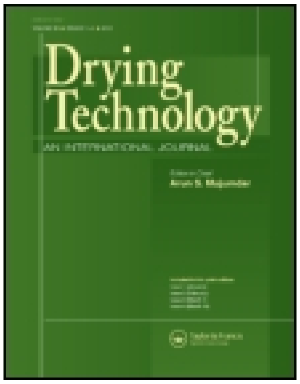

Drying Technology: An International Journal
Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/ldrt20

Effects of Spray-Drying Conditions on the Physicochemical Properties of Blackberry Powder

Cristhiane Caroline Ferrari [a] , Silvia Pimentel Marconi Germer [a] & José Maurício de Aguirre [a]
[a] Fruit and Vegetable Technology Centre (FRUTHOTEC), Institute of Food Technology, Campinas SP, Brazil
Published online: 07 Nov 2011.

Drying Technology, 30: 154–163, 2012

ISSN: 0737-3937 print/1532-2300 online
DOI: 10.1080/07373937.2011.628429

Effects of Spray-Drying Conditions on the Physicochemical Properties of Blackberry Powder

Cristhiane Caroline Ferrari, Silvia Pimentel Marconi Germer, and José Maurício de Aguirre
Fruit and Vegetable Technology Centre (FRUTHOTEC), Institute of Food Technology, Campinas SP, Brazil

The purpose of this work was to study the effects of spray-drying conditions on the physicochemical characteristics of blackberry powder using a central composite rotatable design. Inlet air temperature (140–180°C) and maltodextrin concentration (5–25%) were employed as independent variables. Moisture content, hygroscopicity, anthocyanin retention, color, powder morphology, and particle size were analyzed. A higher inlet air temperature significantly increased the hygroscopicity of the powder, decreased its moisture content, and led to the formation of larger particles with smooth surfaces. Powders produced with higher maltodextrin concentrations were less hygroscopic, slightly lighter and less red, and had a lower moisture content. Anthocyanin retention was mainly affected by drying temperature due to the heat sensitivity of the pigment. The optimal processing conditions were an inlet air temperature of 140–150°C and maltodextrin concentration of 5–7%. Overall, these results indicate that good quality powders can be obtained by spray drying, with potential applications for the food industry.

Keywords Anthocyanin; Atomization; Hygroscopicity; Maltodextrin; Powder morphology; *Rubus* spp

INTRODUCTION

The blackberry (*Rubus* spp.) is a small fruit that is native to Asia and cultivated in Europe and North America and in temperate regions of Brazil. Several phytochemicals found in the blackberry, such as phenolic compounds and anthocyanins, have been shown to have beneficial health properties in humans due to their high antioxidant activity. These functional benefits include protection against liver injury, reduction in blood pressure, strong anti-inflammatory and antimicrobial activities, and the suppression of human cancer cell proliferation.[1] Many studies have demonstrated the strong antioxidant capacity of blackberry fruits.[2–4] Although the blackberry has high potential in the food industry as a result of its sensory and nutritional qualities, it is highly perishable and has high respiration rates and a short postharvest life, which limits the in natura fruit market. Furthermore, the anthocyanins responsible for the attractive color of blackberries are generally susceptible to degradation reactions during processing and storage due to their sensitivity to adverse environmental conditions, such as high temperatures, light, and oxygen.[5] In this context, spray-drying represents an alternative to improve conservation of the final product and can result in a powder with a higher stability and longer shelf life, which facilitates the storage, handling, and transportation of the product. There are no available data in the literature concerning the production of blackberry powders using the spray-drying process.

Spray drying is a widely used technique that turns liquid food into a powder form. This method is commonly used in food and pharmaceutical manufacturing processes. Its short production time and use of lower temperatures make spray drying suitable for heat-sensitive products because it promotes a higher retention of flavor, color, and nutrients. The quality of the powders produced by spray drying depends on the characteristics of the feed solution (e.g., viscosity, flow rate) and the drying air (e.g., temperature, pressure, air flow), contact between the hot air and droplets in the drying chamber (cocurrent or countercurrent flow), and the type of atomizer used.[6]

According to Cano-Chauca et al.[7] the spray drying of sugar-rich foods, such as fruit juices, has great economic potential because the transformation of these products into a dry form results in reduced volume and a longer shelf life. However, fruit juice powders are highly hygroscopic and can easily absorb moisture from the surrounding air, resulting in stickiness and flow problems. The sticky behavior is attributed to the high concentration of low-molecular-weight sugars and organic acids, which have low glass transition temperatures and tend to stick to the chamber walls of the dryer and decrease the yield process. To overcome these problems, the addition of carrier agents such as maltodextrin is recommended during spray drying.

Correspondence: Cristhiane Caroline Ferrari, Fruit and Vegetable Technology Centre (FRUTHOTEC), Institute of Food Technology (ITAL), P.O. Box 139, CEP 13070-178, Campinas SP, Brazil; E-mail: cferrari@ital.sp.gov.br

The higher molecular weight of maltodextrin increases the glass transition temperature of the product, preventing stickiness and reducing powder hygroscopicity. Carrier agents also protect sensitive food components against unfavorable environmental conditions.[8]

The purpose of this study was to obtain blackberry juice powder by spray drying and to evaluate the effects of the inlet air temperature and maltodextrin concentration on the physicochemical characteristics (e.g., moisture content, hygroscopicity, anthocyanin retention, color parameters, particle size, and morphology) of the final product.

MATERIALS AND METHODS

Materials

Frozen blackberry pulp (De Marchi Ind. and Com. Ltd., Jundiaí, Brazil) was stored in a freezer chamber at −18°C and thawed in a refrigerator (4–5°C) for 18 h. Table 1 presents the physicochemical properties of the blackberry pulp. Maltodextrin (Maltogill 20, Cargill, Uberlândia, Brazil) with 20 dextrose equivalents (DE) was used as a carrier agent.

Sample Preparation and the Spray-Drying Process

Maltodextrin was added to the pulp before spray-drying and homogenized in a colloid mill (Meteor, São Paulo, Brazil) until complete dissolution.

The process was performed using a laboratory-scale spray dryer (model B290, Büchi, Flawil, Switzerland) at a drying rate of 1.0 kg water/h. The mixture was fed into the drying chamber at room temperature through a peristaltic pump with the flow rate adjusted to 0.49 kg/h. Spray drying was performed with a concurrent regime using a two-fluid nozzle atomizer (0.7 mm diameter), a drying air flow rate of 0.36 m^3/h, and an aspirator flow rate of 35 m^3/h (100% of its maximum capacity). These conditions were established in a previous work.[9] The tests were performed under different processing conditions according to the experimental design explained in the following section. The inlet air temperature varied between 140 and 180°C, and the concentration of the carrier agent ranged from 5 to 25 g maltodextrin/100 g fresh juice (w/w), which corresponds to 38.06 g maltodextrin/100 g total solids and 75.75 g maltodextrin/100 g total solids, respectively. The different powders produced were placed in hermetic containers and stored in desiccators containing silica gel until utilization.

TABLE 1
Physicochemical properties of blackberry pulp subjected to spray drying

Analysis	Mean value	Method
Moisture content (%)	91.96 ± 0.14	AOAC[10]
Ash (%)	0.20 ± 0.01	AOAC[10]
Protein	<0.1	AOAC[10]
Lipid	0.10 ± 0.01	AOAC[10]
Reducing sugar (g/100 g)	5.35 ± 0.26	AOAC[10]
Total sugar (g/100 g)	6.43 ± 0.13	AOAC[10]
Titratable acidity (% citric acid)	0.76 ± 0.01	AOAC[10]
Anthocyanins (mg/100 g)	77.90 ± 2.11	AOAC[10]
pH	3.31 ± 0.02	pH Meter
Total soluble solids (°Brix)	6.77 ± 0.12	Bench refractometer

AOAC = Association of Analytical Chemists.

Experimental Design

Response surface methodology was applied to optimize spray drying of the blackberry pulp. A 2^2 central composite rotatable design with four axial and three central points resulting in 11 trials was used to obtain a second-order model for the prediction of moisture content, anthocyanin retention, hygroscopicity, particle size, and color parameters (lightness and hue angle), which were considered the dependent variables. The two independent variables evaluated were inlet air temperature and maltodextrin concentration. Table 2 shows the experimental design, the coded and real values of these variables, and the responses evaluated. Experimental data were fitted to a second-order polynomial equation as follows:

$$y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_{12} x_{12} + \beta_{11} x_{11}^2 + \beta_{22} x_{22}^2 \qquad (1)$$

where y is the response (dependent variable); β_0, β_1, and β_2 are the regression coefficients for the linear terms; β_{11} and β_{22} are the quadratic terms; β_{12} is the interaction term; and x_1 and x_2 represent the coded values of the independent variables, inlet air temperature and maltodextrin concentration, respectively.

Results were statistically evaluated using analysis of variance (ANOVA) and the software Statistica 8.0 (StatSoft, Inc., Tulsa, OK) to obtain the regression coefficients. The effects of the process variables on the responses were analyzed with a confidence level above 90% ($p \leq 0.10$).

Analytical Methods

Moisture Content

The moisture content of the powder was determined gravimetrically. Samples were weighed and dried in a vacuum oven at 70°C for 24 h.[10]

Hygroscopicity

Hygroscopicity was evaluated based on the method described by Cai and Corke[11] with modifications. Samples

TABLE 2
Experimental design for spray drying of blackberry pulp, including the independent variables and the responses evaluated

Run	T_{in} (°C)	MC (%)	X (%)	H (g/100 g)	AR (%)	L^*	H^* (°)	D[4.3] (μm)	T_{out} (°C)
1	146 (−1)	8 (−1)	2.44	24.55	76.40	36.56	9.57	14.46	99
2	174 (+1)	8 (−1)	0.71	26.39	71.93	33.78	9.68	18.85	112
3	146 (−1)	22 (+1)	1.25	20.47	77.42	44.33	5.40	20.10	97
4	174 (+1)	22 (+1)	1.08	21.78	72.87	37.42	5.69	27.92	117
5	140 (−1.41)	15 (0)	1.94	22.11	73.42	37.82	7.99	12.52	109
6	180 (+1.41)	15 (0)	1.02	23.01	69.43	39.56	7.70	14.10	115
7	160 (0)	5 (−1.41)	1.98	27.32	80.76	34.74	10.91	34.18	101
8	160 (0)	25 (+1.41)	0.47	18.77	76.80	39.93	5.63	14.85	102
9	160 (0)	15 (0)	1.51	22.75	70.40	38.68	6.36	13.33	110
10	160 (0)	15 (0)	1.42	23.08	70.10	39.35	6.72	13.10	109
11	160 (0)	15 (0)	1.37	22.32	71.28	38.63	6.38	13.01	109

T_{in} = inlet air temperature; MC = maltodextrin concentration; X = moisture content; H = hygroscopicity; AR = anthocyanin retention; L^* = lightness; H^* = hue angle; D[4.3] = mean particle diameter; T_{out} = outlet air temperature.

(approximately 1 g) were placed in a container at 25°C with a saturated NaCl solution (75.29% relative humidity). Samples were weighed after one week, and hygroscopicity was expressed as grams of adsorbed moisture per 100 grams of dry matter.

Anthocyanins

Powder anthocyanin content was determined according to the spectrophotometric pH differential, which is based on the anthocyanin structural transformation that occurs with a change in pH (colored at pH 1.0 and colorless at pH 4.5).[10] Two dilutions of each sample were prepared with potassium chloride (0.025 M) and sodium acetate (0.4 M), which were used as buffer solutions at a pH of 1.0 and 4.5, respectively. Anthocyanins were extracted with an acetone solution (70%) according to the methodology described by Awika et al.[12] and Falcão et al.[13] with some modifications. Absorbance was measured in a spectrophotometer (model 700Plus, Femto, São Paulo, Brazil) at 520 and 700 nm. Total anthocyanin content was calculated using the molar extinction coefficient of 26,900 L/cm · mol mol for cyanidin-3-glucoside (cyd-3-glu), which is the predominant anthocyanin found in blackberry pulp.[14] Results were expressed as milligrams of cyd-3-glu per 100 grams of dry matter. Total anthocyanin content in the mixture fed into the spray dryer was also determined. Anthocyanin retention (AR) after spray drying was calculated according to Eq. (2):

$$A\,\mathrm{R} = \frac{\text{TAC in spray dried powder}}{\text{TAC in feed solution}} \times 100 \qquad (2)$$

where AR is anthocyanin retention (%) and TAC is the total anthocyanin content (mg/100 g dry matter).

Color

The color of the blackberry powder was measured using a colorimeter (model CR400, Konica Minolta, Osaka, Japan) with a CIELAB scale (L^*, a^*, and b^*), D65 as an illuminant, and a 10° observer angle as a reference system. The color measurements were expressed in terms of lightness L^* ($L^* = 0$ for black and $L^* = 100$ for white) and the chromaticity parameters a^* (green [−] to red [+]) and b^* (blue [−] to yellow [+]). The cylindrical coordinate H^* (hue angle) was determined from these parameters using the following equation (Eq. (3)). The measurements were made in triplicate.

$$H^* = \arctan\left(\frac{b^*}{a^*}\right) \qquad (3)$$

Particle Size

The particle size was determined using a laser light diffraction instrument (Mastersizer S, model MAM 5005, Malvern Instruments, Malvern, UK). A small amount of powder was dispersed in 99% isopropanol under magnetic agitation, and the distribution of particle size was monitored during five successive measurements. The particle size was expressed as D[4,3] (De Brouckere mean diameter), or the mean diameter over the volume distribution, which is generally used to characterize a particle.

Morphology

The microstructure of the particles was evaluated using scanning electron microscopy (SEM). Powders were attached to SEM stubs using a double-sided adhesive tape coated with gold/palladium under a vacuum in a sputter coater (model SC7620, VG Microtech, Ringmer, UK) at a coating rate of 0.51 Å/s, 3–5 mA, 1 V, and 0.08–0.09 mbar for 180 s. The coated samples were observed with a LEO440i scanning electron microscope (Leica Electron Microscopy Ltd., Oxford, England). The SEM was operated at 20 kV and 150 pA with a magnification of 1,000×.

RESULTS AND DISCUSSION

Experimental Design

Experimental data from the analysis of moisture content, hygroscopicity, anthocyanin retention, lightness, hue angle, and particle size are shown in Table 2. Estimated regression coefficients for the coded second-order polynomial model, F values, and coefficients of determination (R^2) are presented in Table 3. The models were tested for adequacy and fitness using ANOVA without considering the nonsignificant terms ($p > 0.01$). If the F_c value was higher than the F_t value the model was considered predictive. Although all of the responses evaluated showed higher F_c values compared to F_t values, the coefficients of determination for lightness and particle size were not satisfactory (0.56 and 0.43, respectively). In these cases, it was not possible to explain the results using the models or to generate the response surfaces.

Moisture Content

All of the powders produced showed moisture contents lower than 3% (Table 2), which is in agreement with the results published by Tonon et al.[15] and Quek et al.[16] for spray-dried açai and watermelon powders, respectively. Moisture content is the major factor that affects powder stability. Because water acts as a plasticizer, only a small amount is required to depress the glass transition temperature, which increases the food matrix mobility during storage and causes changes in the powder products, such as stickiness and caking.[17,18]

According to Table 3, the linear parameters of the inlet air temperature, maltodextrin concentration, and the interaction term were significant at $p \le 0.01$ for the response moisture content. Inlet air temperature had a negative effect on moisture content (Fig. 1). An increase in the temperature gradient between the atomized feed and the drying air led to an increase in heat transfer into the particles, providing a higher driving force for moisture evaporation and, consequently, powder products with a reduced moisture content. Similar behaviors have been reported for cactus pear,[19] acerola,[17] and orange juice[18] produced by spray drying.

A higher maltodextrin concentration led to the reduction of moisture content in the powders, as shown in Fig. 1. The addition of maltodextrin to the feed solution prior to spray drying increased the total solids content and reduced the total amount of water available for evaporation. Abadio et al.[20] Grabowski et al.[21] and Kha et al.[22] observed a decrease in moisture content with increasing maltodextrin concentration in studies evaluating the spray drying of pineapple juice, sweet potato puree, and Gac fruit, respectively. In a similar study with bayberry juice, Gong et al.[23] reported that the concentration of the carrier agent has an important effect on moisture content. A higher maltodextrin concentration (17%) improved the drying efficiency, whereas a lower maltodextrin concentration (7%) was not sufficient to dry the juice and resulted in a powder with a higher moisture content (around 8–10%).

Hygroscopicity

Hygroscopicity values ranged from 18.77 to 27.32 g/100 g dry matter (Table 2). Only the linear terms inlet air temperature and maltodextrin concentration showed statistical significance at $p \le 0.01$ (Table 3). Maltodextrin concentration was the independent variable that most

TABLE 3
Coded second-order regression coefficients, F values, and coefficients of determination (R^2) for the responses evaluated during spray drying of blackberry pulp

Coefficients	X (%)	H (g/100 g)	AR (%)	L^*	H^* (°)	D[4.3] (μm)
β_0	1.38	23.01	70.88	38.25	6.49	13.56
β_1	−0.40	0.62	−1.83	Ns	Ns	Ns
β_2	−0.37	−2.67	Ns	2.34	−1.95	Ns
β_{11}	Ns	Ns	Ns	Ns	0.56	Ns
β_{22}	Ns	Ns	3.89	Ns	0.77	5.91
β_{12}	0.39	Ns	Ns	Ns	Ns	Ns
R^2	0.89	0.94	0.91	0.56	0.98	0.43
F_c	19.98	63.66	40.50	11.25	118.95	6.76
F_t	4.35	4.46	4.46	5.59	4.35	5.59

X = moisture content; H = hygroscopicity; AR = anthocyanin retention; L^* = lightness; H^* = hue angle; D[4.3] = mean particle diameter; F_c = calculated F values; F_t = tabulated F values ($p \le 0.01$); Ns = nonsignificant ($p > 0.01$).

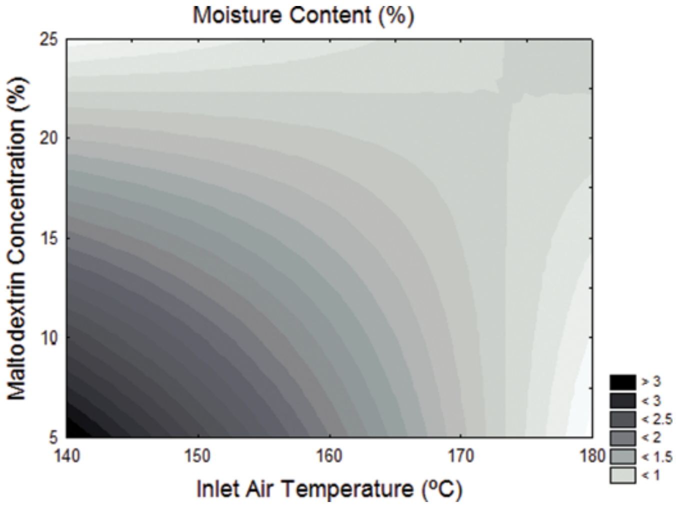

FIG. 1. Contour plot for the moisture content of blackberry powder produced by spray drying as a function of inlet air temperature and maltodextrin concentration (color figure available online).

influenced hygroscopicity values (Fig. 2). As the maltodextrin concentration increased, powder hygroscopicity decreased due to the low hygroscopicity of maltodextrin, confirming its efficiency as a carrier agent during spray drying. Quek et al.[16] reported that when the maltodextrin concentration is too high, the resulting powder has a lower quality because the nutrients present in the fruit are diluted.

The inlet air temperature positively affected hygroscopicity. The highest hygroscopicity values for blackberry powder were obtained with increased drying temperatures. Hygroscopicity values were inversely increased with moisture content such that a lower powder moisture content

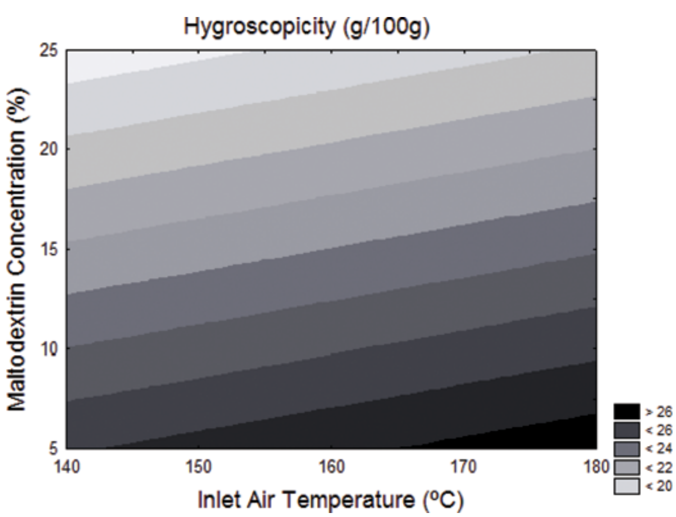

FIG. 2. Contour plot for hygroscopicity of blackberry powder produced by spray drying as a function of inlet air temperature and maltodextrin concentration (color figure available online).

indicated higher hygroscopicity. This observation was also made by Tonon et al.[15] According to the authors, powders containing a lower moisture content have a greater capacity to absorb ambient moisture, which is related to the higher water concentration gradient between the product and the surrounding air.

In spray drying cactus pear juice, Rodrígues-Hernández et al.[19] observed that the powders produced with a higher maltodextrin concentration and at a lower inlet air temperature were the least hygroscopic. Goula and Adamopoulos[18] described the effect of process variables on powder hygroscopicity as being dependent on their effect on the glass transition temperature. Increasing the inlet air temperature and maltodextrin concentration during spray drying of orange juice concentrate resulted in a higher powder glass transition temperature and lower hygroscopicity values, which contributed to an increase in the stability of the powder during storage.

Anthocyanin Retention

The linear term of inlet air temperature and the quadratic term of maltodextrin concentration were statistically significant at $p \leq 0.01$ in the retention of anthocyanin (Table 3), which varied between approximately 69 and 80% (Table 2). These values were remarkably lower compared to the results published by Fang and Bhandari[24] for spray-dried bayberry powder (about 94%), which was mainly due to the relatively low outlet air temperature (80°C) used in that study. According to Masters,[6] the temperature of the spray droplets is approximately 40°C (the wet bulb temperature) when the outlet air temperature is 80°C. Consequently, the powder temperature does not reach more than 55°C. In a similar work, Tonon et al.[15] observed that the anthocyanin retention for açai powder produced by spray drying varied from 77 to 86% when the outlet air temperatures ranged from 82 to 114°C, depending on process conditions. In the present study, a higher outlet air temperature (ranging from 97 to 117°C) likely caused a reduction in the anthocyanin content of the powder, suggesting that drying temperatures are very important for the spray drying of thermolabile biological materials.

In studying the microencapsulation of anthocyanin pigments extracted from *Garcinia indica* Choisy, Nayak and Rastogi[25] verified that the pigment retention varied between 65 and 79% after spray drying was performed using 5% maltodextrin with different dextrose equivalents (6, 19, 21, and 33 DE) and inlet and outlet air temperatures of 150 and 100°C, respectively. The authors attributed this behavior to the different chemical structures of maltodextrin because maltodextrin with a lower DE contains a large proportion of long-chain saccharides, which might result in surface cracking and a reduced barrier to oxygen. Maltodextrin with a higher DE could form denser and more oxygen-impermeable wall systems to provide better retention of the anthocyanin pigments.

Higher inlet air temperatures promoted greater losses of anthocyanin (Fig. 3), indicating that these pigments are very heat sensitive. A similar observation was reported by Ersus and Yurdagel[26] during the microencapsulation of anthocyanins extracted from black carrots using three different inlet air temperatures (160, 180, and 200°C). Cai and Corke[11] stated that, despite a higher drying rate and productivity, increased drying temperatures (above 180°C) were not suitable for spray drying of *Amaranthus* bethacyanin pigments because these compounds are very thermosensitive. Quek et al.[16] observed that spray-dried powders produced at lower inlet air temperatures have a tendency to agglomerate due to higher moisture contents. The agglomeration process decreases exposure of the powder to oxygen and protects anthocyanin pigments from oxidation.

The lowest anthocyanin retention (around 70%) using a concentration of 15% maltodextrin (runs 9–11) is related to the smaller particle size that was verified in these conditions (around 13 μm), as seen in Table 2. According to Rodrigués-Hernández et al.,[19] the increase in the particle surface area due to decreased particle size accelerated the degradation of vitamin C in cactus pear juice produced by spray drying. However, the effect of particle size was not verified in experimental run 5 (Table 2). In that run, the particles produced at 140°C and 15% maltodextrin showed better protection of anthocyanins (around 73%) compared to the treatments performed using 160°C (runs 9–11). In this case, the use of a lower inlet air temperature probably prevailed over the powder particle size and contributed to higher retention of anthocyanin.

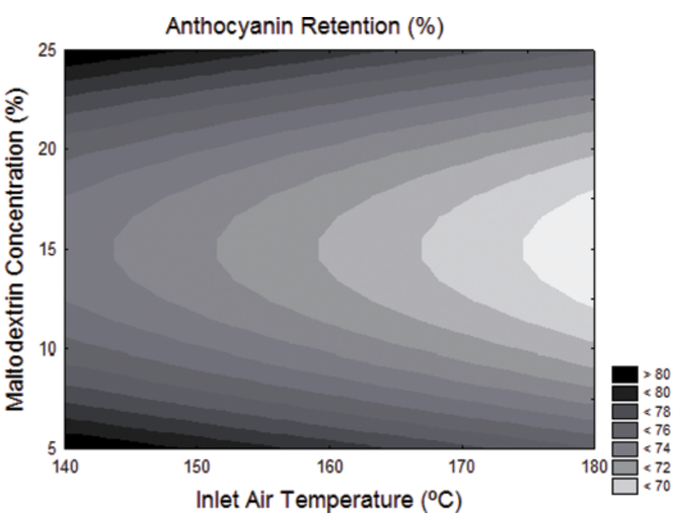

FIG. 3. Contour plot for the anthocyanin retention of blackberry powder produced by spray drying as a function of inlet air temperature and maltodextrin concentration (color figure available online).

Higher anthocyanin retention was observed at the lower and upper concentrations of maltodextrin (5 or 25%), according to the experimental design (Fig. 3). However, addition of a higher maltodextrin concentration to the feed mixture before spray drying may result in dilution of the pigments and could affect powder quality.

Color Parameters: Lightness and Hue Angle

The second-order polynomial model obtained for lightness was not suitable for the prediction of this color parameter due to a low coefficient of determination ($R^2 = 0.56$), as shown in Table 3.

According to Table 2, powder lightness increased with increased maltodextrin concentration due to the dilution effect caused by the addition of maltodextrin to the blackberry pulp, resulting in loss of color. Similar results have also been observed in purple sweet potato[21,27] and pineapple[20] produced by spray drying. Kha et al.[22] reported that when the maltodextrin concentration increased from 10 to 30%, the total carotenoid content of spray-dried Gac fruit was reduced from 1.95 to 0.61 mg/g dry matter. Higher lightness values were obtained at higher carrier agent concentrations. Therefore, a lower maltodextrin concentration should be used in situations where the powder should have a color similar to the fruit pulp.

With respect to the influence of drying temperature, lightness values were lower at a higher inlet air temperature when comparing treatments 1 and 3 to treatments 2 and 4 (Table 2). These results are consistent with those of Quek et al.[16] for spray drying of watermelon juice. The authors observed a decrease in L^* values when the inlet air temperature increased from 145 to 175°C due to the high sugar content present in watermelon, which contributed to browning of the powder. In a similar study with fermented mixed carrot and watermelon juices produced by spray drying, Mestry et al.[28] reported a decrease in red and orange color as the inlet air temperature increased from 120 to 160°C. In Ahmed et al.[27] color changes verified in spray-dried purple sweet potato were related to degradation reactions that promoted the formation of polymeric anthocyanins, which have a more brownish shade, and resulted in darker powders (lower lightness values).

The hue angle varied from 5.40 to 10.91 (Table 2), which is associated with the pink color characteristic of the blackberry flesh. These values indicate that the color parameters of blackberry powders are located in the first quadrant of the CIELAB color diagram ($+a^*$ and $+b^*$), corresponding to the region of red and yellow color, where 0° is pure red and 90° is yellow.

The linear and quadratic terms of maltodextrin concentration and the quadratic term of inlet air temperature were statistically significant at $p \leq 0.01$ (Table 3). Maltodextrin concentration was the independent variable that most affected the hue angle (Fig. 4). As the maltodextrin

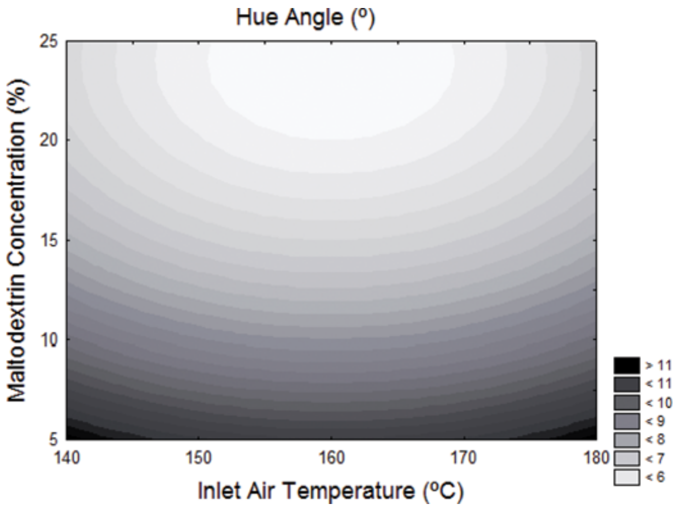

FIG. 4. Contour plot for the hue angle of blackberry powder produced by spray drying as a function of inlet air temperature and maltodextrin concentration (color figure available online).

concentration increased, the hue angle values decreased and enhanced the purple color of the blackberry powders. Ahmed et al.[27] also observed the reduction of hue angle values as the maltodextrin concentration increased from 3 to 10% for spray drying of purple sweet potatoes.

Inlet air temperature did not show a significant effect on hue angle in this study, as shown in Fig. 4. Quek et al.[16] and Kha et al.[22] reported that higher drying air temperatures provided an increase in hue angle values for spray-dried watermelon and Gac fruit powders, respectively. This behavior was attributed to the thermal degradation of lycopene and beta-carotene, causing a loss of redness and an increase in yellowness, which resulted in higher H^* values.

In general, the color of the blackberry powder will become whiter and less red as the maltodextrin concentration increases, which must be taken into consideration depending on the application of the final product.

Particle Size

Low coefficients of determination were obtained for the response particle size ($R^2 = 0.43$), indicating that the variation was explained by the residue and not by the regression (Table 3). Thus, it was not possible to evaluate these results through the second-order polynomial model and response surfaces.

Table 2 shows that the mean particle diameter of all powder samples ranged from 12.52 to 34.18 μm, indicating that spray drying promoted the formation of small particles. Obón et al.[29] obtained an average particle size of 10–12 μm for cactus pear juice produced by spray drying, whereas the mean diameter of spray-dried açai powder varied between 13 and 21 μm.[15]

Higher inlet air temperatures resulted in larger particles, which can be related to increased swelling as the drying temperature increased. When a particle is subjected to higher drying rates, the evaporation of moisture is rapid and promotes the formation of a hard crust that does not allow particle shrinkage during spray drying. However, if the inlet air temperature is lower, the particle remains moist for a longer period of time and shrinks, thus decreasing its size.[30] Tonon et al.[15] and Kurozawa et al.[31] reported similar behavior in spray-dried açai and chicken meat powder, respectively.

The maltodextrin concentration also affected the mean particle diameter. Increasing the carrier agent concentration from 8 to 22% or from 15 to 25% led to an increase in the particle size of the powder (Table 2). According to Goula and Adamopoulos,[32] the size of the spray-dried particles depends on the size of the atomized droplets, which is influenced by the atomizer type, physical properties of the feed solution, and concentration of the feed solids. Larger droplets are formed during atomization when the viscosity of the feed solution is increased, resulting in larger particles. This is in agreement with the results obtained for açai, cactus pear juice, and chicken meat hydrolysate powders produced by spray drying.[15,29,31]

A higher mean particle diameter and better protection of anthocyanins was observed when using 5% maltodextrin concentration and an inlet air temperature of 160°C (Table 2). It is possible that higher hygroscopicity values contributed to initiation of the agglomeration process, leading to the formation of larger particles. Rodríguez-Hernández et al.[19] reported that vitamin C retention in cactus pear juice obtained by spray drying was also associated with the particle size of the powder. The particle surface area increases as the particle size decreases, which enhances oxidation reactions and reduces the vitamin C content of the powder. According to Kurozawa et al.[31] as the particle size decreases, the solubility and flowability of the spray-dried powder also decrease, which directly influences its handling.

Figure 5 shows the particle size distribution of the spray-dried blackberry powders produced using 15% maltodextrin and different inlet air temperatures. The particle size of the blackberry powders ranged from 0.1 to 103.5 μm, and a bimodal distribution was observed, indicating two predominant sizes: one smaller size peak with a lower volume (<2%) and smaller particle diameters (predominant sizes of 1.0–1.2 μm) than the major peak. The major peak had a larger volume (about 7–9%) and a larger particle size and showed predominant sizes around 14–16 μm. This type of distribution was verified for all of the conditions studied. According to Tonon et al.,[15] a bimodal distribution is important for powder products because the smaller particles can penetrate into the spaces between the larger ones and occupy less space. The

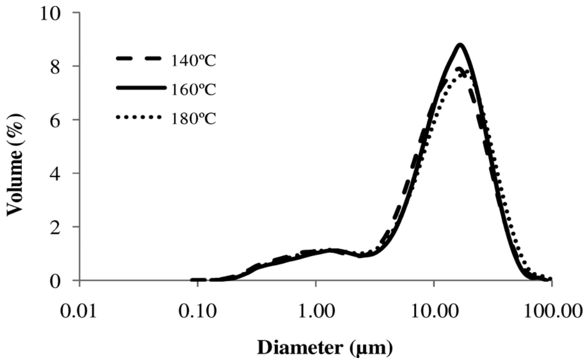

FIG. 5. Particle size distribution of spray-dried blackberry powders produced using 15% maltodextrin and different inlet air temperatures.

presence of larger particles may be attributed to the initiation of the agglomeration process.

Morphology

The morphological characteristics of spray-dried blackberry powders are shown in Figs. 6 and 7. A large number of irregular particles with spherical shapes were observed, which is characteristic of powders that are produced by

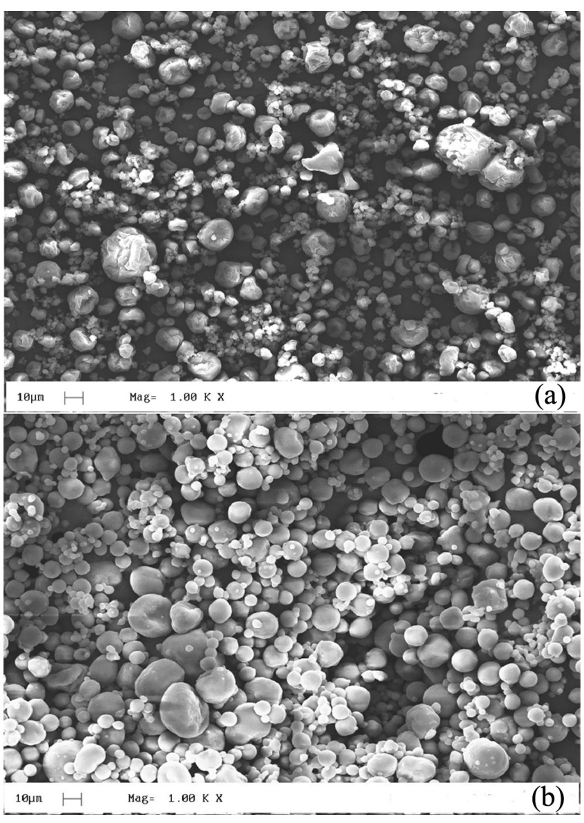

FIG. 6. Micrographs of spray-dried blackberry powder produced using 5% maltodextrin and different inlet air temperatures: (a) 140°C and (b) 180°C. Magnification = 1,000×.

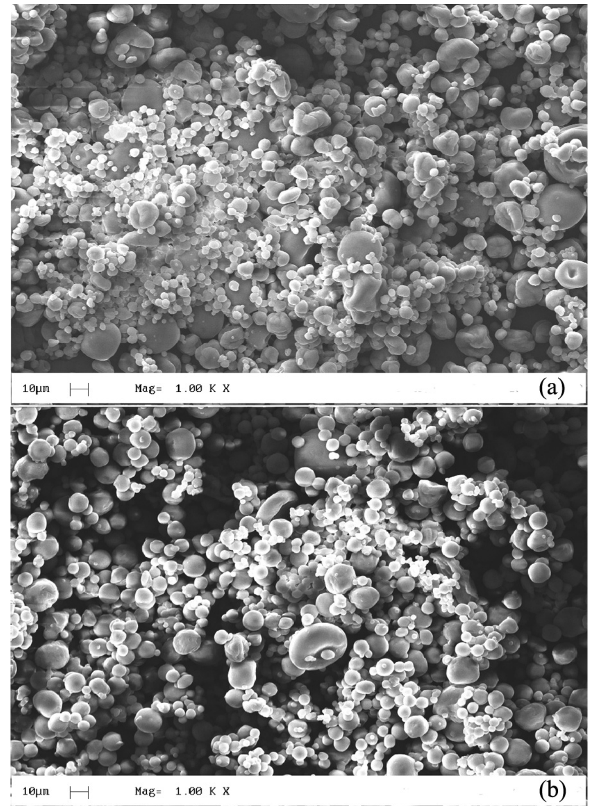

FIG. 7. Micrographs of spray-dried blackberry powder produced using an inlet air temperature of 160°C and different maltodextrin concentrations: (a) 5% and (b) 25%. Magnification = 1,000×.

spray drying. The use of a low inlet air temperature (140°C) promoted the production of particles with a shriveled surface (Fig. 6a), whereas spray-dried particles obtained at higher drying temperature (180°C) showed smoother surfaces (Fig. 6b). Similar behaviors were verified for cactus pear,[33] açai,[15] and milk[30] products produced by spray drying. Kurozawa et al.[31] reported that shrinkage of the spray-dried particles is related to differences in the drying rate, which is lower when the spray-drying process is performed with a lower inlet air temperature. In this case, water diffusion is slower, allowing more time for structures to deform, shrink, and collapse. The authors verified that spray-dried chicken meat powder obtained at 120°C presented with some dents, which had an adverse effect on the flow properties of the powders, whereas the particles produced using 180°C were smoother and more regular. According to Ré,[34] surface imperfections, such as wrinkles, cracks, or collapses, occur when a film slowly forms during the drying of atomized droplets. At higher drying temperatures, particles tend to inflate and form a crust, which is related to rapid water evaporation and the high pressure generated inside the particles.[35] Mestry et al.[28] stated that the differences in morphology and particle size of spray-dried powders occur as a result of the drying temperatures and atomization pressure. The authors obtained smaller and irregularly shaped particles at lower inlet air temperatures when spray drying carrot and watermelon juices, but they observed more regular and spherical particles at higher inlet air temperatures. An increase in atomization pressure resulted in finer droplets in the drying chamber, which led to the formation of smaller particles with lower residual moisture contents.

All of the spray-dried particles produced with different maltodextrin concentrations were similar to the particles shown in Fig. 7b. However, a lower maltodextrin concentration (5%) and an inlet air temperature of 160°C resulted in the strong adherence of smaller particles to the surface of larger ones to form many agglomerates (Fig. 7a), which increased the particle size.

To determine optimal processing conditions, the desired product characteristics were established as follows: lower hygroscopicity and moisture content, higher anthocyanin retention, and better preservation of powder color. Moisture content was not a critical parameter because all of the powders produced had low moisture content (<3%). Higher inlet air temperatures led to an increase in powder hygroscopicity and greater anthocyanin losses, whereas a higher maltodextrin concentration resulted in powders that were whiter and less red. Optimal spray-drying conditions were an inlet air temperature of 140–150°C and a maltodextrin concentration of 5–7%.

CONCLUSIONS

The concentration of the carrier agent had a significant effect on all of the responses evaluated. Increasing maltodextrin concentration caused a reduction in moisture content and powder hygroscopicity, whereas the spray-dried powders produced using higher inlet air temperatures showed lower moisture contents and were more hygroscopic. Higher anthocyanin losses were verified as the drying temperature increased due to the thermal degradation of these pigments. The color of the blackberry powder was mainly affected by maltodextrin concentration, which led to the formation of powders that were whiter and less red as the concentration of maltodextrin increased. With respect to powder morphology, higher inlet air temperatures resulted in larger particles with smooth surfaces, whereas particles produced with lower maltodextrin concentrations were smaller. The best processing conditions for the preservation of powder anthocyanin content and color characteristics were an inlet air temperature of 140–150°C and a maltodextrin concentration of 5–7%. In general, the results obtained in this study indicate that good quality powders with high anthocyanin content can be produced by spray drying, which demonstrates the great potential for use of such powders in dry mixes, beverages, desserts, and other food and nutraceutical products.

ACKNOWLEDGMENT

The authors thank FAPESP (process number 2010/02561-1) for financial support.

REFERENCES

1. Konczak, I.; Zhang, W. Anthocyanins—More than nature's colours. *Journal of Biomedicine and Biotechnology* **2004**, *5*(1), 239–240.
2. Gancel, A.L.; Feneuil, A.; Acosta, O.; Pérez, A.M.; Vaillant, F. Impact of industrial processing and storage on major polyphenols and the antioxidant capacity of tropical highland blackberry (*Rubus adenotrichus*). *Food Research International* **2011**, *44*(7), 2243–2251.
3. Wu, R.; Frei, B.; Kennedy, J.A.; Zhao, Y. Effects of refrigerated storage and processing technologies on the bioactive compounds and antioxidant capacities of 'Marion' and 'Evergreen' blackberries. *LWT – Food Science and Technology* **2010**, *43*(8), 1253–1264.
4. Acosta-Montoya, O.; Vaillant, F.; Cozzano, S.; Mertz, C.; Pérez, A.M. Phenolic content and antioxidant capacity of tropical highland blackberry (*Rubus adenotrichus* Schltdl.) during three edible maturity stages. *Food Chemistry* **2010**, *119*(4), 1497–1501.
5. Antunes, L.E.C. Blackberry: A new crop option to Brazil. *Ciência Rural* **2002**, *32*(1), 151–158 (in Portuguese).
6. Masters, K. *Spray Drying Handbook*, 5th ed.; Longman Scientific and Technical: London, 1991.
7. Cano-Chauca, M.; Stringheta, P.C.; Ramos, A.M.; Cal-Vidal, J. Effect of the carriers on the microstructure of mango powder obtained by spray drying and its functional characterization. *Innovative Food Science and Emerging Technologies* **2005**, *6*(4), 420–428.
8. Bhandari, B.R.; Data, N.; Howes, T. Problems associated with spray drying of sugar-rich foods. *Drying Technology* **1997**, *15*(2), 671–684.
9. Ferrari, C.C.; Ribeiro, C.P.; Liserre, A.M.; Moreno, I.; Germer, S.P.M.G.; Aguirre, J.M. Spray drying of blackberry juice using maltodextrin or gum arabic as carrier agents. In *Proceedings of the 6th Section VI International Symposium—Towards a Sustainable Food Chain: Food Process, Bioprocessing and Food Quality Management*, Nantes, France, April 18–20, 2011.
10. Association of Official Analytical Chemists. *Official Methods of Analysis of the Association of Official Analytical Chemists,* 18th ed.; AOAC Press: Gaithersburg, MD, 2006.
11. Cai, Y.Z.; Corke, H. Production and properties of spray-dried *Amaranthus* betacyanin pigments. *Journal of Food Science* **2000**, *65*(6), 1248–1252.
12. Awika, J.M.; Rooney, L.W.; Waniska, R.D. Anthocyanins from black sorghum and their antioxidant properties. *Food Chemistry* **2004**, *90*(1–2), 293–301.
13. Falcão, A.P.; Chaves, E.S.; Kuskoski, E.M.; Fett, R.; Falcão, L.D.; Bordignon-Luiz, M.T. Total polyphenol index, total anthocyanins and antioxidant activity of a model system of grape jelly. *Ciência e Tecnologia de Alimentos* **2007**, *27*(3), 637–642 (in Portuguese).
14. Koca, I.; Karadeniz, B. Antioxidant properties of blackberry and blueberry fruits grown in the Black Sea Region of Turkey. *Scientia Horticulturae* **2009**, *121*(4), 447–450.
15. Tonon, R.V.; Brabet, C.; Hubinger, M.D. Influence of process conditions on the physicochemical properties of açai (*Euterpe oleraceae* Mart.) powder produced by spray drying. *Journal of Food Engineering* **2008**, *88*(3), 411–418.
16. Quek, S.Y.; Chok, N.K.; Swedlund, P. The physicochemical properties of spray-dried watermelon powders. *Chemical Engineering and Processing* **2007**, *46*(5), 386–392.
17. Moreira, G.E.G.; Costa, M.G.M.; Souza, A.C.R.; Brito, E.S.; Medeiros, M.F.D.; Azeredo, H.M.C. Physical properties of spray dried acerola pomace extract as affected by temperature and drying aids. *LWT – Food Science and Technology* **2009**, *42*(2), 641–645.

18. Goula, A.M.; Adamopoulos, K.G. A new technique for spray drying orange juice concentrate. *Innovative Food Science and Emerging Technologies* **2010**, *11*(2), 342–351.
19. Rodríguez-Hernández, G.R.; González-García, R.; Grajales-Lagunes, A.; Ruiz-Cabrera, M.A.; Abud-Archila, M. Spray-drying of cactus pear juice (*Opuntia streptacantha*): Effect on the physicochemical properties of powder and reconstituted product. *Drying Technology* **2005**, *23*(4), 955–973.
20. Abadio, F.D.B.; Domingues, A.M.; Borges, S.V.; Oliveira, V.M. Physical properties of powdered pineapple (*Ananás comosus*) juice—Effect of malt dextrin concentration and atomization speed. *Journal of Food Engineering* **2004**, *64*(3), 285–287.
21. Grabowski, J.; Truong, V.D.; Daubert, C.R. Spray-drying of amylase hydrolyzed sweetpotato puree and physicochemical properties of powder. *Journal of Food Science* **2010**, *71*(5), 209–217.
22. Kha, T.C.; Nguyen, M.H.; Roach, P.D. Effects of spray drying conditions on the physicochemical and antioxidant properties of the Gac (*Momordica cochinchinensis*) fruit aril powder. *Journal of Food Engineering* **2010**, *98*(3), 385–392.
23. Gong, Z.; Zhang, M.; Mujumbar, A.S.; Sun, J. Spray drying and agglomeration of instant bayberry powder. *Drying Technology* **2007**, *26*(1), 116–121.
24. Fang, Z.; Bhandari, B. Effect of spray drying and storage on the stability of bayberry polyphenols. *Food Chemistry* **2011**, *129*(3), 1139–1147.
25. Nayak, C.A.; Rastogi, N.K. Effect of selected additives on microencapsulation of anthocyanin by spray drying. *Drying Technology* **2010**, *28*(12), 1396–1404.
26. Ersus, S.; Yurdagel, U. Microencapsulation of anthocyanin pigments of black carrot (*Daucuscarota* L.) by spray dryer. *Journal of Food Engineering* **2007**, *80*(3), 805–812.
27. Ahmed, M.; Akter, M.S.; Lee, J.C.; Eun, J.B. Encapsulation by spray drying of bioactive components, physicochemical and morphological properties from purple sweet potato. *LWT – Food Science and Technology* **2010**, *43*(9), 1307–1312.
28. Mestry, A.P.; Mujumdar, A.S.; Thorat, B.N. Optimization of spray drying as an innovative functional food: Fermented mixed juice of carrot and watermelon. *Drying Technology* **2011**, *29*(10), 1121–1131.
29. Obón, J.M.; Castellar, M.R.; Alacid, M.; Fernández-López, J.A. Production of a red–purple food colorant from *Opuntia stricta* fruits by spray drying and its application in food model systems. *Journal of Food Engineering* **2009**, *90*(4), 471–479.
30. Nijdam, J.J.; Langrish, T.A.G. The effect of surface composition on the functional properties of milk powders. *Journal of Food Engineering* **2006**, *77*(4), 919–925.
31. Kurozawa, L.E.; Morassi, A.G.; Vanzo, A.A.; Park, K.J.; Hubinger, M.D. Influence of spray drying conditions on physicochemical properties of chicken meat powder. *Drying Technology* **2009**, *27*(11), 1248–1257.
32. Goula, A.M.; Adamopoulos, K.G. Spray drying of tomato pulp: Effect of feed concentration. *Drying Technology* **2004**, *22*(10), 2309–2330.
33. Gandía-Herrero, F.; Jiménez-Atiénzar, J.; Cabanes, J.; García-Carmona, F.; Escribano, J. Stabilization of the bioactive pigment of *Opuntia* fruits through maltodextrin encapsulation. *Journal of Agricultural and Food Chemistry* **2010**, *58*(19), 10646–10652.
34. Ré, M.I. Microencapsulation by spray drying. *Drying Technology* **1998**, *16*(6), 1195–1236.
35. Alamilla-Beltrán, L.; Chanona-Pérez, L.L.; Jiménez-Aparicio, A.R.; Gutiérrez-López, G.F. Description of morphological changes of particles along spray drying. *Journal of Food Engineering* **2005**, *67*(1–2), 179–184.