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Use of gelatin-maltodextrin composite as an encapsulation support for clarified juice from purple cactus pear (*Opuntia stricta*)

Roberto Castro-Muñoz [a], Blanca E. Barragán-Huerta [b, **], Jorge Yáñez-Fernández [a, *]

[a] Departamento de Biotecnología Alimentaria, Unidad Profesional Interdisciplinaria de Biotecnología, Instituto Politécnico Nacional, Av. Acueducto S/N Col. Barrio La Laguna, Ticoman, Mexico, D.F., CP 07340, Mexico

[b] Departamento de Ingeniería en Sistemas Ambientales, Escuela Nacional de Ciencias Biológicas, Instituto Politécnico Nacional, Av. Wilfrido Massieu s/n, Unidad Profesional Adolfo López Mateos, México, D.F., CP 07738, Mexico

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ABSTRACT

Juice rich in betalains (with 62.69 mg L^{-1}) and with a high antioxidant activity (14.35 TEAC) from purple cactus pear was encapsulated by spray-drying using gelatin (G) and maltodextrin (M) as the wall material. This investigation was conducted using an experimental design of 2^2 mixes with two replicates at the center point. The microstructures generated in all of the experiments (yield = 7.76–14.86 g 100 g^{-1}) were characterized in terms of their moisture content (1.87–6.95 g 100 g^{-1} dry basis), a_w (<0.3), betalain content (11–35 mg 100 g^{-1}), antioxidant activity (4–29 TEAC), colorimetry (L = 17.26–40.9; a^* = 17.16 –37.20; b^* = 6.26–15.46), h° (15.06–29.66), C (18.75–43.29), hygroscopicity (19.39–28.13 g 100 g^{-1}). Samples with G:M ratio (2.5:7.5) and high antioxidant capacity were analyzed by SEM which showed that the powders presented a spherical shape (particle size of 6.14μm ± 4.96) with melting points ranging from 205 to 235 °C.

1. Introduction

Cactus pear (*Opuntia* spp.) is a native fruit from America that grows in arid and semiarid regions (Saénz, Tapia, Chávez, & Robert, 2009). There are colored fruits (green, red, yellow, or purple) due to the presence of various pigments, such as betalains (Castellar, Obón & Fernández, 2006; Díaz, Santos, Kerstupp, Villagómez & Scheivar, 2006). Fruits of the *Opuntia* genus have little industrial utilization but are widely consumed as fresh fruits. Due to their high water content (a_w > 0.8), neutral pH, and soluble solid content, the pulp has high susceptibility to microbial attack, which makes the post-harvest handling of these fruits difficult (León, Méndez & Rodríguez, 2010). Specifically, *Opuntia stricta*, which is better-known purple as cactus pear, is one of the few natural sources of betalains, in addition to beetroot. For this reason, cactus pear is an attractive alternative to the synthetic colorants that are commonly are used in foods. Furthermore, betalains have valued properties, such as antioxidant activity (Stintzing et al., 2005), which plays an important role in the human health. Betalains have many disadvantages, such as low stability in response to sudden changes in temperature and pH (Moβhammer, Maier, Stintzing, & Carle, 2006; Moβhammer, Rohe, Stintzing, & Carle, 2007) and are affected by water activity, exposure to light, oxygen, and enzymatic activities (Castellar, Obón, Alacid & Fernández, 2003). One advantage associated with the isolation of betalains from *O. stricta* is that these compounds are solubilized directly in water without the need of an organic solvent. This differs from the main methods, which employ solvents such as diethyl-ether, ethyl-acetate, methanol, and acetic acid (El-Gharras, Hasib, Jaouad, El-bouadili, & Schoefs, 2008; Galati et al., 2003;). Due to the high water solubility of betalains, these compounds can be separated through membrane processes mainly using the ultrafiltration technology, which it is a non-thermal separation process with a fine cut off of 100–200 kDa (Cassano, Conidi, & Drioli, 2010). This process has been used for the recovery of several compounds, including phenols and acids, i.e., citric, ascorbic, folic, and glutamic acids from fruit juices, such as yellow cactus pear (Cassano et al., 2010), orange (Cassano, Marchio, & Drioli, 2007) and bergamot (Conidi, Cassano, & Drioli, 2011).

One of the means that have been used to conserve the properties of certain bioactive compounds from cactus pear is the use of spray drying with several encapsulating agents, such as glucose syrup, maltodextrin, and inulin (Obón, Castellar, Alacid, &

* Corresponding author. Tel.: +52 55 57296300x56477; fax: +52 55 57296300x52300.
** Corresponding author. Tel.: +52 55 57296300x52310; fax: +52 55 57296300x52300.
E-mail addresses: bbarraganh@ipn.mx, bbarraga59@yahoo.com (B.E. Barragán-Huerta), jyanezfe@ipn.mx, jyanezfe.ipn@gmail.com (J. Yáñez-Fernández).

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Fernández-López, 2009; Saénz et al., 2009). The gelatin is a protein that it may be an ideal candidate for the wall material due to its surface activity with a positive or negative change depending of the pH, which is able to result in mixtures with traditional encapsulants, such as some polysaccharides, oils, and inclusive materials in ethanolic solution. Additionally, gelatin has other attractive characteristics, such as biocompatibility, degradation, non-toxicity, and readily excreted products (Bruschi, Cardoso, Lucchessi, & Gremiao, 2003; Kim et al., 2006). The objectives of this study were to produce spray-dried microcapsules of bioactive compounds from purple cactus pear using a gelatin-maltodextrin composite as the encapsulation support and to characterize the complexes generated with low-stability components.

2. Materials and methods

2.1. Materials

The purple cactus pear fruits used in this study were harvested in July 2012 from San Martin de las Piramides, México (geographical coordinates: 19° 37′ 05″ and 19° 46′ 20″ north latitude and between 98° 45′ 40″ and 98° 53′ 27″ west longitude). The chemicals used were of analytical grade. Maltodextrin DE10 was purchased from ®Projugo (México), and gelatin was purchased from ®Reasol (México).

2.2. Methods

2.2.1. Clarification of juice from purple cactus pear

The fruits were chopped and pulped using a ®TURMIX juice extractor. The juice obtained was centrifuged at 10,332 × *g* for 20 min in a Beckman Coulter centrifuge (model J2-MC, USA). The supernatant obtained was clarified by ultrafiltration (UF) in a module of polysulfone hollow fibers (Amersham Biosciences Corp. Model CFP-1-E-4A, USA) with an area of 420 cm^2 (pore size 100 kDa). The operating conditions for the separation processes were: transmembrane pressure (179 kPa), pH (6.1), and temperature (27 °C). The permeate was maintained in the dark at −20 °C until use.

2.2.2. Antioxidant activity

The antioxidant activity was determined using the DPPH method (stability of 1,1-diphenyl-2-picrylhydrazyl radical) according to Kuskoski, Asuero, García, Mancini, and Fett (2005). Briefly, 0.05 mL of the clarified juice was added to 2.95 mL of methanol (dilution factor = 60) and 2 mL of the DPPH 0.1 μmol L^{-1} methanolic solution. The absorbances of the solution samples were measured at 517 nm at 30-min intervals, and the antioxidant activity was calculated by interpolation using a standard curve constructed with Trolox and DPPH 0.1 μmol L^{-1} methanolic solution. The results are expressed as μmol of Trolox equivalents per milliliter of sample (TEAC) (Kuskoski, Asuero, Troncoso, Mancini-Filho, & Fett, 2005). The antioxidant activity of the powders was determined using 50 mg of spray-dried powder and 2.95 mL of methanol; this mixture was magnetically stirred for 5 min. After centrifugation at 10 332 × *g* for 10 min, 2 mL of the DPPH 0.1 μmol L^{-1} methanolic solution was added to the supernatant.

2.2.3. Quantification of betalains

The betalains was quantified through the spectrophotometric method described by Cassano et al. (2010) with some modifications. First, 10 g of juice or the spray-dried powders were mixed with 80 mL 100 mL^{-1} ethanol-aqueous solution to a final volume of 100 mL. The mixture was filtered and stored in an amber bottle, and 1 mL of the mixture was diluted to a final volume of 5 mL using 80 mL 100 mL^{-1} ethanol-aqueous solution. The resulting diluted mixture was magnetically stirred for 1 min. The absorbance of the solution was measured at 538 nm (for betacyanins) and 476 nm (for betaxanthins). The pigment content was determined using the Equation (1) (Soriano et al., 2007):

$$BC = \left(\frac{A \cdot DF \cdot V \cdot 1000}{E^{1\%}_{1\ cm} \cdot L \cdot P}\right) \quad (1)$$

where BC is the betalain content (mg betalains L^{-1} in juice and mg betalains kg^{-1} in powders), A is the absorbance for each pigment, DF is the dilution factor, P is the weight or volume of the sample, V is the aliquot volume, is the extinction coefficient in ethanol (betacyanins = 1120 and betaxanthins = 750) and L is the path length (1 cm).

2.2.4. Color measurement

First, 30 mL of the juice was placed in a Gerber container, and the L*, a*, and b* color parameters of the CIELAB scale were measured using a colorimeter (®Konica Minolta Colorimeter CR-10, Japan). Alternatively, 15 g of the powders was placed in a Petri dish, and the color readings were performed as described above. Triplicate samples were analyzed, and the mean was recorded. The hue angle (h°) and chroma (C) values were calculated using the Equations (2) and (3), respectively (Mapari, Meyer, & Thrane, 2006):

$$h^{\circ} = \tan^{-1}\left(\frac{b^*}{a^*}\right) \quad (2)$$

$$C = (a^{*2} + b^{*2})^{1/2} \quad (3)$$

2.2.5. Preparation and spray-drying of microcapsules

First, 380 mL of the clarified juice was mixed with 38 g of wall material to obtain a solution of 10 g 100 mL^{-1} solids. The ratio of gelatin (G) to maltodextrin (M) was defined through an experimental design of 2^2 mixes with two replicates at the central point (Design Expert 8.0v, USA). This design included 19 experiments, in which the drying inlet air temperature (Ti) was changed from 110 to 140 °C and the G:M ratio was varied from 0 to 1.0 (Table 1).

Table 1
Experimental design of 2^2 mixes used in the spray-drying experiments.

No. Treatment	Encapsulants (10 g 100 g^{-1})		
	Gelatin (g 100 g^{-1})	Maltodextrin (g 100 g^{-1})	Temperature (°C)
1	0.000	1.0w00	140.0
2	0.250	0.750	125.0
3	0.375	0.625	117.5
4	0.250	0.750	140.0
5	0.500	0.500	140.0
6	0.125	0.875	117.5
7	0.500	0.500	110.0
8	0.500	0.500	140.0
9	0.500	0.500	110.0
10	0.000	1.000	110.0
11	0.000	1.000	117.5
12	0.125	0.875	132.5
13	0.000	1.000	110.0
14	0.375	0.625	132.5
15	0.000	1.000	140.0
16	0.500	0.500	125.0
17	0.250	0.750	140.0
18	0.250	0.750	110.0
19	0.000	1.000	125.0

A Büchi mini spray dryer (Model B-290, Germany) was employed for the spray-drying process. The spray-drying was conducted at an aspirator rate of 32.90 $m^3\ h^{-1}$, a pressure of 40 kg cm^{-2}, a temperature of 25 °C, and a feed flow of 1 mL min^{-1}. All of the spray-dried powders were collected, weighted to calculate the yield, and maintained in polypropylene bags under vacuum in the dark until analysis. The spray-dried powders were characterized in terms of their moisture content, yield, water activity, color, antioxidant activity, betalain content, solubility, hygroscopicity and scanning electron microscopy (SEM).

2.2.6. Yield of microencapsulation

The spray-drying yield was calculated through a determination of the power recovered. The yield was determined using Equation (4):

$$\%YE = \left(\frac{W_2}{W_1}\right)*100 \quad (4)$$

where YE is the yield (g 100 g^{-1}), W_2 is the weight (g) of the collected product, and W_1 is the weight (g) of the non-solvent mass in the feed (Xue et al., 2013).

2.2.6.1. Encapsulation efficiency. The encapsulation efficiency (EE) was calculated as the ratio of actual to theoretical on BC and TEAC in dry microspheres, the percentage of EE was determined as Equation (5) (Su et al., 2008):

$$\%EE = \left(\frac{\text{Amount of actual BC or TEAC}}{\text{Amount of theorical BC or TEAC}}\right)*100 \quad (5)$$

2.2.7. Characterization of microcapsules

2.2.7.1. Moisture content and water activity. The moisture content of the powders was measured with a thermo balance (Adam AMB 310, UK). One gram of each of the microcapsules was used to determinate the moisture, which is expressed as a percentage on a dry basis (g 100 g^{-1}). The measurement of the water activity was conducted using a water activity meter (Aqualab model 0301962, USA). Triplicate samples were analyzed, and the mean was recorded.

2.2.7.2. Hygroscopicity. The hygroscopicity was determined according to the method followed by Tonon, Brabet and Hubinger (2008). Briefly, 1 g of the spray-dried powder was placed in a container at 25 °C with a NaCl saturated solution (76% RH). After one week, the samples were weighed, and the hygroscopicity was expressed as g of absorbed moisture per 100 g of dry solids (g 100 g^{-1}).

2.2.7.3. Solubility. One gram of spray-dried powder was dispersed into 100 mL of distilled water at 30 °C, and the suspension was mixed for 5 min in a vortex and then centrifuged at 3780 × *g* for 5 min. Subsequently, an aliquot of 25 mL of the supernatant was placed in a Petri dish, weighed, and dried in an oven at 105 °C for 5 h. The solids were recovered and weighed, and the solubility (g 100 mL^{-1}) was calculated based on the weight difference (Shittu & Lawal, 2007).

2.2.7.4. Scanning electron microscopy (SEM). The particle size and structure of the spray-dried microcapsules were evaluated using a scanning electron microscope (JEOL, JSM-6390LV, Japan). The microcapsules were attached to SEM graphite stubs with a diameter of 1 in using two-sided adhesive tape. The specimens were coated through a sputtering process (Desk IV, Denton Vacuum, USA) with gold–palladium (Au/Pd) for 60 s at an accelerating voltage of 15 kV, and the corresponding images were captured. Images at 2000× magnification were obtained with the software associated with the equipment, and the mean particle size was calculated.

2.2.7.5. Differential scanning calorimetry (DSC). The thermal behavior of the microcapsules and raw materials were evaluated through differential scanning calorimetry (DSC) using a Perkin Elmer calorimeter (Pyris 1, Malaysia). The intervals were established using the method described by Sansone, Mencherini, et al. (2011) at 25–350 °C and 10 °C/min. The melting temperature (T_M) was determined from the thermograms obtained.

2.2.7.6. Statistical analysis. The data are expressed as the means ± standard deviation, and one-way analysis of variance (ANOVA) and correlation analysis were performed using the SAS statistical software (V6.0, SAS Institute S.A de C.V, Mexico). Tukey's multiple range test was used to compare the means. Differences among the means of $p < 0.05$ were considered significant. The dependent variables, such as the betalain content and antioxidant activity, were analyzed by response surface methodology to analyze the effect of the encapsulating agent and temperature on these properties. The analyses were performed using the Design Expert software (version 8.0, USA).

3. Results and discussion

3.1. Clarification of purple cactus pear juice

As shown in Table 2, the membrane process favorably influences the properties of normal juice by decreasing the L values from 18.76 to 11.43 and the C values from 17.62 to 3.36. The high values obtained may be due to the high turbidity of the juice due to the presence of polysaccharides, which were in the membrane step. The data obtained from the measurements of the color parameters in both juices are shown in the first quadrant of the CIELAB color chart (Mapari et al., 2006). The h° value increased from 11.62 in normal juice to 30.39 in the clarified juice, which indicates an increase in the degree of redness in the permeate. In addition, the a* values decreased from 17.26 in normal juice to 2.9 in clarified juice, and the values of b* decreased from 3.55 in normal juice to 1.7 in clarified juice. Compared with the results reported by Obón et al. (2009), who found L, C, a* and b* colorimetry values for purple cactus pear juice of 13.99, 8.00, 7.98, and 0.57, respectively, the respective values for the permeate obtained in this study are lower, i.e., 11.43, 3.36, 2.90, and 1.7, respectively. The h° value of the

Table 2
Colorimetry, betalain content, and antioxidant activity in normal and clarified juices.

	Colorimetry					Quantifications of betalains (mg L^{-1})	Antioxidant activity (TEAC)
	Lightness (L)	a*	b*	Chroma (C)	Hue angle (h°)		
Normal juice	18.76 ± 0.05[a]	17.26 ± 0.00[a]	3.55 ± 0.01[a]	17.62 ± 0.00[a]	11.62 ± 0.05[b]	37.73 ± 0.34[b]	10.91 ± 2.02[a]
Permeate (Clarified juice)	11.43 ± 0.05[b]	2.90 ± 0.05[b]	1.7 ± 0.01[b]	3.36 ± 0.03[b]	30.39 ± 2.33[a]	62.69 ± 2.72[a]	14.35 ± 1.19[a]

Data represents the means ± deviation standard with triplicate for each test. Different superscript letters in the column indicate statistical significance ($p < 0.05$) according to the Tukey least significant different test.

permeate was 30.39, which is higher than the value of 0.07 reported by Obón. All of these differences can be attributed to the membrane process used in our study and another study that only used a simple centrifugation. Additionally, the concentration of betalains (62.69 mg L^{-1} in clarified juice and 37.73 mg L^{-1} in normal juice) caused an increase in the antioxidant capacity. The maximal antioxidant capacity obtained in the clarified juice was 14.35 ± 1.19 TEAC, whereas that found in normal juice was 10.91 ± 2.02 TEAC (or 11.48 μmol Trolox g^{-1}). This value is higher than the values of 4.20 ± 0.51 μmol Trolox g^{-1} and 3.64 ± 0.27 μmol Trolox g^{-1} reported by Butera et al. (2002) and Stintzing et al. (2005), respectively.

In addition, there was a positive correlation (0.7946, $p < 0.0001$) between the betalain content and the antioxidant activity. A positive correlation (0.9628, $p < 0.0001$) was also found between the h° value and the betalain content, and a positive correlation (0.8173, $p < 0.0001$) was observed between the h° value and the antioxidant activity.

3.2. Yield of microencapsulation

The microencapsulation yield of clarified juice was 7.76–14.85 g 100 g^{-1} (Table 3), which is lower than the values of 58 g 100 g^{-1} and 23–81 g 100 g^{-1} reported by Obón et al. (2009) and Saénz et al. (2009), respectively, who used glucose syrup and maltodextrin-inulin composite as encapsulant agents, respectively. The low encapsulation yields may be due in part to wall material being retained in the dryer, a process that is attributed to the incompatibility in solution of the gelatin and maltodextrin, which is likely due to the inverse relationship between the entropy of mixing and the molecular weight of the maltodextrin (Kasapis, Morris, Norton, & Gidley, 1993). The highest yield using the encapsulant G:M ratio of 2.5:7.5 at a drying temperature of 125 °C was 14.85 g 100 g^{-1}, and this also resulted in the highest values in the h° angle (29.66) related to the color strength and a high antioxidant activity.

3.3. Characterization of microcapsules

3.3.1. Water activity and moisture content of the microcapsules

The moisture content of the microencapsulated juice varied from 1.87 to 6.95 g 100 g^{-1} (Table 3). The powder moisture content was observed to decrease with an increase in the inlet air temperature, and a larger temperature gradient in the process allowed an increased evaporation of water (Queck, Chok, & Swedlund, 2007; Tonon et al. 2008). Similarly, the powders have a_w values less than 0.3, which are similar to those reported by Queck et al. (2007) in their study on the spray-drying of watermelon juice (a_w ~ 0.3).

3.3.2. Hygroscopicity and solubility of the microcapsules

The hygroscopicity increased with an increase in the inlet temperature. This finding is likely because the high temperatures allowed greater evaporation of water and thus reduced the moisture of the powders and conversely increased the capture of water molecules by the samples. The microencapsulated juice presented hygroscopicity between 19.34 and 28.13 g water 100 g^{-1} sample, which is higher than the value of 13–15 g 100 g^{-1} reported by Tonon et al. (2008). One of the main factors that affect the hygroscopicity of the powders is the drying temperature to which it was dried, but the high ability of maltodextrin to capture water may also play a role. In fact, the hygroscopicity of maltodextrin has been reported to vary between 72 and 83 g 100 g^{-1} (Ersus & Yurdagel, 2007). The solubility of the powders ranged from 67.71 to 99.71%, which is slightly higher than the value of 77–90% reported by Cano-Chauca, Stringheta, Ramos, and Cal-Vidal (2005) for powders of mango juice. The high solubility of the powders is due to the attribution of the properties of the water-soluble encapsulants that were used for drying (mainly maltodextrin) because the mixes with the highest solubilities correspond to those produced with the greatest amount of the encapsulant.

3.3.3. Determination of antioxidant activity and betalain content of the microcapsules

Pure gelatin or maltodextrin did not present antioxidant activity. However, encapsulated pigments obtained by spray drying showed antioxidant capacities with marked variations, i.e., 4 to 14 TEAC values depending on G:M ratio. Also, the encapsulation efficiencies on TEAC were from 29.71 % to 213.21 % (Table 3).

Thus, some % EE values in terms on antioxidant activity can be higher than 100% due to generation browning compounds by thermal effect (data do not shown) (Kuntcheva & Obretenov, 1996).

There were significant differences ($p < 0.05$) in the BC of the gelatin-maltodextrin encapsulates (11.33–35.93 mg betalain per

Table 3
Yield, moisture, water activity, hygroscopicity, and solubility of the powders produced.

Treatment No	Yield (g 100 g^{-1})	Moisture (g 100 g^{-1})	Water activity (ad)	Solubility (%)	Higroscopicity (g water 100 g^{-1})	Antioxidant activity (TEAC)	EE_{TEAC} (%)
1	8.74 ± 0.08^{h}	2.96 ± 0.03^{j}	0.25 ± 0.00^{d}	97.87 ± 0.85^{b}	23.28 ± 0.67^{d}	$13.43 \pm 0.91^{d,e}$	95.93
2	14.85 ± 0.22^{a}	5.01 ± 0.02^{d}	0.26 ± 0.00^{c}	94.14 ± 0.87^{d}	$22.78 \pm 0.58^{d,e}$	22.83 ± 0.77^{e}	163.07
3	7.76 ± 0.01^{l}	4.83 ± 0.06^{e}	0.21 ± 0.00^{m}	79.29 ± 0.66^{i}	$21.99 \pm 0.88^{e,f}$	4.16 ± 0.97^{k}	29.71
4	10.37 ± 0.01^{e}	3.52 ± 0.05^{h}	0.20 ± 0.00^{o}	99.71 ± 0.25^{a}	23.31 ± 0.04^{d}	10.62 ± 0.60^{f}	75.86
5	10.36 ± 0.19^{e}	6.75 ± 0.02^{b}	0.24 ± 0.00^{e}	80.23 ± 0.86^{i}	23.09 ± 0.41^{d}	$7.96 \pm 0.72^{i,j}$	56.86
6	$8.32 \pm 0.22^{i,j}$	6.95 ± 0.03^{a}	0.21 ± 0.00^{l}	91.44 ± 0.85^{e}	$22.12 \pm 0.74^{e,f}$	29.85 ± 0.38^{a}	213.21
7	$8.16 \pm 0.19^{j,k}$	5.06 ± 0.03^{d}	0.29 ± 0.00^{a}	77.73 ± 0.53^{j}	28.13 ± 0.56^{a}	$9.54 \pm 0.18^{f,g,h}$	68.14
8	9.17 ± 0.06^{g}	4.52 ± 0.02^{f}	0.24 ± 0.00^{f}	72.18 ± 0.72^{l}	25.74 ± 0.82^{c}	7.79 ± 0.93^{j}	55.64
9	$7.96 \pm 0.25^{l,k}$	5.53 ± 0.03^{c}	0.26 ± 0.00^{b}	75.81 ± 0.43^{k}	25.43 ± 0.06^{c}	$9.13 \pm 0.89^{g,h}$	65.21
10	8.90 ± 0.22^{h}	2.15 ± 0.13^{n}	0.23 ± 0.00^{h}	85.96 ± 0.27^{g}	25.52 ± 0.58^{c}	$10.09 \pm 0.59^{f,g}$	72.07
11	8.85 ± 0.06^{h}	2.64 ± 0.05^{l}	0.22 ± 0.00^{i}	85.94 ± 0.87^{g}	$26.05 \pm 0.35^{b,c}$	6.90 ± 0.57^{j}	49.29
12	11.72 ± 0.14^{d}	2.59 ± 0.02^{l}	0.20 ± 0.00^{n}	86.30 ± 0.21^{g}	26.83 ± 0.19^{b}	21.03 ± 0.68^{c}	150.21
13	8.48 ± 0.13^{i}	2.97 ± 0.07^{j}	0.23 ± 0.00^{g}	67.71 ± 0.08^{m}	21.03 ± 0.85^{g}	10.26 ± 0.98^{f}	73.29
14	10.09 ± 0.02^{f}	4.22 ± 0.02^{g}	0.20 ± 0.00^{n}	71.91 ± 0.90^{l}	28.09 ± 0.70^{a}	23.82 ± 0.39^{b}	170.14
15	$8.99 \pm 0.07^{g,h}$	1.87 ± 0.01^{o}	0.22 ± 0.00^{i}	79.37 ± 0.64^{i}	$22.09 \pm 0.66^{e,f}$	$8.96 \pm 0.65^{h,i}$	64.00
16	10.43 ± 0.04^{e}	2.26 ± 0.00^{m}	0.19 ± 0.00^{q}	83.35 ± 0.60^{h}	$22.53 \pm 0.18^{d,e,f}$	14.23 ± 0.49^{d}	101.64
17	11.48 ± 0.22^{d}	2.73 ± 0.01^{k}	0.20 ± 0.00^{p}	88.36 ± 0.97^{f}	$21.67 \pm 0.60^{f,g}$	23.34 ± 0.58^{b}	166.71
18	14.48 ± 0.13^{b}	3.27 ± 0.00^{i}	0.21 ± 0.00^{k}	86.59 ± 0.23^{g}	19.34 ± 0.29^{h}	21.46 ± 0.32^{c}	153.29
19	14.16 ± 0.13^{c}	2.33 ± 0.00^{m}	0.22 ± 0.00^{j}	95.91 ± 0.70^{c}	20.04 ± 0.53^{h}	$10.14 \pm 0.22^{f,g}$	72.43

Data represents the means ± deviation standard from nineteen tests independent with triplicate for each test. Different superscript letters in the column indicate statistical significance ($p < 0.05$) according to the Tukey least significant different test.

100 g of powder). The microcapsules with the highest pigment content were obtained at Ti = 125 °C and G:M ratio (5:5). The encapsulation efficiencies on BC were from 18.07% to 57.30 % (Table 4). There are considerable losses of betalains, which are retained in the matrix of the encapsulant, coupled with their degradation by thermal process (El-Gharras et al., 2008).

The effect of temperature on the pigment content in the microcapsules is shown in Fig. 1. A higher content of betalains corresponds to structures formed at a G:M ratio of 5:5 and a drying temperature in the range from 117 to 125 °C but the highest antioxidant capacities were obtained with a G:M ratio of 2.5:7.5, and the drying temperature does not markedly influence the antioxidant capacity.

The maximum antioxidant capacities did not correlate with the maximum pigment concentrations (Figs. 1 and 2). As was reported by Butera et al. (2002), Pavlov, Kovatcheva, Georgiev, Koleva, and Ilieva (2002), and Cai, Sun, and Corke (2003), the presence of betalamic acid (an uncolored precursor molecule of all betalains) with hydroxyl radicals and other hydrogen-donating groups, such as NH groups, enable this acid to act as an antioxidant agent; however, if the acid molecule no presented these hydroxyl groups, the betalamic acid does not present antioxidant capacity (Miller, Sampson, Candeias, Bramley, & Rice-Evans, 1996; Villaño, Fernández-Pachón, Troncoso, & García-Parrilla, 2005). Furthermore, it is known that sugars and proteins (gelatin) at high temperature can generate Maillard products known as melanoidins, which have high antioxidant activity (Wang, Qian, & Yao, 2011), and this antioxidant activity can be observed despite the presence of betalain. However, the mixing of sugars (from maltodextrin or clarified juice) and nitrogenous components (from gelatin or betalains) may result in a reaction between the carbonyl group of the reducing sugar and the amino group of nitrogenous components at temperatures of at least 95 °C. The melanoidins obtained via this route are considered to have a high molecular weight. It has been shown that there is a positive correlation between antioxidant activity and the molecular weight and color intensity of melanoidin and the drying temperature (Wang et al., 2011).

3.3.4. Color measurement

The results of the color measurement of the powders are presented in Table 4. The microcapsules showed values of 17.26–40.90 and 6.26 to 15.46 for the a* and b* parameters, which are the characteristic colors of betalains from juice. This finding indicates that the pigment is incorporated in the matrix used (Gharsallaoui, Roudaut, Chambin, Voilley, & Saurel, 2007). The h° values ranged

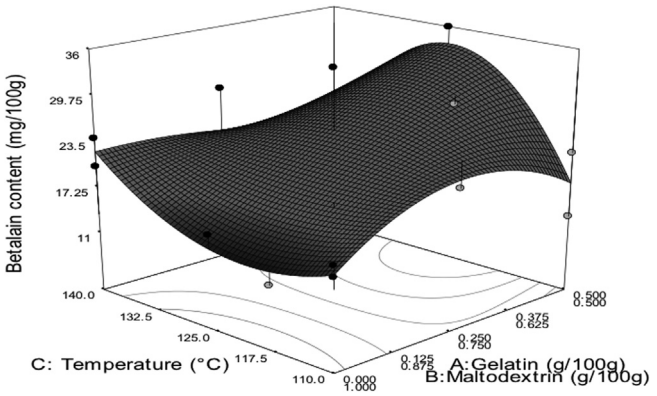

Fig. 1. Response surface analysis for determination of pigment contents of the powders.

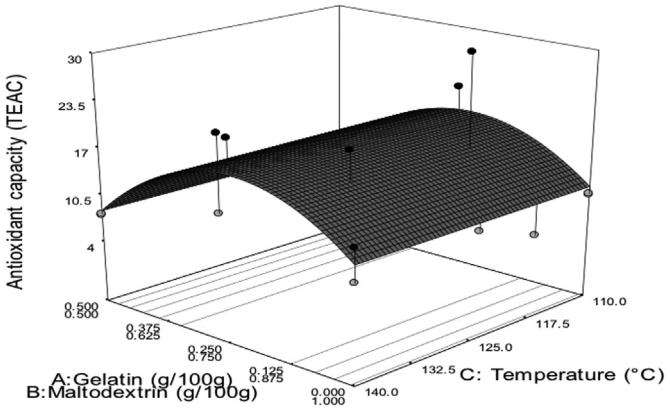

Fig. 2. Response surface analysis for determination of antioxidant capacity of the powders.

Table 4
Colorimetry, betalain content, and antioxidant capacity of the powders produced.

No.	Colorimetry.					Betalains content (mg 100 g^{-1})	EE_{BC} (%)
	Lightness (L)	a*	b*	Chroma (C)	Hue angle (h°)		
1	$27.00 \pm 0.85^{c,d}$	$26.16 \pm 0.97^{d,e,f}$	15.46 ± 0.75^{a}	$31.26 \pm 0.84^{c,d,e}$	$15.74 \pm 1.39^{e,f}$	24.07 ± 0.55^{d}	38.38
2	$23.43 \pm 1.10^{e,f}$	$32.30 \pm 2.65^{b,c}$	$9.06 \pm 0.15^{f,g,h}$	$33.55 \pm 2.52^{b,c,d}$	29.66 ± 1.65^{a}	33.56 ± 0.95^{b}	53.52
3	$30.26 \pm 3.96^{b,c}$	$28.16 \pm 2.98^{c,d,e}$	$10.06 \pm 1.56^{e,f,g}$	$29.91 \pm 3.33^{d,e,f}$	$19.60 \pm 0.98^{c,d,e,f,}$	28.68 ± 0.61^{c}	45.74
4	28.86 ± 1.40^{c}	$26.30 \pm 3.36^{d,e,f}$	$10.33 \pm 1.02^{d,e,f}$	$28.29 \pm 2.98^{e,f,g}$	$21.68 \pm 3.80^{c,d}$	$19.21 \pm 0.97^{g,h,i}$	30.63
5	$27.50 \pm 1.66^{c,d}$	$23.56 \pm 3.42^{e,f,g}$	$8.00 \pm 0.10^{g,h,i}$	$24.96 \pm 3.25^{g,h,i}$	$18.75 \pm 5.59^{c,d,e,f}$	12.60 ± 0.70^{m}	20.09
6	17.16 ± 0.30^{h}	$19.63 \pm 0.85^{g,h}$	6.26 ± 0.92^{i}	$20.62 \pm 0.73^{i,j}$	$17.72 \pm 2.85^{d,e,f}$	$18.39 \pm 0.92^{h,i,j}$	29.33
7	$20.70 \pm 1.80^{g,f}$	17.26 ± 3.45^{h}	$7.26 \pm 0.92^{h,i}$	18.76 ± 3.31^{j}	$23.17 \pm 3.89^{b,c}$	$13.23 \pm 0.81^{l,m}$	21.10
8	16.23 ± 1.43^{h}	$20.36 \pm 4.55^{g,h}$	$7.26 \pm 1.55^{h,i}$	$21.74 \pm 3.94^{i,j}$	$20.38 \pm 7.14^{c,d,e,f}$	$22.69 \pm 0.98^{d,e}$	36.18
9	$19.43 \pm 1.25^{g,h}$	$26.30 \pm 1.60^{d,e,f}$	$7.26 \pm 0.35^{h,i}$	$27.28 \pm 1.56^{e,f,g,h}$	15.47 ± 1.07^{f}	$22.08 \pm 0.80^{d,e,f}$	35.21
10	$18.63 \pm 0.32^{g,h}$	$28.26 \pm 0.28^{c,d}$	$9.66 \pm 0.50^{f,g}$	$29.87 \pm 0.42^{d,e,f}$	$15.05 \pm 2.13^{c,d,e,f}$	$16.57 \pm 0.79^{j,k}$	26.42
11	$18.70 \pm 1.66^{g,h}$	$27.23 \pm 3.40^{d,e,f}$	$7.26 \pm 0.65^{h,i}$	$28.19 \pm 3.31^{e,f,g}$	18.87 ± 0.75^{f}	11.33 ± 0.39^{m}	18.07
12	$27.13 \pm 4.55^{c,d}$	$29.03 \pm 4.44^{c,d}$	$10.63 \pm 2.89^{d,e,f}$	$30.95 \pm 4.96^{d,e,f}$	$15.05 \pm 2.13^{c,d,e,f}$	$16.88 \pm 0.96^{i,j,k}$	26.92
13	$23.26 \pm 0.55^{e,f}$	35.96 ± 1.60^{b}	$11.93 \pm 0.49^{c,d,e}$	37.89 ± 1.67^{b}	$18.35 \pm 0.20^{c,d,e,f}$	$15.08 \pm 0.91^{k,l}$	24.05
14	$24.33 \pm 1.82^{d,e}$	$21.53 \pm 2.51^{g,h}$	$9.00 \pm 0.30^{f,g,h}$	$23.35 \pm 2.36^{h,i,j}$	$22.82 \pm 2.16^{b,c,d}$	$16.63 \pm 0.90^{j,k}$	26.52
15	$22.90 \pm 2.17^{e,f}$	40.9 ± 2.77^{a}	$14.2 \pm 1.30^{a,b}$	43.29 ± 3.04^{a}	$19.12 \pm 0.42^{c,d,e,f}$	$20.20 \pm 0.66^{f,g,h}$	32.21
16	37.20 ± 3.65^{a}	$32.76 \pm 2.75^{b,c}$	$13.40 \pm 1.99^{a,b,c}$	$35.40 \pm 3.28^{b,c}$	$22.16 \pm 1.46^{c,d}$	35.93 ± 0.55^{a}	57.30
17	$30.06 \pm 2.47^{b,c}$	$23.33 \pm 2.37^{f,g}$	$12.40 \pm 1.58^{b,c,d}$	$26.42 \pm 2.78^{f,g,h}$	$27.96 \pm 1.35^{a,b}$	27.34 ± 0.62^{c}	43.60
18	33.43 ± 0.80^{b}	$22.70 \pm 3.56^{f,g}$	$9.30 \pm 1.73^{f,g,h}$	$24.64 \pm 2.73^{g,h,i}$	$22.75 \pm 6.52^{b,c,d}$	$21.60 \pm 0.90^{e,f,g}$	34.44
19	$27.43 \pm 0.97^{c,d}$	$32.60 \pm 0.60^{b,c}$	$12.40 \pm 0.65^{b,c,d}$	$34.88 \pm 0.78^{b,c}$	$20.81 \pm 0.71^{c,d,e}$	$15.45 \pm 0.74^{k,l}$	26.64

Data represents the means ± deviation standard from nineteen tests independent with triplicate for each test. Different superscript letters in the column indicate statistical significance ($p < 0.05$) according to the Tukey least significant different test.

from 15.06 to 29.66, which indicates that the microcapsules of betalains exhibit a redness grade, regardless of the treatment. The C values in the powders ranged from 18.76 to 43.29, which demonstrates that the microcapsules present very bright colors, and these values are associated with their L values, which ranged from 17.16 to 37.20 (Mapari et al., 2006). The microcapsules of clarified juice rich in betalains may be used as an ingredient in formulated aqueous foods because a bright color is a desirable attribute for these powders.

Moreover, it can be observed that there is a direct correlation (0.6486, $p < 0.0001$) between higher values of h° and the highest concentration of pigments found in the microcapsules, and this correlation was also observed in the mixtures containing higher ratios of gelatin (G:M ratios of 2.5:7.5, 3.75:6.25, and 5:5). Thus, this factor can also increase the use of this carrier for the encapsulation of food components.

3.3.5. Scanning electron microscopy (SEM)

Fig. 3 shows SEM micrograph at a magnification of 2000× of microencapsulates with G:M ratio (2.5:7.5). The particles are spherical in shape with inhomogeneous morphologies and exhibit multiple sizes ranging from 1.15 to 11.09 μm (mean = 6.14μm ± 4.96). In addition, the samples present a higher apparent surface collapsed based on the associated volume lost to the evaporation of water. This feature is dependent primarily on the surfaces dried at a low speed, as determined by the low operating temperatures (Tonon et al., 2008). An increase in the speed of the driving force allows the rapid removal of water, leading to a reduction in the surface damage of the microcapsules, as demonstrated with the samples dried at a higher temperature (León et al., 2010).

The particles showed an agglomerated distribution, which is mainly attributed to a higher concentration of gelatin in the mixtures based on the results presented by Díaz et al. (2006) and Saénz et al. (2009). One of the great advantages that were observed with the use of gelatin-maltodextrin mixtures as an encapsulant is that it results in more rigid structures with no ruptures in the microcapsules. In contrast, the use of only maltodextrin and gelatin is more likely to result in weak and partially fractured structures (Bruschi et al., 2003; Krishnan, Bhosale, & Singhal, 2005). However, even with these disadvantages, maltodextrin has been more used as encapsulation support of several bioactive compounds such as anthocyanins and polyphenols because it has been presented the best pigment protection and also offer better protection for phenolics during storage (Bakowska-Barczak & Kolodziejczyk, 2011; Tonon et al., 2008).

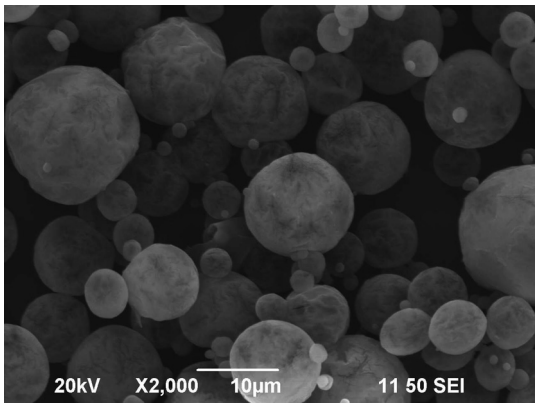

Fig. 3. Micrograph of powder obtained with G:M ratio of 2.5:7.5.

3.3.6. Differential scanning calorimetry (DSC)

The characterization of the microcapsules included differential scanning calorimetry analysis, which provides information on the transition temperature of the structure (i.e., melting point) and the instability of solid structures due to an increase in the molecular disorder of the molecule. The composites with G:M ratios of 2.5:7.5, 3.75:6.25, and 5:5 ratio had melting temperatures ranging from 205 to 235 °C (Fig. 4), which demonstrates a similar thermal behavior to their base components. This finding indicates that the clarified juice was well encapsulated within the matrix (Sansone, Picerno, et al., 2011). The melting temperatures of the base materials were the following: M, 174.7 °C; G, 88.74 °C (Fig. 4). The analysis of the thermograms revealed that the mixtures had higher melting points likely because of the structures were more rigid systems, which also provides stability and protection to the bioactive component (Sansone, Picerno, et al., 2011).

It has been reported that encapsulated nutraceutical extracts using mixes of biopolymers (Maltodextrin-pectin) with a maximum concentration of 10% have a melting point of 219.31 °C (Sansone, Mencherini, et al., 2011), which is markedly lower that the result obtained in our study for the mixtures with G:M ratios of 5:5 and 2.5:7.5 (melting temperatures of 220.78 and 235.44 °C, respectively). Hence, the use of the maltodextrin-gelatin complex generate directly microcapsules with better stability which provides indirectly a good protection to the bioactive components, but there are other polysaccharides that provide wider temperature intervals, as reported by Kalogeropoulos, Yannakopoulou, Gioxari, Chiou, and Makris (2010), who studied microencapsulated flavonoids and obtained a β-cyclodextrin complex with a melting temperature up to 300 °C, and Sansone, Picerno, et al. (2011), who used carboxymethylcellulose to stabilize flavonoids and obtained a melting temperature in the range of 280–300 °C.

4. Conclusions

The carriers proposed in this study showed good performance for microencapsulated food components, such as betalains, from clarified purple cactus pear juice through an ultrafiltration process. The encapsulation yield and the properties of the microcapsules were found to depend on the ratio of gelatin-to-maltodextrin used, although low yields were generally obtained. In all cases, powders with a red color were obtained, which suggests that the pigment was embedded into the matrix. The formed composite had a low

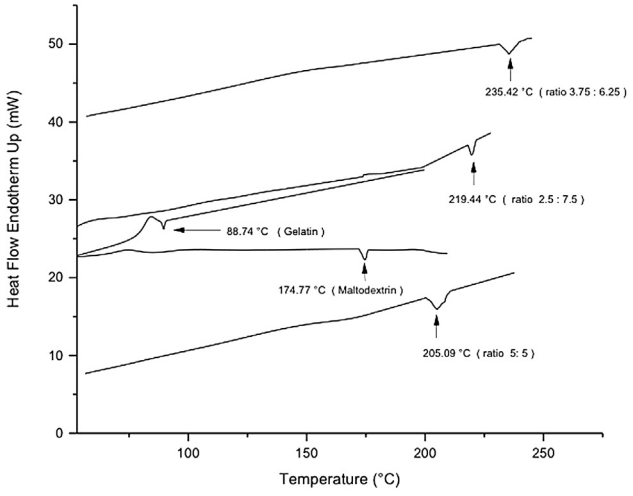

Fig. 4. Thermograms of gelatin and maltodextrin powders. The microencapsulated juice (G:M) ratios were 2.5:7.5, 3.75:6.25, and 5:5.

moisture content, low water activities, excellent solubility, and high hygroscopicity. The microstructures generated are rigid structures, which is attributed to the presence of gelatin. The betalain content depends on both the temperature and the encapsulant ratio, but the antioxidant activity depends only on the ratio of the carriers. The selection of the best ratio of carriers is a difficult task, but the best ratio G:M in this study was 2.5:7.5 due to the higher yield, maximal colorimetry values related to the betalain content, and good antioxidant activity. Moreover, the gelatin-maltodextrin composite is recommended for the encapsulation of food ingredients because it can be used for components with low stability, such as betalains.

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