermostatic bath to maintain the temperature at 25 °C. The extractions were carried out in a probe ultrasound (Ultronique QR500, Indaiatuba, Brazil) working with a power density of 2.5 W/mL and frequency of 20 kHz holding a 4 mm diameter microtip submerged in the sample at a depth of 15 mm. The suspension was centrifuged at 10,000× *g* for 10 min and filtered through Whatman nº 01 filter paper. The processing was repeated 15 times to guarantee the volume necessary for the spray drying.

2.3. Production of Anthocyanin Powder by Spray Dryer

A 600 mL solution containing the anthocyanin extract and maltodextrin was prepared for each experiment. Each solution contained 80 mL of maltodextrin, corresponding to a carrier agent concentration of 13% (*w*/*v*). Drying was carried out in a spray drier (MSD 1.0, Labmac do Brasil, Ribeirão Preto, Brazil) containing a stainless-steel chamber, a spray nozzle diameter of 1.2 mm, a drying airflow of 1 m^3/min, and a pressurized airflow of 30 L/min. These conditions were set based on equipment operation conditions. The carrier agent concentration and the feed flow rate were defined in preliminary tests, evaluating flow rates of 0.5 and 1 L/h and low contents of the carrier agent. The feed flow rate of 0.5 L/min and a concentration of 13% were chosen because they resulted in powders with lower moisture content and small adhesion to the chamber wall. The inlet air temperatures were 120, 130, 140, 150, 160, and 170 °C.

The powders were collected in 250 mL screw-capped bottles and stored in a refrigerator for 24 h before moisture content, $a_{w,}$ and powder flowability analysis. For the chemical analysis, the powder was stored in laminated packages (E.S.A.) composed of P.E.T., aluminum, and polyamide and frozen at –20 °C. Before analysis, the powders were thawed at 4 °C for 24 h.

2.4. Process Characterization

The air outlet temperatures were measured with a thermocouple attached to the spray drier between the drying chamber and the cyclone. The process yield was estimated as the ratio between the mass of powder obtained after drying (g) and the mass of solids at the spray dryer inlet (g), with results expressed as a percentage (%) [15].

2.5. Physico-Chemical Properties of the Liquid and Powder Extracts

The liquid extract was characterized according to its total anthocyanin content, and the powder was characterized according to total anthocyanins, moisture content, a_w, and powder flowability.

2.5.1. Anthocyanin Content and Anthocyanin Retention

The powder was previously diluted in water in the proportion of 1 g/5 mL to determine the total anthocyanins. Before quantification, sugar was removed from the extracts by solid-phase extraction using a 6 mL Strata C18 cartridge [20].

The differential pH method determined monomeric anthocyanin content [21] by reading the absorbances at 520 and 700 nm. Pigment content was calculated as malvidin 3-glucoside (493 g/mol) using an extinction coefficient (ϵ) of 28,000 L/(cm mol) [22]. The results were expressed as malvidin 3-glucoside/L (liquid extract) and malvidin 3-

glucoside/g powder (powder extract). The measurements were carried out using an Elisa reader spectrophotometer (Biotek Epoch, Winooski, VT, USA), and the Gen 5 1.10 software was used to process the data [23,24].

Anthocyanin retention (%) was determined by comparing the total anthocyanin content in the extract before the drying process with the anthocyanin content in the powder [16].

2.5.2. Moisture Content and Water Activity (a_w)

Moisture content was measured on an infrared balance at 105 °C (ID50, Marconi). A_w was measured with an a_w meter at 25 °C (AquaLab Decagon CX-2, Pullman, WA, USA).

2.5.3. Particle Morphology

Particle morphology was evaluated by scanning electron microscopy (S.E.M.). The powder obtained from spray drying was disposed in a thin layer on the carbon tape, glued in a stub, without pressure. The excess material was removed mechanically so that only the firmly adhered particles remained on the carbon tape. Subsequently, the material was sputtered and covered with 20 nm gold. Analysis was performed under a scanning electron microscope using a Quanta 450 FEG-FEI with a nominal resolution of 5 nm and an acceleration voltage of 20 kV operating at 25 kV at 10 nm of working distance.

For particle measurements, 150 particles from different images were chosen for observation. The measurements were performed using Fiji software (Image J 1.54f, Java 1.8.0-322, Bethesda, MD, USA) [25]. The size calibration was performed according to microscope parameters.

2.6. Powder Flowability

Powder flow characteristics of the samples were evaluated using Powder Flow Tester (PFT) equipment (Brookfield Engineering Laboratories, Middleborough, MA, USA). Approximately 15 g of anthocyanin powder was placed in circular stainless-steel trays and transferred to the device, where the flow function and wall friction tests were applied to determine bulk density, Carr index (CI) [26], Hausner ratio (H.R.) [27], flow index (I_F) [28], and wall friction angle. CI, H.R., and I_F were determined according to Equations (1)–(3), respectively. Then, the powders were classified according to their flowability, according to Table 1.

$$\mathrm{CI} = \frac{\alpha_c - \alpha_a}{\alpha_c} \tag{1}$$

$$\mathrm{H.R.} = \frac{\alpha_c}{\alpha_a} \tag{2}$$

$$I_F = \frac{\sigma_1}{\sigma_c} \tag{3}$$

where CI = Carr index (%); H.R. = Hausner ratio; α_a = bulk density (kg/m^3); α_c = packed density (kg/m^3); I_F = flow index; σ_c = principal consolidation stress (kPa); and σ_1 = unconfined sliding stress (kPa).

Table 1. Classification of powder flowability [26,27].

Flowability	Compressibility Index (%)	Hausner Ratio
Excellent	<10	1.00–1.11
Good	11–15	1.12–1.18
Adequate	16–20	1.19–1.25
Acceptable	21–25	1.26–1.34
Difficult	26–30	1.35–1.45
Very difficult	32–37	1.46–1.59
Excessively difficult	>38	>1.60

2.7. Statistical Analysis

Analyses for anthocyanin retention, moisture content, and a_w were performed in triplicate. Powder flow properties were measured in duplicate. Tukey's test was carried out to compare the means of samples dried at different temperatures. All statistical analyses were conducted with STATISTICA software (Statsoft v14.0). The results were expressed as mean ± standard deviation.

3. Results

3.1. Process Characterization: Outlet Air Temperature and Process Yield

As Table 2 shows, the outlet air temperatures increased with the increase in the inlet temperature, ranging from 68.2 (120 °C) to 103.5 °C (170 °C).

Table 2. Air outlet temperature and drying yield of anthocyanin powders obtained by spray drying at different temperatures.

Inlet Air Temperature (°C)	Outlet Air Temperature (°C)	Process Yield (%)
120	68.2	45.35 ± 2.75
130	76	43.02 ± 2.67
140	82.4	50.00 ± 3.06
150	90	45.35 ± 2.34
160	97.6	48.84 ± 3.80
170	103.5	48.84 ± 3.26

The outlet air temperature is a crucial parameter in the spray-drying process. Along with the inlet air temperature, it significantly impacts the powder's physicochemical properties. Goula et al. [29] stated that there is a narrow range of adequate outlet air temperature, which depends on the characteristics of the food product and the drying process. According to the authors, the process will not be economically feasible when this temperature is below the adequate range. When the temperature is above this range (Tg + 20 °C), the powder may present a sticky behavior, influencing the process yield.

Process yield is a crucial process variable for industries since it is directly related to production costs and efficiency [30]. Retention of the product on the drying chamber wall reduces process yield and has several negative impacts on the product and process: (1) it is not economical, as it requires the dryer to be shut down more frequently for cleaning; (2) it affects product quality, as accumulated material can contaminate the powder and exhibit different properties such as moisture content, solubility, and retention of bioactive compounds due to prolonged heat exposure; and (3) retention on the equipment walls affects drying capacity and reduces the heat transfer between the chamber walls and the flowing fluids [29].

This study's process yield ranged from 43.02% to 50%, with the best yield obtained at a temperature of 140 °C (Table 2). These yields are consistent with those reported in the literature for spray drying using maltodextrin as a carrier agent for açaí extracts (39.02–55.66%) [31]. Increases in temperature from 120 to 140 °C increased the process yield by enhancing the temperature gradient between the drying air and the food product, thus improving heat and mass transfer and reducing the mass fraction of incomplete dried powder. When these incomplete dried powders hit the chamber, they tend to agglomerate. Therefore, when the drying is performed at a low temperature, a higher agglomeration in the chamber wall occurs, reducing the process yield. Then, increases in drying temperature (from a low to an adequate temperature) may reduce the powder's moisture content and improve process yield.

However, drying at temperatures above 140 °C led to a slight reduction in the process yield in this study. When the temperature is too high, it presents a reverse relationship with drying yield, resulting in greater adhesion of the powder to the chamber wall. At high temperatures, particles and the dryer chamber may stick together, forming wall deposits [32]. Adhesion to the walls begins when the product temperature exceeds Tg + 10 °C, and stickiness begins when the temperature surpasses Tg + 20 °C [33].

The literature presents conflicting conclusions about the impact of temperature on process yield. Some studies indicate that higher drying temperatures result in increased process yield. Jafari et al. [34] reported increases in the yield when changing the temperature from 124 to 143 °C during the drying of pomegranate juice, and Laokuldilok et al. [35] observed that increasing the drying temperature from 140 to 180 °C increased the drying yield of an anthocyanin extract. Tonon et al. [31] reported increases in açai powder's recovery when the drying temperature was increased from 138 to 202 °C. Some studies indicate an optimal temperature beyond which the process yield decreases. Rosales-Chimal et al. [15] observed an increase in process yield when the drying temperature increased from 90 to 125 °C, followed by a decrease at higher temperatures (125–160 °C). Goula and Adamopoulos [32] dried tomato pulp and observed that wall deposits decreased by increasing the inlet air temperature from 110 to 130 °C. In contrast, the increase from 130 to 140 °C increased the residue accumulation in the wall. Moreover, the authors suggested that the deposition began to occur when outlet air temperatures reached 80 °C.

The differences obtained for the best drying temperature from several food extracts are closely related to the feed mixture's initial composition (sugar content in the extract and type and concentration of carrier agent) and other drying conditions, such as the temperature range and flow rate. It is well-stated that increasing the concentration of the carrier agent increases the Tg [36,37], thus increasing the temperature that optimizes the yield.

3.2. Powder Characterization

3.2.1. Anthocyanin Retention

The liquid extract obtained by UAE from grape pomace anthocyanins presented an anthocyanin content of 230.49 ± 3.80 mg/L. After the spray drying, the retention of anthocyanins in the powder ranged from 64.26% (160 °C) to 91.94% (140 °C), and significant differences ($p < 0.10$) in anthocyanin retention were observed among the samples (Figure 1).

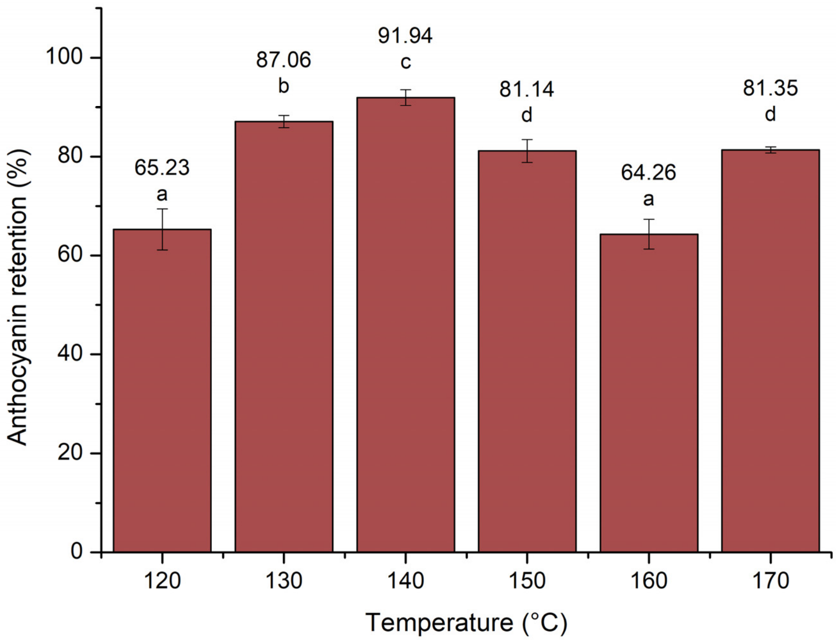

Figure 1. Anthocyanin retention of powders obtained by spray drying at different temperatures. Tukey's test compared the means of samples dried at different temperatures. Means that do not share a letter are significantly different at a significance level of 10%.

The results presented herein (Figure 1) agree with those reported by other authors. De Souza et al. [16] found anthocyanin retention between 88.35 and 97% when drying an ethanolic extract of grape anthocyanin, using maltodextrin as a carrier agent (10–30%) and inlet temperatures 130–170 °C. Tonon et al. [31] reported an anthocyanin retention of 77–86% after drying açai pulp with maltodextrin (10–30%) over a temperature range of 138–202 °C. Alternatively, Silva et al. [38] evaluated the drying of an ethanolic acidified extract. The authors produced a *jussara* anthocyanin powder and observed an 88–98% anthocyanin retention.

In this work, an acidified aqueous extract was used, and the retention reached values greater than 90%, even using low concentrations of carrier agents (13%). This is highly significant as using a carrier agent alters the composition of the final product. Maltodextrin is a carbohydrate well suited for encapsulating anthocyanins due to its high resistance to temperature and the hydrophilic character of the pigment [39]. It is also suitable for food applications and classified as generally recognized as safe (GRAS) by the Food and Drug Administration [40]. However, using maltodextrin can affect the nutritional and organoleptic properties of the food product. Higher maltodextrin contents increase the product's carbohydrate content, thereby reducing the levels of bioactive compounds and diminishing its nutritional value. Additionally, increased maltodextrin levels may impact the color of the product, as maltodextrin is a white filler that can reduce the powder's redness [9].

Anthocyanins are pigments with low stability, influenced by pH, temperature, light, oxygen, etc. Due to their ionic nature, anthocyanins can undergo several transformations depending on the pH of the solution, with the ion flavylium (pH = 1–2) being the more stable form of anthocyanin [17]. Thus, using acid solvents for extract preparation can help protect anthocyanin from degradation during spray drying. Santos et al. [41] conducted a study that produced several spray-dried anthocyanin powders and examined the effect of pH (ranging from 2 to 6) on powder stability. They observed that the lowest pH (pH = 2) led to the best anthocyanin content immediately after the spray-drying process and improved anthocyanin retention during powder storage. Similarly, Betz and Kulozik [42] compared microencapsulation of extracted berry anthocyanins using whey protein at pH 1.5 and 3 and found that anthocyanin degradation was pH-dependent, with low pH values improving pigment retention.

When studying the impact of temperature on anthocyanin retention, it was observed that an increase in retention occurred when the inlet air temperature was raised from 120 °C to 140 °C. The higher temperatures improved heat and mass transfer and reduced the deposition of the food product on the chamber wall, which may contain anthocyanins. Additionally, higher drying temperatures resulted in a quicker formation of crust on the particles, preventing the leaching of anthocyanins and enhancing heat penetration in the droplets. Silva et al. [38] observed an increase in anthocyanin retention with the initial rise in temperature from 140 °C to 160 °C, followed by a decrease as the temperature increased from 160 °C to 180 °C. Their study evaluated drying at 140 °C, 160 °C, and 180 °C.

In the present study, temperatures above 140 °C promoted losses in anthocyanin retention, likely due to thermal degradation and oxidation [34]. Although the contact time during spray drying is very short, it may be enough to cause anthocyanin degradation at high temperatures. Interestingly, an increase in anthocyanin retention was observed when the temperature rose from 160 to 170 °C. De Souza et al. [16] also noted a drop in the retention of anthocyanins between 130 and 150 °C, followed by a slight increase between 150 and 170 °C when maltodextrin was used at a concentration of 10% (like the concentration used in this study, of 13%). The authors suggest that the "increase" in retention observed at 170 °C is, in fact, an effect of the non-enzymatic browning due to the caramelization of the carrier agent. Caramelization produces dark compounds that can be quantified by the total anthocyanin content. Furthermore, colorless proanthocyanidins can be converted into colored anthocyanidins during processing, especially when acids are present and high temperatures are used. It is worth noting that, unlike anthocyanin standards, the powder produced from grape pomace extracts contains several other phenolic compounds. These compounds can act as copigments that stabilize anthocyanins and increase color retention [43]. Indeed, recent studies have combined strategies of pigmentation and microencapsulation to overcome the limitations regarding anthocyanin stability [44].

3.2.2. Powder's Moisture Content and Water Activity (a_w)

Moisture content and a_w are two key parameters that influence powder quality and safety [12,45]. The powder's moisture content ranged from 2.36 (170 °C) to 5.07% (120 °C), and the a_w ranged from 0.081 (160 °C) to 0.359 (120 °C), as shown in Figure 2. The moisture content and a_w decreased significantly ($p < 0.10$) with increases in the inlet air temperature. The results are shown in Figure 2.

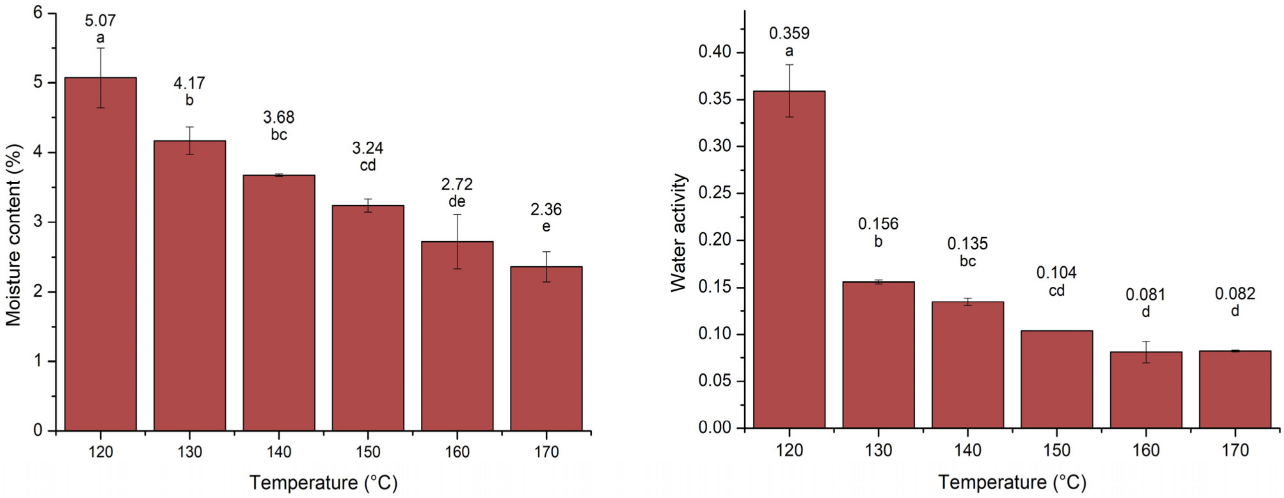

Figure 2. Moisture content and water activity of anthocyanin powders obtained by spray drying at different temperatures. Tukey's test was conducted to compare the means of samples dried at different temperatures. Means that do not share a letter are significantly different at a significance level of 10%.

In a constant feed flow rate, as applied in the present study, the higher the difference between the temperature of the atomized feed and the drying air, the greater the driving force for water evaporation, resulting in powders with lower moisture content [46]. Similar results have been observed in various products obtained by spray drying. Nguyen et al. [47] found that the moisture content decreased from 7.66 to 5.57 when the temperature was increased from 150 to 170 °C, using an anthocyanin to maltodextrin ratio of 1/100.

In addition, the moisture content and a_w values obtained in this study fall within the range of results from several other studies. For moisture content, reported ranges for spray-dried products include 3–4% for soluble sage extract [48], 0.47–2.44% for powdered blackberry extract [49], 2.28–4.18% for *jussara* anthocyanin extract [50], 3.71–4.92 for black mulberry juice powder [51], and 2.2–3.22% for chokeberry extract [52]. Reports for a_w include values below 0.26 for chokeberry juice powder [53] and between 0.216 and 0.314 for *jussara* anthocyanins powder [50]. In addition, the inlet air temperature, feed rate, matrix proprieties, and extract-to-matrix ratio also influence the powder's moisture content [9].

Moisture availability, known as a_w, is crucial for biochemical reactions and microbial growth [50], as the growth of molds, yeasts, and bacteria is expected at a_w above 0.6 [54]. In an environment with low a_w ($a_w < 0.3$), the growth of microorganisms and undesirable biochemical reactions is reduced, improving product quality and shelf life. A low a_w also suggests that the encapsulated powder is amorphous, as there is minimal free water, typical of crystallized powders. When the a_w exceeds 0.3, amorph sugars can begin to recrystallize, leading to physical changes such as stickiness and agglomeration [12,45].

Moisture content and a_w are crucial factors affecting the stability of powdered products. Even a tiny amount of water can lower the glass transition temperature, increasing the mobility of the food particles during storage and resulting in changes to the product, such as stickiness and caking [49,55]. Additionally, higher moisture content can lead to the formation of more liquid bridges, which in turn increases cohesion forces [56].

The high feed flow rate is another operational parameter that negatively affects the moisture content of the powder. This is because the reduced contact time between the atomized feed and the drying agent leads to inefficient heat transfer and a decreased water evaporation rate [47]. In this study, the feed flow rate was fixed at 0.5 L/h based on preliminary investigations, which was the same condition selected by Nguyen et al. [47] for ensuring an adequate moisture content.

3.2.3. Particle Morphology

The particle morphology of the powder obtained at various drying temperatures can be assessed using the images obtained by scanning electron microscopy (S.E.M.), as depicted in Figure 3. Studying particle morphology is significant because it directly influences manufacturing efficiency, product quality, and consumer satisfaction [57].

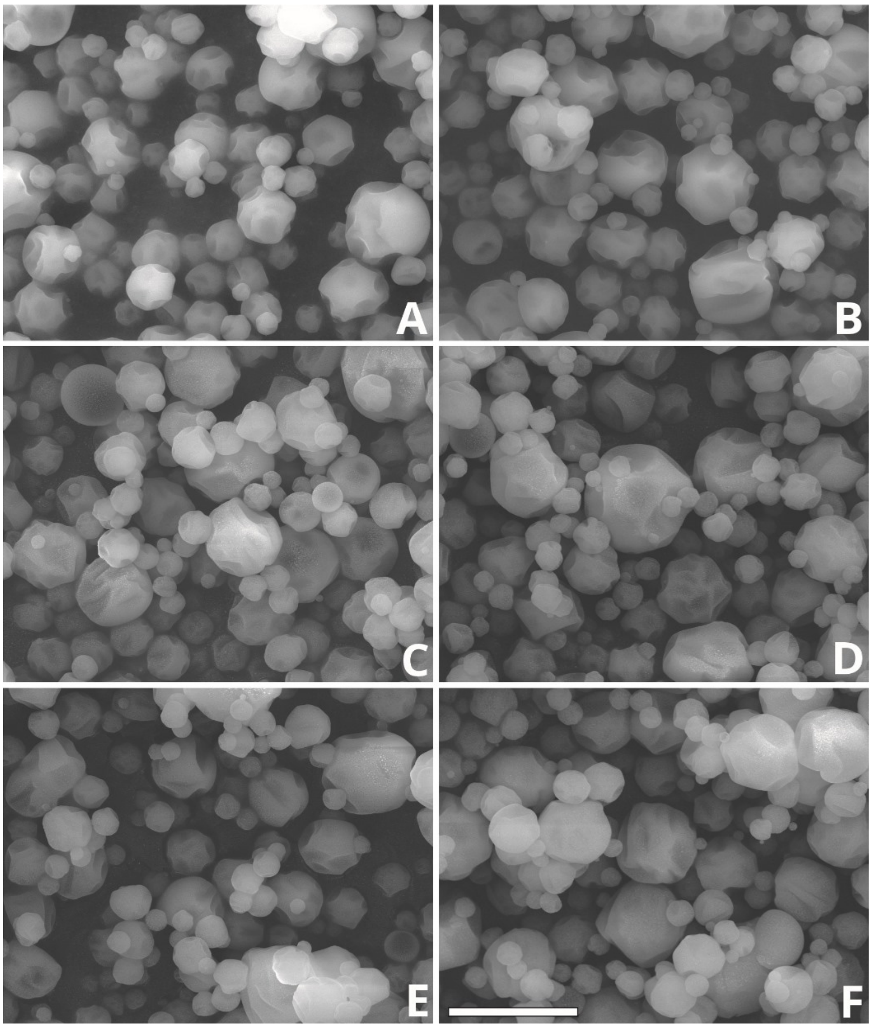

Figure 3. Scanning electron microscopy of anthocyanin powders spray dried at different temperatures. (**A**) 120 °C; (**B**) 130 °C; (**C**) 140 °C; (**D**) 150 °C; (**E**) 160 °C; (**F**) 170 °C. Bar: 10 µm.

All samples presented spherical particles varying in size, which is typical of materials produced by spray drying [31]. The particles presented a morphology with small coalescences and a curved and shriveled surface (Figure 3A–F). Powders dried at 120 and 130 °C presented a smoother surface (Figure 3A,B), while the others have small ornamentations in the form of dots on the surface (Figure 3C–F). The curved shape can be associated with particle shrinkage during drying and cooling [9]. Moreover, particles with very rough surfaces, as observed for those dried at 140 °C or higher temperatures, may present a higher anthocyanin degradation associated with forming a more porous structure [14]. Conversely, the highest anthocyanin recovery in this study was observed at 140 °C, with a subsequent decrease in pigment recovery.

Particle morphology depends on the drying rate and food composition. The drying rate is related to drying temperature and several other operational parameters. When the drying rate is too low, the surface remains moist and supple for a long time, and the hollow particle can deflate and shrivel as it cools [31]. Regarding the food composition, the low-molecular-weight sugar present in the food matrix can act as a plasticizer, reducing irregular shrinkage of the microparticle surface during the drying process [50]. The powder morphology found in this work is like several reports of spray-dried anthocyanin powders using maltodextrin as a carrier agent [9,31,58] and the combination of maltodextrin with other carrier agents [50].

Moreover, the particles' shape impacts the powder's density and flow. Irregular shapes cause particles to interlock, increasing flow resistance, while rounded particles may reduce interparticle forces [50]. The particle size distribution was similar in most samples (Figure 4). One hundred and fifty particles were measured, with the measurements ranging from ranging from 0.256 to 8.798 µm.

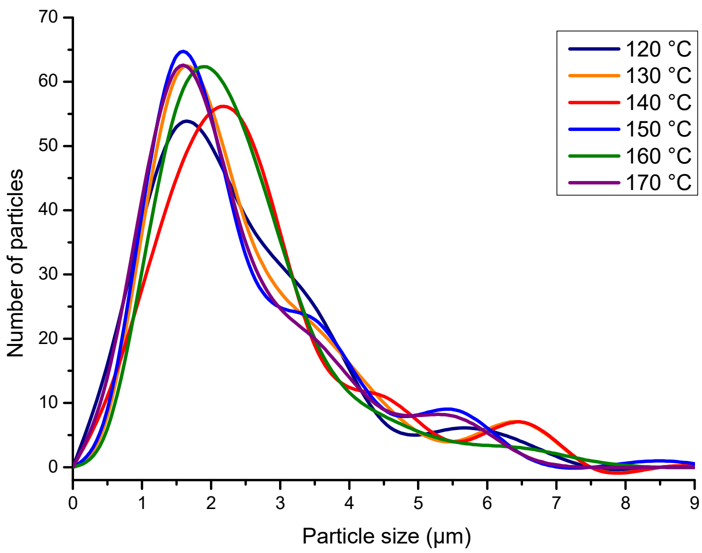

Figure 4. Particle size distribution of anthocyanin powders obtained by spray drying at different temperatures.

In addition to the nozzle diameter, the particle size of spray-dried food powders is affected by the processing conditions and the food composition. Thus, the particle size of spray-dried food powders is largely variable. Some studies showed increases in particle size with increases in inlet air temperature. Controversially, in this study, the mean particle size increased when the temperature was increased from 120 to 140 °C and decreased at higher temperatures.

When particle size is too small, the cohesive forces become more significant than the other interactions. Above a certain size threshold, more energy is needed to move the particles, and the particle shape becomes more influential in the flow characteristics of the powder [18,56]. This threshold depends on the product characteristic, the value of 50 µm reported by Lumay et al. [56], which is greater than the particle size found in this work. For this reason, cohesive forces are primarily responsible for the flow behavior of this anthocyanin powder.

Milinia et al. [9] encapsulated a roselle anthocyanin extract and observed mean particle sizes of 4.48–5.62 μm for powders formulated with maltodextrin. The sizes are slightly higher than the values found in this work. Rosa et al. [59] encapsulated anthocyanins from a blueberry extract using maltodextrin and hi-maize as carrier agents and obtained powders with particle sizes ranging from 10 to 21 μm. The authors also stated the importance of particle size evaluation before using the powder as a food ingredient since it may influence the food texture.

Moreover, particle size is inversely proportional to the powder's surface area, with increases in the surface area being related to higher degrees of surface sticking and interaction with neighboring particles [60]. The particle size is a key factor in the flow behavior of the powders and is influenced by equipment parameters such as the size of the nozzle, the flow rate, and the pressure, as well as feed solution properties like the type and concentration of the carrier agent [59]. These factors play a crucial role in determining the particle size and shape and, consequently, the flow behavior of the powder [60].

3.3. Powder Flowability

3.3.1. Bulk Density

Powders are particles with pores and empty spaces between them. The density of powder is usually expressed as bulk density [19], which is a key property related to food products' quality and economic aspects. High bulk density reduces shipping and packaging costs, while low bulk density is related to agglomeration, impacting powder flowability [48].

The bulk density of the anthocyanin powders ranged from 347 ± 52.3 to 367.8 ± 28.6 kg/m^3 (Figure 5). These results are like those found for black mulberry juice powders spray dried using maltodextrin [51]. The authors observed that the bulk density of powders prepared with different concentrations of carrier agents ranged from 0.34 to 0.40 g/mL (340–400 kg/m^3).

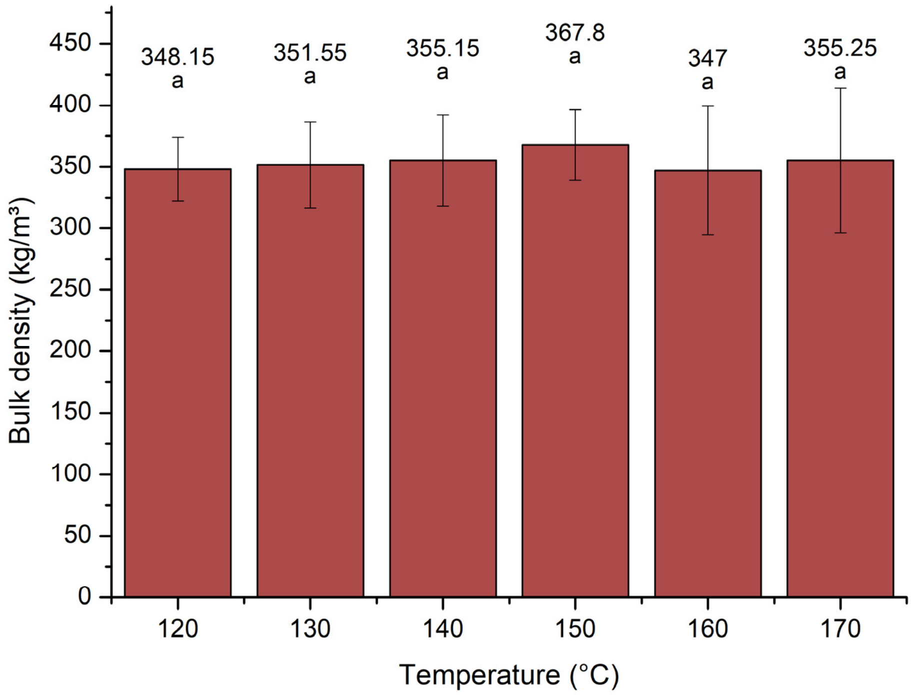

Figure 5. Bulk density of anthocyanin powders obtained by spray drying at different temperatures. Tukey's test was conducted to compare the means of samples dried at different temperatures. Means that do not share a letter are significantly different at a significance level of 10%.

In this work, no significant differences ($p > 0.10$) were observed between the bulk densities of powders dried at different temperatures. Similarly, Lima et al. [19] and Jafari et al. [34] also observed no impact of the drying temperature on the bulk density of soluble spray-dried sage powder. However, some researchers have reported a decrease in bulk density with increasing drying temperature [35,37]. This is due to faster evaporation rates at higher temperatures, which result in a more porous or fragmented structure and less shrinkage of the droplets. Consequently, larger particle sizes may be formed and the powder contains more entrapped air, thus reducing the bulk density [31,35].

3.3.2. Carr Index (CI), and Hausner Ratio (H.R.)

Several studies in the literature utilize the relationship between bulk and tapped density to evaluate the flowability of a powder, such as CI and H.R. These results rely on empirical relations to offer insights into the macroscopic properties of a powder and have become popular since they are simple and fast methods [18,60].

The values for CI and H.R. obtained for anthocyanin powders are presented in Table 3. Regardless of the temperature, all values obtained for CI and H.S. were greater than 38% and 1.60, respectively, which indicates excessively difficult flowability.

Table 3. Carr index and Hausner ratio of anthocyanin powders obtained by spray drying at different temperatures.

Inlet Air Temperature (°C)	Carr Index (%)	Hausner Ratio
120	44.99 ± 4.24 [a]	1.82 ± 0.25 [a]
130	43.58 ± 3.48 [a]	1.77 ± 0.21 [a]
140	45.25 ± 2.97 [a]	1.83 ± 0.18 [a]
150	43.91 ± 2.73 [a]	1.78 ± 0.17 [a]
160	42.29 ± 4.08 [a]	1.73 ± 0.23 [a]
170	48.07 ± 4.20 [a]	1.93 ± 0.24 [a]

Tukey's test was conducted to compare the means of samples dried at different temperatures. Means that do not share a letter are significantly different at a significance level of 10%.

Lima et al. [19] found that values of CI and H.R. were greater than 38% and 1.60 for spray-dried powders containing 20% maltodextrin. Ribeiro et al. [61] observed that maltodextrin concentrations equal to or greater than 30% were necessary for the flow to be classified as acceptable, evaluating the CI and H.R. values. The authors also observed a smaller particle diameter in powders containing a 15% maltodextrin concentration. Generally, larger particle diameters result in better powder flowability due to decreased surface area per unit mass and reduced cohesivity [62]. High values of CI indicate low flowability and high compressibility of the powder, while high values of H.R. represent powders with poor flowability [63].

The irregularly shaped surfaces and small particle diameters observed by MEV (Session 3.2.3) can make the powder flowability difficult. Irregularly shaped particles typically have poor flow properties due to increased friction between them. Additionally, small particle sizes can have a negative effect as they increase surface area, leading to stronger adhesive and cohesive forces with neighboring particles and surfaces. The greater the cohesive interactions between particles within a powder, the worse the flow [60]. It is important to note that these properties may change due to variations in handling and storage conditions, such as fluctuations in temperature and relative humidity [57].

3.3.3. Flow Index (I_F)

The unconfined sliding stress is the parameter that indicates the compressive strength of the powders and depends on the principal consolidation stress applied. A higher unconfined sliding stress value indicates harder powder flow [64].

Flow index (I_F) values were calculated from principal consolidation stress and unconfined sliding stress values, ranging from 1.528 (130 °C) to 1.682 (160 °C), as shown in

Table 4. I_F is a parameter used to predict the flow behavior of powdered products, which can vary from "no flow" to "free flow". The flow can be classified as no flow ($I_F < 1$), very cohesive ($1 < I_F < 2$), cohesive ($2 < I_F < 4$), easy ($4 < I_F < 10$), and free ($I_F > 10$) [64]. Therefore, all anthocyanin powders fall into the classification of very cohesive.

Table 4. Flow index of anthocyanin powders obtained by spray drying at different temperatures.

Inlet Air Temperature (°C)					
T = 120 °C		**T = 130 °C**		**T = 140 °C**	
δ_c	δ_1	δ_c	δ_1	δ_c	δ_1
1.674	1.338	1.360	1.351	1.350	1.261
3.014	2.635	2.936	2.529	2.808	2.291
5.819	4.044	5.746	4.249	5.608	3.907
11.714	7.511	11.654	7.575	11.279	6.992
23.596	12.781	22.964	13.528	22.853	11.877
I_F	1.618	I_F	1.528	I_F	1.667
Inlet Air Temperature (°C)					
T = 150 °C		**T = 160 °C**		**T = 170 °C**	
δ_c	δ_1	δ_c	δ_1	δ_c	δ_1
1.338	1.346	1.342	1.273	1.331	1.262
2.843	2.775	2.775	2.219	2.813	2.2
5.765	5.685	5.685	3.952	5.683	4.053
11.702	11.376	11.376	6.911	11.381	7.184
23.570	22.735	22.735	11.745	22.641	12.106
I_F	1.611	I_F	1.682	I_F	1.636

δ_c = principal consolidation stress (kPa); δ_1 = unconfined sliding stress (kPa); I_F = flow index.

Lima et al. [19] obtained an I_F of 2.06 for dried sapodilla pulp in a spray dryer using 20% (*w*/*v*) maltodextrin. Maciel et al. [65] obtained an I_F ranging from 3.25 to 4.01 when freeze drying *cupuaçu* pulp with different maltodextrin concentrations. They found that lower levels of maltodextrin resulted in lower I_F values. Their research used maltodextrin concentrations ranging from 13%. However, this lower concentration may have affected the flow properties of the powder.

The cohesive forces play a significant role in the interaction of particles due to their small sizes. This interaction is caused by capillary forces, electrostatic forces, liquid bridges, dipole–dipole interactions, and van der Waals interactions [56,60,66]. For particles of small diameters, such as the particles from this study, van der Walls is the main force influencing particle interactions and thus is responsible for powder cohesiveness [60]. Moreover, powders obtained from food matrices with low pH and high amounts of sugars tend to be more cohesive due to the influence of the pH on particle interaction. Rigolon et al. [67] demonstrated that hydrogen bonding occurs between maltodextrin and the anthocyanins, which is already expected given the amount of OH groups in the carrier agent and the anthocyanins. These interactions are enhanced in acidic environments due to the protonation of other molecules, such as anthocyanins. Moreover, this acidic environment favors anthocyanin stability [17,67].

At higher moisture contents, liquid bridges become more significant. Additionally, when the extract is exposed to temperatures above the glass transition temperature (Tg), the powder transitions from a glassy to a rubbery state, impacting its flow characteristics. Contamination can detrimentally affect powder flow, as highly sticky materials exhibit poor flow due to increased interaction forces [18,60]. These powders may form aggregates by the formation of liquid instead of solid bridges [68].

3.3.4. Wall Friction Angles

The wall friction angle indicates the adhesion between the powder and the hopper wall surface. The larger the angle, the more difficult the powder's movement on the wall is. This plays a critical role in determining how the powder flows during discharge [19,69]. A lower angle requires a less steep slope to properly ensure the powders' flow [70].

In this study, the highest and the lowest wall friction angles for each powder were evaluated, with values ranging from 33.4 (170 °C) to 37.4 (120 °C) for the highest wall friction angle and from 29.2 (140 °C) to 30.7 (120 °C) for the lowest wall friction angle. The inlet air temperature did not affect the wall friction angles (Figure 6). Moreover, the wall friction angles did not show a direct correlation with the flow function results, which may have occurred due to differences in the shape and size of the particles [71].

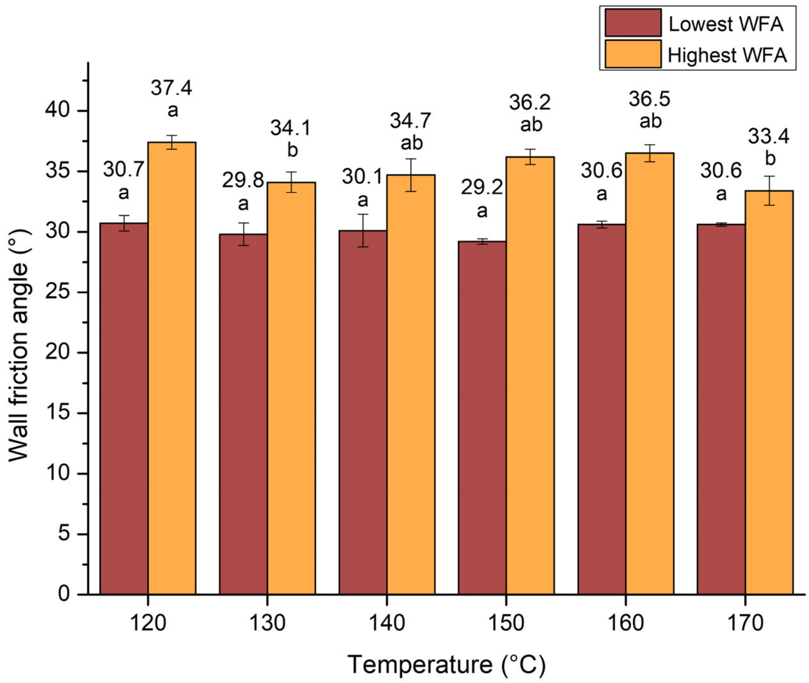

Figure 6. Wall friction angles of anthocyanin powders obtained by spray drying at different temperatures. W.F.A. = wall friction angle. Tukey's test was conducted to compare the means of samples dried at different temperatures. Means that do not share a letter are significantly different at a significance level of 10%.

4. Conclusions

This study conducted spray drying of anthocyanin extract from grape residue using maltodextrin (13% *w*/*v*) as a carrier agent. The inlet drying air at 140 °C resulted in the highest process yield (50.00 ± 3.06%) and the greatest retention of anthocyanins (91.94 ± 1.59%) among the temperatures studied.

Furthermore, the acidified aqueous extract proved to be an effective method for preparing extracts before drying, with a low pH significantly improving anthocyanin stability during high-temperature processing. Samples dried at 130 to 170 °C displayed appropriate values of moisture content and a_w (moisture content < 5% and a_w < 0.3), indicating microbiological powder stability.

Overall, the most favorable physicochemical properties were achieved by drying the anthocyanin extract at 140 °C, ensuring a higher content of bioactive compounds as well as low moisture content and a_w. It is noteworthy that all powders exhibited poor flowability, likely due to the low concentration of maltodextrin and the acidic media used to prepare the extract.

The research approach used resulted in a powder with a low carrier agent content and high retention of bioactive compounds. This helps to efficiently utilize grape waste by producing anthocyanin powder, a versatile functional ingredient. The powder's poor flowability is attributed to the small particles, low pH, and high sugar content of the grape extract. To improve flowability, the powder needs to undergo an agglomeration process to produce larger rounded or spherical granules that can reduce potential interlocking and improve powder flowability.

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Conflicts of Interest: The authors declare no conflicts of interest.

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