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Effect of spray drying at 150, 160, and 170 °C on the physical and chemical properties of maqui extract (*Aristotelia chilensis* (Molina) Stuntz)

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ABSTRACT

Maqui (*Aristotelia chilensis* (Molina) Stuntz) is a native Chilean berry that has the highest antioxidant level compared with other fruit. Anthocyanins, which are the compound with the highest functional level found in maqui, are relatively unstable and quite susceptible to degradation during processing and storage. In addition, maqui is a highly perishable seasonal fruit and it is necessary to find conservation methods for the developed products. Microencapsulation is one way to protect anthocyanins from degradation reactions; it is defined as a process by which certain bioactive substances are introduced in a matrix or wall systems aimed at impeding their loss and protecting them from the reaction with other compounds and/or prevent them from suffering oxidation reactions caused by light or oxygen. The objective of the present study was to evaluate the effect of drying inlet temperature on the physical properties, total polyphenol content, total anthocyanin content, and antioxidant activity of spray-dried maqui extract. Inlet temperatures were 130, 150, and 170 ºC, while other parameters were constant, such as feed flow (18 mL min^{-1}) and encapsulating agent concentration (maltodextrin and gum Arabic). The best drying inlet temperature was 170 ºC; it produced powders with lower moisture content (1.61%), water activity (0.15), and L* coordinate (11.16), as well as increased hygroscopicity (24.01%) and solubility (92.70%). The lowest total polyphenol content loss (23.05 mg gallic acid equivalents g^{-1}), total anthocyanin content (21.46 mg cyanidin-3-glucoside g^{-1}), and antioxidant activity (85.76% 2,2-diphenyl-1-picrylhydrazyl inhibition) occurred at 170 °C. The size of the powder particles allowed classifying them as microcapsules. Maqui extract microencapsulation provides powders with adequate stability during storage.

Key words: Encapsulating agents, maqui, microencapsulation, spray drying.

INTRODUCTION

Maqui (*Aristotelia chilensis* (Molina) Stuntz) is an indigenous berry found in Chile; it is characterized as having one of the highest antioxidant levels when compared to other fruit worldwide (Girones-Vilaplana et al., 2012). Some of the antioxidants found in maqui are anthocyanins, which are responsible for its intense characteristic purple color. Furthermore, their high antioxidant activity is associated with health benefits, such as anti-inflammatory, antidiabetic, antiteratogenic, hypoglycemic, and cardioprotective effects (Fredes et al., 2012; Girones-Vilaplana et al., 2014; Daville et al., 2015; Vergara et al., 2015). This is why interest for this berry has grown both nationally and internationally in recent years.

However, this berry exhibits high water activity and is therefore highly perishable and susceptible to microbial deterioration, enzymatic reactions, and oxidation (Rodríguez et al., 2016). The anthocyanins are also unstable during processing and storage (Cavalcanti et al., 2011). Microencapsulation is a solution to this problem; it is applied to protect sensitive compounds and increase useful life. Different methods currently exist to carry out microencapsulation; spray drying is the method most used in the food industry because it is economical, rapid, and effective in protecting this compound (Parra, 2010). Therefore, the present study investigated the influence of drying inlet temperature on maqui extract microencapsulation with maltodextrin and gum Arabic.

MATERIALS AND METHODS

Maqui (*Aristotelia chilensis* (Molina) Stuntz) fruit of the 2016 harvest were obtained from the Fundo Las Pataguas in the commune of Coihueco (38°37'60" S, 72°13'50" W), Ñuble Region, Chile. Fruits were prepared (weeds were removed) and weighed to produce juice. Fruits were mature, purple-colored, with 3 to 5 seeds per fruit, soluble solids at 32 °Brix, and 53.4% RH. The juice was later extracted in a juicer (HR1832, Phillips, Amsterdam, The Netherlands) and the resulting tissue components (skin, seeds, and pulp) were extracted with water at 1:1 (p/v) ratio. Both the juice and extract were mixed and filtered using Whatman Nr 1 filter paper to eliminate suspended solids and prevent clogging of the atomizer nozzle during drying, and this filtrate was used as an extract for spraying. The food-grade encapsulating agents were maltodextrin (GLUCIDEX IT 19, Roquette, Lestrem, France) and gum Arabic (Instantgum BB, Nexira, Sao Paulo, Brazil).

The encapsulating agents were weighed at 1:1 gum Arabic and maltodextrin ratio, which was 10% of the final solution to be encapsulated. These were then added directly to the extract (15 °Brix) when it reached 23 ± 2 ºC under shaking, and the mixture was diluted to 100 mL with distilled water (Dragon Lab, model MS-H-S, Beijing, China) until it was completely dissolved. The ratio between encapsulating agent weight and maqui extract was 2:3. The food solution exhibited a solid soluble content of approximately 25 °Brix at the time of encapsulation.

Spray drying

Spray drying was carried out with laboratory-scale Lab Scale Spray equipment (Pilotech Instrument, YC-015, Shaanxi, China) with a 0.7 diameter atomization nozzle and 450 mm × 225 mm main pulverization chamber. The inlet/outlet drying temperatures were 130/70 ºC, 150/72 ºC, and 170/75 ºC, respectively, and the feed flow was maintained constant (18 mL min^{-1}).

Moisture content, water activity, hygroscopicity, and solubility

Powder moisture content was gravimetrically determined at 105 ºC in a forced air oven (UFE 700, Memmert, Schwabach, Germany) until reaching constant weight according to the AOAC method 925.10 (AOAC, 2005). Water activity was determined with a dew point hygrometer (Aqualab 4TE Decagon, Devices Inc., Pullman, Washington, USA) at 25 ºC. The methodology described by Cai and Corke (2000) was modified to determine hygroscopicity. Approximately 0.5 g each powder was weighed, placed in previously weighed Petri dishes, and put in a desiccator containing a saturated sodium chloride solution at room temperature. Samples were weighed until reaching constant weight and the result was expressed as a percentage of absorbed moisture. The methodology described by Eastman and Moore (1984) was modified to evaluate powder solubility. Solubility was calculated as the difference in weight and expressed as a percentage (%).

Scanning electron microscopy (SEM)

Powder morphology was evaluated with a scanning electron microscope (model SU3500, Hitachi, Krefeld, Germany). Powders were placed on strips of carbon double-sided adhesive tape (Shinto Chemitron Co., Tokyo, Japan), which were then affixed to metal slides, and immediately sealed with carbon (Conductive Carbon Glue, Pelco, Ted Pella Inc., Redding, California, USA). Images were captured with 10.0 kV acceleration voltage.

Color measurement

Color parameters were evaluated by reflectance with a colorimeter (CR-400, Minolta Chroma Meter, Tokyo, Japan) that was previously calibrated with a blank. The colorimetric coordinates L*, a*, and b* of the CIELAB system were obtained and used to calculate chroma ($C_{ab}^* = [a^2 + b^2]^{1/2}$) and tone ($h_{ab}^o = \tan^{-1}(b^*/a^*)$).

Total polyphenol content

Powder total polyphenol content (TPC) was evaluated by the Folin-Ciocalteu method in accordance with the procedure described by Sarkis et al. (2014), which was modified. To extract TPC, 0.5 g powder were dissolved in 10 mL water and shaken in a vortexer (Heathrow Scientific Vortexer, Vernon Hill, Illinois, USA) at 3000 rpm for 10 min and the supernatant was immediately collected in a Falcon tube. The following were added to a test tube: 40 µL supernatant, 3.16 mL distilled water, 200 µL Folin-Ciocalteu reagent, and 600 µL 20% anhydrous sodium carbonate; the test tube was shaken and left in darkness for 30 min after which an absorbance reading was taken at 765 nm with a spectrophotometer (Spectronic Genesys 2PC, Rochester, New York, USA). Gallic acid solutions between 0 and 1000 µL g^{-1} were used to construct the calibration curve. Results were expressed in mg of gallic acid equivalents per gram of sample (mg GAE g^{-1}).

Total anthocyanin content

Total anthocyanin content was determined by the pH differential method described by Wrolstad et al. (2005). Two buffer solutions of potassium chloride (0.025 M, pH 1.0) and acetic acid (0.4 M, pH 4.5) were prepared, while two sample dilutions were prepared to quantify total anthocyanin content using the buffer solution at 1:9 ratio (sample:buffer). The mixture was shaken and left in darkness for 5 min at which time absorbance readings were taken at 510 and 700 nm with a spectrophotometer (Spectronic Genesys 2PC). Anthocyanin content was calculated as cyanidin-3-glucoside per gram using the molar extinction coefficient 26 900 L cm^{-1} mol^{-1} and molecular weight of 449.6 g L^{-1}.

Antioxidant activity

Antioxidant capacity was determined by the 2,2-diphenyl-1-picrylhydrazyl (DPPH) spectrophotometric method according to the method described by Brand-Williams et al. (1995), which was modified. Results of antioxidant activity on the effect of inlet temperature on maqui extract drying were expressed as a percentage of discoloration. Microencapsulated powder stability results were expressed as mg Trolox equivalents per gram sample (mg TE g^{-1}), and different Trolox concentrations between 0 and 100 µg g^{-1} were prepared for the calibration curve. The concentration was determined at 510 nm wavelength with a spectrophotometer (Spectronic Genesys 2PC).

Study of powder stability under the effect of light

Microencapsulated maqui powder was used for the stability study because of the instability of the functional compounds when exposed to light. Maqui powders were placed on Petri dishes (50 mm diameter) that were sealed with Parafilm and stored in a light chamber with two fluorescent tubes (80 W, 2.28 W m^{-2}) parallel to the samples at a distance of 50 cm; the chamber reached 36 ± 2 °C and 12 ± 2% RH. Total polyphenol content, total anthocyanin content, antioxidant activity, and color were measured to evaluate stability. Samples were stored for 42 d and analyzed every 7 d.

Statistical analysis

All the analyses were performed in triplicate with the exception of the stability study that was carried out in duplicate with a one-way ANOVA. Treatment analysis was performed by the least significant difference (LSD) test ($p < 0.05$) with the Statgraphics Centurion XVI.I program (Statgraphics Technologies, The Plains, Virginia, USA).

RESULTS AND DISCUSSION

Effect of drying inlet temperature on powder physical properties

Table 1 displays the physical properties of the powders obtained at different drying inlet temperatures. Moisture content in the maqui powders varied between 1.61 and 3.43 g 100 g^{-1} dry solids and decreased as spray drying inlet temperature increased. This is because of a higher temperature gradient in the process when inlet temperatures are higher, resulting in rapid water evaporation and powder production with lower moisture. These values were similar to those found by Tonon

Table 1. Physical parameters of microencapsulated maqui powders at different inlet temperatures.

Inlet temperature	Moisture	Water activity	Solubility	Hygroscopicity	Mean diameter
ºC	g 100 g^{-1}		%	%	µm
130	3.43 ± 0.72a	0.24 ± 0.01a	90.67 ± 0.64b	21.13 ± 1.06b	47.41 ± 16.68c
150	2.41 ± 0.08b	0.19 ± 0.01b	92.69 ± 1.45a	22.76 ± 1.52ab	51.70 ± 20.02b
170	1.61 ± 0.19c	0.15 ± 0.01c	92.70 ± 0.69a	24.01 ± 0.75a	62.58 ± 19.17a

Different letters in the same column indicate significant differences among samples ($p < 0.05$).

et al. (2008), Frascareli et al. (2012), and Daza et al. (2015), who observed a reduction in moisture content when spray drying inlet temperature increased.

Water activity in the maqui powders varied between 0.15 and 0.24, which indicates good powder stability; the maqui powders can be considered as microbiologically stable because there is no microbial growth when water activity is low (Reid and Fennema, 2010). The statistical analysis indicates that there is a significant difference ($p < 0.05$) in all the treatments for both moisture content and water activity in the microencapsulated maqui powders, which is lower at 170 ºC.

Powder hygroscopicity varied between 21.13% and 24.01%, and the increase in inlet temperature caused an increase in maqui powder hygroscopicity. These results concur with Tonon et al. (2008), Ferrari et al. (2012), and Daza et al. (2015) in their research about spray drying cagaita (*Eugenia dysenterica* D.C.) extract, rosemary (*Rosmarinus officinalis* L.) essential oil, and acai (*Euterpe oleracea* Mart.) juice, respectively. Therefore, higher water content in powders generates a lower water concentration gradient between the powder and the atmosphere; this is related to higher water elimination and thus higher moisture absorption to reach its equilibrium in an environment with high relative humidity (Tonon et al., 2008; Fernandez et al., 2013). As for the hygroscopicity values obtained for the maqui powders in the present study, these were similar to those found by Mahdavi et al. (2016), who used maltodextrin and gum Arabic in the microencapsulation of barberry (*Berberis vulgaris* L.)

Powder solubility varied between 90.67 and 92.70 and the statistical analysis indicates nonsignificant difference ($p < 0.05$) between treatments at 150 and 170 ºC, but a difference at 130 ºC related to lower solubility. According to the data obtained, increased inlet temperature caused increased microencapsulated maqui powder solubility; these results concur with Daza et al. (2015) and Venil et al. (2016), who found that solubility increases as drying inlet temperature increases. This could be due to the effect of inlet temperature on moisture content that leads to an increase in particle size and thus decreases the time required to dissolve the powder (Sarbandi et al., 2014). Solubility values of maqui microcapsules are similar to those attained by Mahdavi et al. (2016) in barberry extract microencapsulation in which the same encapsulating agents were used.

Powder morphology

Micrograph analysis (Figure 1) indicates that all the powders exhibited smooth spherical shapes in different sizes. Ferrari et al. (2012), Tonon et al. (2008), and Ersus and Yurdagel (2007) observed this smooth spherical structure in spray drying microencapsulation of blackberry, acai, and black carrot extracts, respectively.

Table 1 reports the mean diameter values of maqui powders at different temperatures, which exhibit significant differences ($p < 0.05$) between each other, and size allows classifying them as microcapsules (Azeredo, 2005). The increase in temperature generates particles with higher mean diameters. These results coincide with those pointed out by Tonon et al. (2008) and Shishir et al. (2014). These authors concluded that processes performed at higher temperatures produce larger particles than processes at lower temperatures because more rapid drying promotes a more immediate structure formation and thus prevents particle shrinkage during drying.

Color parameters

Table 2 establishes that the L* parameter decreases when drying inlet temperature increases, indicating that the sample is darker. These results coincide with studies by Daza et al. (2015) and Quek et al. (2007) showing that increasing inlet temperature produces cagaita and acai powders with lower luminosity. This can be related to the higher water loss found in more concentrated and darker products (Tonon et al., 2009).

Figure 1. Scanning electron micrograph of spray-dried powders at different temperatures: 130 (a), 150 (b), and 170 °C (c).

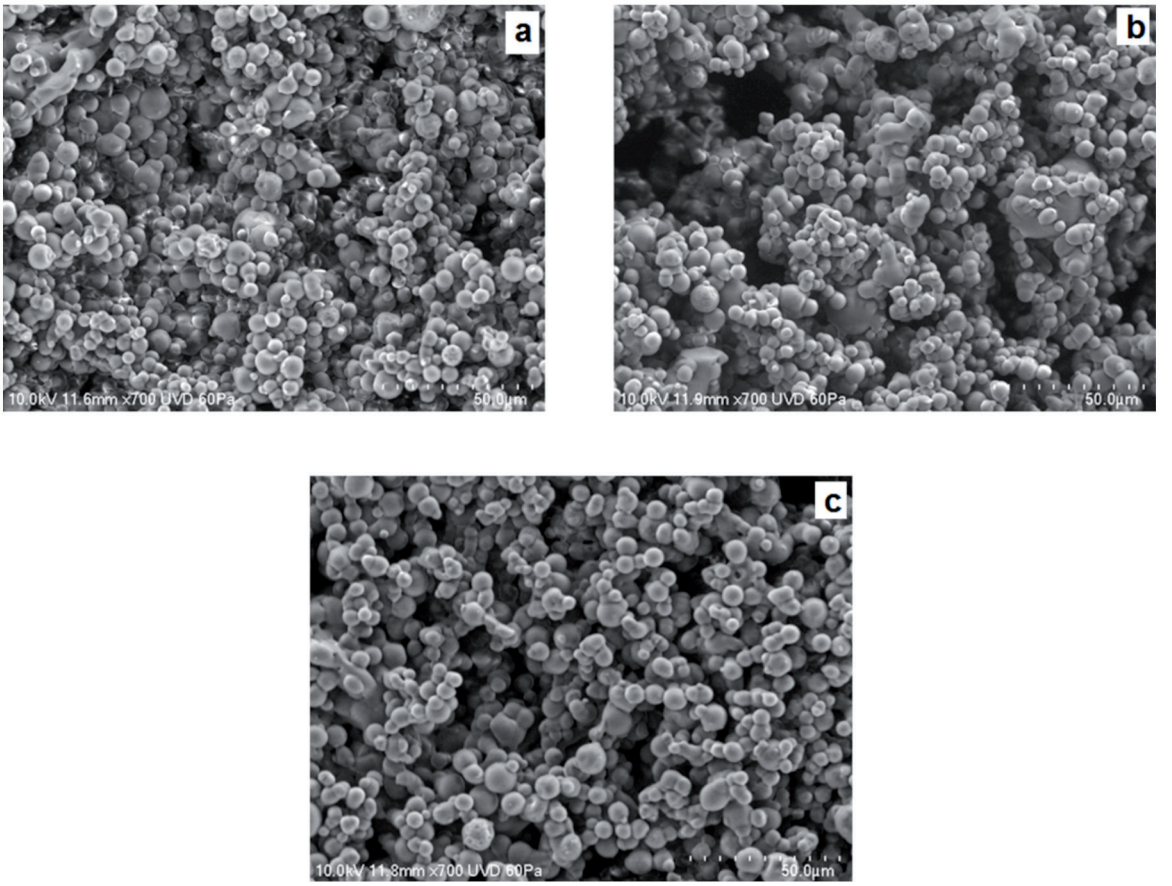

Table 2. Color parameters of microencapsulated maqui powders at different inlet temperatures.

Inlet temperature (ºC)	L*	a*	b*	C_{ab}*	h_{ab}*
130	13.55 ± 0.21a	14.42 ± 0.17c	-0.87 ± 0.18a	14.45 ± 0.18c	356.54 ± 0.66a
150	12.91 ± 0.10ab	16.05 ± 0.47a	-0.87 ± 0.11a	16.17 ± 0.47a	356.94 ± 0.29a
170	11.60 ± 0.20c	15.16 ± 0.40b	-2.85 ± 0.02b	15.39 ± 0.37b	349.33 ± 0.21b

L*: Luminosity; a*: red to green colorimetric coordinate; b*: yellow to blue colorimetric coordinate; C^*_{ab}: chrome; h^*_{ab}: angle tone.
Different letters in the same column indicate significant differences among samples ($p < 0.05$).

Chroma (C_{ab}*) was significantly different ($p < 0.05$) under different work conditions. Tone angle (h_{ab}º) behaved in the same way as luminosity by decreasing when temperature increased. These values ranged from 349.33 ± 0.21 to 356.54 ± 0.66, which correspond to the fourth quadrant of the Cartesian plane, the characteristic red-purple color typical of anthocyanins contributing to that tonality.

In general, luminosity and tone decreased, which indicates that inlet temperature affects powder color and powders become darker when temperature increases. Manolopoulou and Varzakas (2011) observed that when luminosity and tone decrease, the color becomes darker. It is therefore concluded that the treatment at 170 ºC exhibits better product coloration since the color resembles the one found in the fruit.

Total polyphenol content, total anthocyanin content, and antioxidant activity

Table 3 indicates that the loss of total polyphenol content, total anthocyanin content, and antioxidant activity is lower compared with the control when drying inlet temperature increases. This could be due to the fact that high temperatures generate higher water evaporation (Souza et al., 2015). Drying is therefore more rapid, contact time is lower between the components and hot air, and the loss of labile compounds is lower. However, these results are contradictory with those reported by Cai and Corke (2000). Quek et al. (2007) and Tonon et al. (2009) indicate that the higher loss of this type of compounds occurs with the increase in temperature because of their high sensitivity. Mishra et al. (2013), who found a significant reduction in total polyphenol content when the inlet temperature increases from 125 to 175 °C, mentioned similar results.

Likewise, when total polyphenol concentration (Table 3) in the powders decreases, total anthocyanin content and antioxidant activity also decrease. Jiménez-Aguilar et al. (2011) reported a similar result. According to the total polyphenol content, total anthocyanin content, and antioxidant activity, the best drying condition is the treatment at 170 ºC. This occurs because of the higher content of these compounds and antioxidant activity at this temperature.

Evaluation of powder stability during light-exposed storage

Stability of total polyphenols, total anthocyanins, and antioxidant activity. Results in Table 4 refer to the microencapsulated maqui powders obtained by drying at 170 °C. At this temperature, anthocyanin content and antioxidant activity decrease during storage, showing significant differences ($p < 0.05$) between storage times. Ersus and Yurdagel (2007) evaluated the stability of black carrot (*Daucus carota* L. subsp. *sativus* (Hoffm.) Schübl. & G. Martens var. *atrorubens* Alef.) anthocyanin pigments in microencapsulated powders by spray drying and indicated similar results, that is, anthocyanin content decreased by 33% after 64 d storage. Furthermore, Gallo et al. (2015), who worked under similar conditions to those of the present study, determined a 28% loss of the initial content when studying anthocyanin stability against light in microencapsulated grape (*Vitis vinifera* L.) juice.

An increase in total polyphenol content during storage was verified in the present study. Saénz et al. (2009) observed the same behavior during storage of microencapsulated cactus pear (*Opuntia ficus-indica*) powder obtained by spray drying. According to Mishra et al. (2013), the increase in polyphenol content can be explained by the possible formation

Table 3. Total polyphenol content, total anthocyanin content, and antioxidant activity of microencapsulated maqui powders at different inlet temperatures.

Inlet temperature	Total polyphenols	Total anthocyanins	Antioxidant activity
ºC	mg GAE g^{-1}	mg Cyanidin-3-glucoside g^{-1}	% DPPH inhibition
Control*	35.66 ± 13.71a	30.53 ± 0.66a	93.38 ± 0.93a
130	16.35 ± 0.35c	16.25 ± 0.05d	70.72 ± 2.02c
150	22.05 ± 1.49b	19.38 ± 0.22c	74.37 ± 0.76b
170	23.05 ± 0.64b	21.46 ± 0.56b	85.76 ± 2.22a

*Initial extract.
GAE: Gallic acid equivalents; DPPH: 2,2-diphenyl-1-picrylhydrazyl.
Different letters in the same column indicate significant differences among samples ($p < 0.05$).
Results are expressed on a dry basis.

Table 4. Total polyphenol content, total anthocyanin content, and antioxidant activity of maqui powders dried at 170 °C during light-exposed storage.

Time	Total polyphenol content	Total anthocyanin content	Antioxidant activity
d	mg GAE g^{-1}	mg Cyanidin-3-glucoside g^{-1}	mg TE g^{-1}
0	19.82 ± 1.14b	16.51 ± 0.42a	50.95 ± 2.61ab
7	19.92 ± 1.53b	15.36 ± 0.74b	54.13 ± 3.88a
14	20.37 ± 1.12b	15.10 ± 0.69bc	49.58 ± 0.98b
21	25.74 ± 4.02a	14.13 ± 1.09d	39.76 ± 6.94c
28	19.93 ± 1.26b	13.58 ± 0.34d	37.33 ± 1.36c
35	21.14 ± 1.12b	12.17 ± 0.96c	37.65 ± 3.25c
42	24.13 ± 4.33a	14.29 ± 0.57cd	38.22 ± 1.38c

GAE: Gallic acid equivalents; TE: Trolox equivalent.
Different letters in the same column indicate significant differences among different storage times ($p < 0.05$).

of phenolic compounds that could influence their total content. On the contrary, Jiménez-Aguilar et al. (2011) studied the stability of blueberry (*Vaccinium myrtillus*) juice by spray drying and observed a decrease in total polyphenols.

Color parameter stability of powders under the effect of light. Table 5 reveals the color parameters of powders obtained at 170 °C during storage in which there were no important changes in luminosity. On the other hand, there was a significant decrease ($p < 0.05$) in chroma (C_{ab}*), which was probably due to anthocyanin degradation. Souza et al. (2017) found the same behavior and pointed out that chroma (C_{ab}*) best represents color changes during photostability. As for tone (h_{ab}º), there is a significant increase ($p < 0.05$), although there is no important change in tonality during storage. In general, all the color parameters during storage were located in the fourth quadrant of the CIELAB scale in the red-purple color.

Table 5. Color parameters of maqui powders (170 °C) during light-exposed storage.

Time (d)	L*	C_{ab}*	h_{ab}*
0	33.09 ± 0.99a	9.15 ± 0.25a	354.5 ± 1.23c
7	32.96 ± 0.33a	8.36 ± 0.45b	357.9 ± 1.70b
14	32.92 ± 0.89a	7.62 ± 0.39c	357.7 ± 1.39b
21	32.81 ± 1.04ab	7.45 ± 0.39c	358.5 ± 0.77b
28	32.33 ± 0.28ab	6.36 ± 0.26d	358.7 ± 1.10b
35	32.40 ± 0.43ab	6.36 ± 0.34d	359.02 ± 0.50a
42	31.79 ± 0.12b	5.44 ± 0.12e	359.1 ± 1.07a

L*: Luminosity; C_{ab}*: chrome, h_{ab}*: angle tone.
Different letters in the same column indicate significant differences among different storage times ($p < 0.05$).

CONCLUSIONS

We determined that spray drying inlet temperature significantly affected the physical properties under study. The best drying inlet temperature was 170 ºC, which produced powders with lower moisture content, water activity, and luminosity, as well as an increase in solubility, total polyphenol content, total anthocyanin content, and antioxidant activity. Maqui powder particle size increased as temperature increased and produced spherical particles with a smooth surface and size that allowed their classification as microcapsules. Regarding stability, we determined that compound loss was low, and this indicates good behavior of the powder against light. In conclusion, the microencapsulated maqui powder obtained at 170 °C exhibits good physicochemical conditions and high bioactive compound content for processing and consumption.

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