Physical properties of spray dried pitaya

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ABSTRACT. Studies of fruits obtained from a wide variety of cactus have revealed bio-active compounds with a high antioxidant capacity. Encapsulation methods have been used to conserve betalains, the principle bio-active compounds in the juice, which is highly susceptible to degradation in a fresh state. The encapsulation conditions (temperature and encapsulant matrix) affect the physical properties of the dry powders, which in turn influence the retention capacity of bio-active compounds. The objective of this work is to evaluate the effect of the spray dried encapsulation conditions on the following physical properties: color, glass transition temperature (Tg) and adsorption isotherm. The color was determined using a spectrocolorimeter with a L,a*,b* color scale, Tg was calculated using a DSC Q2000 and the adsorption isotherms, at 25 °C, were measured with a VTI-SA$^+$. The experimental data was fitted to the GAB (Guggenheim-Anderson-DeBoer) using non-linear regression. Micrographs of the structure were analyzed. Pitaya fruit (Stenocereus griseus) were selected, washed and pealed. The juice was obtained with manual extrusion and filtered through a metallic sieve. The encapsulation method was spray drying. A 2^2 factorial design was used to evaluate the effect of the independent variables: inlet air temperature (150–180 ◦C) and feed flow rate (1.635 –2.148 L/h) on the powder properties. The atomization speed was kept constant (28,000 rpm). A maltodextrin/pectin combination was used as the encapsulant matrix in a 40/20% ratio based on the total solids concentration. The highest variation was found in the parameter b*, which represents changes of the color yellow. This change is associated with the loss of betaxanthin, a highly thermolabile compound. A type III adsorption isotherm was found. The constants of the GAB equation were determined with a fit of $R^{2\geq}$0.99. Tg between -26.21 y 32.68°C were found for water activity in the range of 0.11 and 0.53. A test of SEM micrographs shows polydisperse powder particles. These particles were observed to be sphere-like with some sections exhibiting compaction of agglomerated particles. The application of different encapsulation conditions and encapsulant matrix ratio no affect significantly the physical properties of pitaya juice powder. A humidity content ≤ 10% and water activity of 0.4 are required to maintain the stability of the powder at room temperature.*

KEYWORDS:Physical properties, Spray drying, Pitaya, Glass transition temperature, Water activity.

1 Introduction

Pitaya is a nutrient-rich fruit from the cactus family which has generated interest for its potential health benefits. Betalains, the principal bioactive compound found in pitayas, are water soluble pigments commonly used as colorants with a wide range of desirable biological activities, including antioxidant, anti-inflammatory, hepatoprotective and anti-cancer properties (Georgiev *et al.* 2010).

Factors such as pH, water activity, light exposure, oxygen, temperature and enzymatic activity all affect betalain stability (Castellar *et al.* 2003). In order to prolong shelf life and improve the stability of these thermo-sensitive compounds, microencapsulation methods with edible matrices, such as biopolymers, have been used (Desai and Park 2005).

Encapsulation conditions, the type of atomizer nozzle, concentration of solids and the type of matrix, all affect the physical properties of powders. Structure, state of material (crystalline or amorphous), size, uniformity and form of the dry particles are some of the properties that influence the retention capacity of bioactive compounds. Moreover, other aspects such as the efficiency of the separation/collection of the cyclones depend on properties such as density. During the drying process, the particle is generally in an amorphous state and, if it has high-sugar levels, is very hygroscopic and is eventually recrystallized (Fitzpatrick 2005). This results in color changes and the loss of the bioactive compounds of interest. Consequently, it is very important to establish the storage conditions required to maintain the stability of the dry powders.

Spray drying is the most commonly used encapsulation method for juices obtained from fruit of the *cactaceae* family. The primary encapsulating material is maltodextrin. In studies of powders from red pitaya (*Hylocereus polyrhizus*) with 20 and 30% of maltodextrin, it was found that the powders exhibit low solubility, with moisture contents below 10%, bulk density between 290.806 and 345.467 kg/cm^3, particle size between 6.891 and 9.478 μm and agglomerate structure. Both powders irrespective of maltodextrin concentrations showed high glass transition temperature (70.49 and 74.40º C) (Tze *et al.* 2012). With the same species, Che *et al.* (2011) reported moisture contents between 3.32 and 7.12 % d.b. and a bulk density from 0.325 to 0.413 g/mL.

In powders obtained from pear fruit juice (*Opuntia ficus-indica*), Saénz *et al.* (2009) found irregularly shaped particles with an extensively dented surface, while Gandía-Herrero*et al.* (2010) found particles with a shrink form. In other varieties of cactus pear fruits (*Opuntia lasiacantha and Opuntia streptacantha*), particle size was from 5 to 50 μm, showed agglomerate structure (Sanchéz *et al.* 2006). The powders had a moisture content between 3.97 and 6.41 % d.b. with a hygroscopicity of 36.30 and 48.93 g of water/ 100 g dry solid (Rodriguez- Hernández *et al.* 2005).

In cactus pear fruits (*Opuntia stricta*) encapsulated in glucose syrup, bulk density was between 0.52 and 0.60 g/mL and particle size from 2 to 12 μm with a spherical form (Obón *et al.* 2009).

Numerous factors influence the physical properties of powders obtained from the spray drying encapsulation process: encapsulation conditions, concentration and type of encapsulant matrices, concentration of solids, chemical composition of the juices, inlet gas direction of flow, temperature and the degree and uniformity of atomization (Masters 1991, Moyers 1997). Variations among the varieties and genotypes of cactus fruits also influence some of these factors.

The objective of this work is to evaluate the effect of pitaya juice (*Stenocereus griseus*) spray-drying encapsulation on physical properties: color, bulk density, glass transition temperature and sorption isotherm.

2 Materials and methods

2.1. Material

Pitayas of the genus *Stenocereus griseus* were obtained from Huitziltepec (18º 46' north latitude, 97º 52' west longitude) in Puebla, Mexico. The fruit was stored at -20°C until analysis. Maltodextrin (13 DE) and pectin (Cytecsa, Mexico) were used as the encapsulant matrix.

2.2. Sample preparation

The chosen pitayas were washed and pealed. The pulp was manually extracted and filtered through a metallic sieve (Standard US. 100) to obtain the juice. A maltodextrin/pectin combination was used as the encapsulant matrix in a 40/20% ratio, calculated as a function of the total solids concentration. The juice and encapsulant matrix were mixed together for 5 min. using an electric homogenizer. Agitation was maintained for 1 h with a magnetic plate.

2.3. Spray drying

A co-current pilot-scale spray dryer (Niro, Copenhagen, Denmark) equipped with rotary atomizer (TS-Minor, M02/A) was used for all samples. Distilled water at room temperature (25 ºC) was used for start-up and shut down operations. The feed flow rate (Fe) was controlled by a peristaltic pump (Watson-Marlow 505S/RL). A 2^2 factorial design was used to evaluated the effect of independent variables: inlet air temperature, Ti (150-180 ºC) and feed flow rate, Fe (1.635-2.148 Lh^{-1}); the atomization speed (As) was kept constant (28,000 rpm). The homogenized mixture was maintained under slow agitation during spray drying. The powders were stored in polyurethane bags until analysis.

2.4 Physical Properties of pitaya juice powder

2.4.1 Color

A spectrocolorimeter HunterLab LabScan XE (Hunter Associate Laboratory Inc., Reston, USA) with illuminant D65, a standard white calibrated platewith reflectance values of X=80.37, Y=85.32, Z=90.64 was used. The results were reported in the coordinates CIE L* a* b*.

2.4.2 Moisture content

Moisture content was determined using the gravimetric technique described in the A.O.A.C. (1984); moisture loss was expressed in percentage dry base (db).

2.4.3 Glass transition temperature

The powders were equilibrated in desiccators with different saturated salt solutions of LiCl, $MgCl_2.6H_2O$, K_2CO_3 y $Mg(NO_3)_2$to maintain water activities (a_w) between 0.11 y 0.53 at 25 ºC. The glass transition temperature (Tg) was determined with a differential scanning calorimeter model Q2000 (TA Instruments, New Castle, USA). The samples were placed in aluminum pans and hermetically sealed. An empty pan was used as a reference. The sample was equilibrated at -40 °C for 10 min and then heated at a rate of 2 °C/min at 120 ° C. All analyses were done in duplicate and Tg was calculated using the Universal Analysis 2000 software (TA Instruments, New Castle, USA).

2.4.4 Sorption isotherm

Sorption isotherms were determined with a Vapor Sorption Analysis VTI-SA$^+$ (TA Instruments, New Castle, DE, USA) with a a_w range of 0.10 to 0.80 at 25 °C. The experimental data was adjusted to the GAB model (eq. 1) using non-linear regression.

$$X_{eq} = \frac{X_m C K a_w}{(1 - K a_w)(1 - K a_w + C K a_w)} \quad (1)$$

where X_{eq} is the equilibrium moisture content (g H_2O/g dry mater), X_m is the monolayer moisture content (g H_2O/g dry matter), C and K are constants related to temperate effects and a_w is water activity.

2.4.5 Bulk density

The bulk density of the powder was measured by weighing 2 g of the sample and placing it into a 10 mL graduated cylinder. This was tapped 10 times onto a rubber mat from a height of 10 cm. The volumewasthen recorded and used to calculate bulk density as g/mL (Chegini and Ghobadian 2007, Leon-Martinez *et al.* 2010,Yousefi *et al.* 2011)

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2.4.6 Particle morphology of powders

Particle shape and surface morphology of powderwas evaluated using a Nova NanoSEM 200 (FEI, Oregon, USA) scanning electron microscope operated at an accelerating voltage of 5 kV. The sample was covered with platinum. The coating was carried out in a sputter coater 208 HR (Cressington Scientific Instruments. England, UK) and analyzed at a magnification of 5000x.

2.5 Statistical analysis

Allexperimentswereconducted in duplicate and the resulting data was analyzed using variance analysis (ANOVA). The average and standard deviation of the values are presented.

3 Results and Discussion

3.1.*Color*

The color parameters of the powders are shown in Table 1. No significant differences were found between treatments for any of the parameters. The b* value of the P2 treatment shows a tendency towards the yellow tone. The temperature, along with other factors, can provoke the degradation of the betalins' chemical composition. Betanin, at high temperatures, can undergo 4 reactions that affects its structure and physical properties: 1) Hydrolytic cleavage, when hydrolysis occurs in the imine bond, producing yellow betalamic acid and the colorless cyclo-Dopa 5-O-β-glucoside, 2) Decarboxylation, in which the loss of carboxylic acid from carbon 15 or 17 of the betalains' structure provokes an orange color, 3) Dehydrogenation, which implies the loss of a hydrogen from carbon 15, along with a yellow color change, 4) Isomerization, which changes the spatial direction of atoms united to carbon 15, an asymmetric carbon (Herbach *et al.* 2004, 2006).

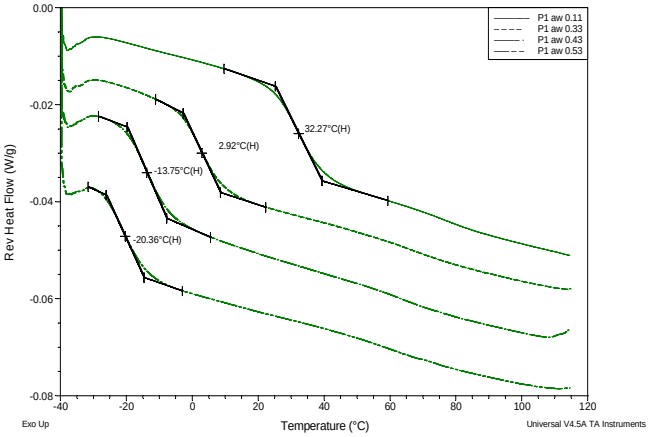
3.2.*Moisture content*

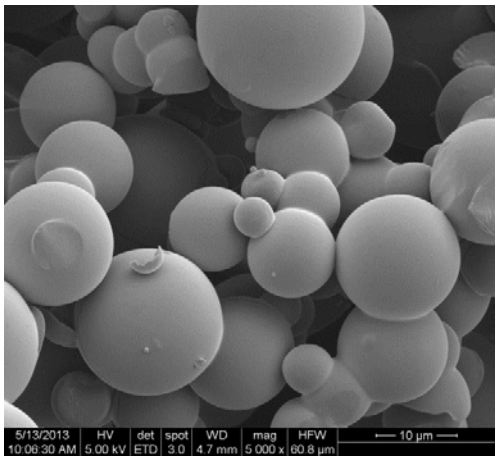
The moisture content of the powders was less than 10% (Table 1), which assures product stability. The P2 treatment resulted in the lowest moisture content. This treatment combines high inlet air

temperature (Ti) with a low feed flow rate (Fe). This is attributed to the fact that with a higher inlet air temperature, there is a greater temperature gradient between the atomized feed and the drying air, resulting in a greater driving force for water evaporation (Tonon *et al*. 2011, Leon-Martinez *et al.* 2010, Quek *et al.* 2007).

Table 1. Physical properties of pitaya juice powder

Treat-ment	Ti (ºC)	Fe (Lh^{-1})	L*	a*	b*	Moisture content (% db)	Bulk density (g/mL)
P1	150	1.635	36.52 ± 0.27	46.43 ±0.22	13.26 ± 0.14	4.601	0.581 ± 0.011
P2	180	1.635	34.80 ± 0.14	42.72 ± 0.05	23.16 ± 0.24	3.106	0.580 ±0.011
P3	150	2.148	36.46 ± 0.23	47.20 ± 0.23	13.46 ± 0.12	4.885	0.608 ± 0.027
P4	180	2.148	35.48 ± 0.64	46.87 ± 0.43	12.69 ± 0.34	3.506	0.581 ± 0.011





a b

Fig 1a). DSC profile for pitaya juice powder to different a_w. and b) SEM image of particle morphology of pitaya juice powder

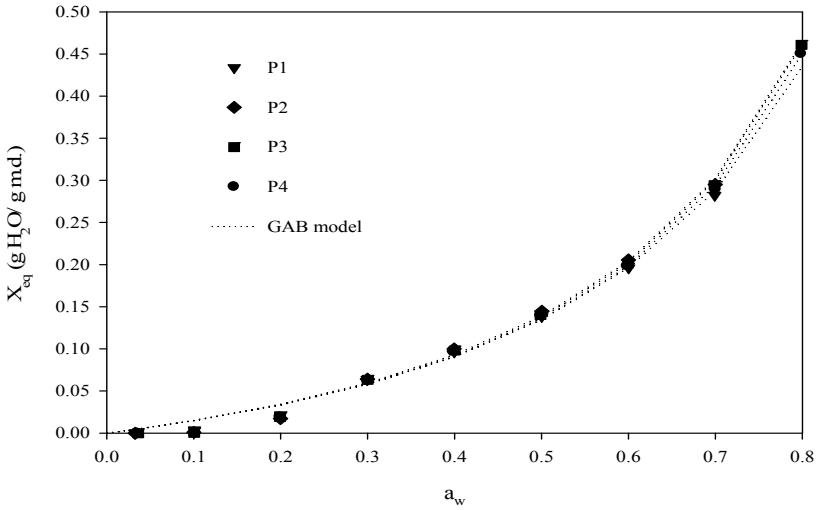


Fig. 2 .Sorption isotherms of pitaya juice powders

3.3. *Glass transition temperature*

Figure 1a shows that DSC themogram of the P1 treatment submitted to different a_w. Similar curves were obtained for all the other samples conditioned at different a_w. Table 2 shows the Tg obtained for each treatment stored at different a_w. As a_w increases Tg decreases. When a_w is close to 0.11, the Tg is higher than the room temperature at which food products are typically stored. In addition, an a_w higher than 0.33 can result in the rapid loss of betalains. When Tg is less than room temperature, the powders go from an amorphous state to a rubbery state, exposing the bioactive compounds to light, moisture, oxygen and metallic compounds that lead to betalain degradation. Overtime, the polymer recrystalization process could appear, not only in the encapsulating materials, but also in the juices, resulting in further betalain loss.

Table 2. Glass transition temperature of pitaya juice powders at different a_w

a_w	Glass transition temperature (ºC)			
	P1	P2	P3	P4
0.11	32.24 ± 0.05	29.22 ± 0.24	31.53 ± 1.32	32.68 ± 0.03
0.33	3.19 ± 0.33	4.14 ± 1.07	-2.39 ± 0.39	3.93 ± 1.17
0.43	-13.59 ± 0.45	-11.28 ± 0.71	-16.32 ± 0.28	-12.45 ± 0.78
0.53	-20.80 ± 1.01	-19.24 ± 0.55	-26.21 ± 0.54	-25.59 ± 0.28

3.4. *Sorption isotherms*

The sorption isotherms (Fig. 2) exhibit type III behavior according to Brunauer's classification (Brunauer *et al.* 1940). These results confirm the limit of a_w where powder the instability is first noted. The same limit was identified in the Tg analysis. In this graph a change in isotherm ($a_w > 0.2$) behavior can be seen where the change in state of material is. However, the moisture content remains less than 0.1 g H_2O/g dry mater until a_w reaches 0.4, at which point the moisture content increases drastically, suggesting that the material hydrates quickly. The GAB parameters of the sorption isotherms are shown in Table 3.

Table 3. Sorption parameters for GAB model

Parameters	Treatment			
	P1	P2	P3	P4
X_m	0.210	0.221	0.242	0.221
C	0.732	0.690	0.611	0.671
K	0.871	0.880	0.868	0.876
R^2	0.997	0.997	0.997	0.996

3.5. Bulk density

The bulk density of the powders varied between 0.580 and 0.608 g/mL (Table 1). No significant differences were found between treatments. The recorded values fell in the range of values reported for similar materials. Fazaeli *et al.* (2012), Kha *et al.* (2010) and Bhandari *et al.*(1993) reported values of 0.40-0.52 g/mL, 0.66-0.78 g/mL, 0.53-0.74 g/mL for black mulberry juice, gac fruit juice and concentrated fruit juices respectively.

3.6. Morphology

Figure 1b shows the micrograph of powder obtained with the P3 treatment. The particles are spherical, well-defined and of varying sizes, which is very common in spray-dried powders. The

natural spherical shape of the particles suggests that droplets dried symmetrically. This morphological characteristic is exhibited by organic materials and foodstuffs containing glucose-like or glucose-based carbohydrates (García-Cruz *et al.* 2012). The particles are agglomerated; some authors have suggested that agglomeration occurs because of static electrical effects and Van der Waals forces (Leon-Martinez *et al.* 2010, García-Cruz *et al.* 2012).

4 Conclusions

Although no significant differences were found between treatments, the P4 treatment best maintained color, bulk density and Tg. Sorption isotherms were well fitted by GAB, with monolayer moisture contents between 0.21 and 0.24 g H_2O/g dry mater. The constants that are best adapted to the GAB model were identified. The morphology shows excellent characteristics; the structure, free of cracks and dents, assures that the encapsulating bioactive compounds remain protected from degradation factors.

Acknowledegments

The authors would like to thank CONACYT, SIP, and the COFAA of the Instituto Politécnico Nacional for the facilities provided over the course of this research (project SIP-20130767) and CONACYT for their generous funding (268716).

5 References

Bhandari B.R, Senoussi A., Dumoulin E.D., and Lebert A.1993 – Spray drying of concentrated fruit juices. Drying Technology, 11(5): 1081-1092.

Brunauer S., Deming L.S., Deming W.E. and Teller E.1940 – On a theory of the van der Waals adsorption of gases. Journal of the American Chemical Society, 62(7): 1723-1732.

Castellar R., Obon J.M., Alacid M. and Fernández-López J.A. 2003 – Color properties and stability of betacyanins from Opuntia fruits. Journal of Agricultural and Food Chemistry, 51(9): 2772–2776.

Che Yunus M.A., Lee C.Y. and Idham Z.2011 – Effects of variables on the production of red-fleshed pitaya powder using response surface methodology. Jurnal Teknologi, 56(1): 15-29.

Chegini G.R. and GhobadianB.2007 – Spray dryer parameters for fruits juice drying. World Journal of Agricultural Sciences, 3(2): 230-236.

Desai K.G.H. and Jin Park H. 2005 – Recent developments in microencapsulation of food ingredients. Drying Technology, 23(7): 1361–1394.

Fazaeli M., Emam-Djomeh Z., Kalbasi A. A. and Omid M. 2012 – Effect of spray drying conditions and feed composition on the physical properties of black mulberry juice powder. Food and Bioproducts Processing, 90(4): 667-675.

Fitzpatrick J.J.2005 – Food powder flowability. In Onwulata C. Encapsulated and powdered foods. Boca Raton: Marcel Dekker Publishers, pp. 247-260.

Gandía-Herrero F., Jiménez-Atiénzar M., Cabanes J., García-Carmona F. and Escribano J. 2010 – Stabilization of the bioactive pigment of opuntia fruits through maltodextrin encapsulation. Journal of agricultural and food chemistry, 58(19): 10646-10652.

García-Cruz E.E., Rodríguez-Ramírez J., Lagunas L.L. and Medina-Torres L. 2012–Rheological and physical properties of spray-dried mucilage obtained from *Hylocereus undatus* cladodes. Carbohydrate polymers. 91: 394-402.

Georgiev V.G., Weber J., Kneschke E.M., Denev P.N., Bley T. and Pavlov, A.I. 2010– Antioxidant activity and phenolic content of betalain extracts from intact plants and hairy root cultures of the red beetroot Beta vulgaris cv. Detroit Dark Red. Plant Foods for Human Nutrition, 65(2): 105-111.

Herbach K.M., Stintzing F.C. and Carle, R. 2004 – Thermal degradation of betacyanins in juices from purple pitaya [*Hylocereus polyrhizus* (Weber) Britton & Rose] monitored by high-performance liquid chromatography–tandem mass spectometric analyses .European Food Research and Technology, 219(4): 377-385.

Herbach K.M., Stintzing F.C. and Carle R. 2006–Betalain stability and degradation—structural and chromatic aspects.Journal of Food Science,71(4): R41-R50.

Kha T.C., Nguyen M.H. and Roach, P. D. 2010– Effects of spray drying conditions on the physicochemical and antioxidant properties of the Gac (*Momordica cochinchinensis*) fruit aril powder. Journal of Food Engineering, 98(3): 385-392.

León-Martínez F.M., Méndez-Lagunas L.L and Rodríguez-Ramírez J. 2010 – Spray drying of nopal mucilage (Opuntia ficus-indica): Effects on powder properties and characterization. Carbohydrate Polymers, 81(4): 864–870.

Obón J.M., Castellar M.R., Alacid M. and Fernández-López J. A.2009–Production of red-purple food colorant from Opuntia stricta fruits by spray drying and its application in food model system. Journal of Food Engineering,90(4): 471-479.

Masters K. 1991– Spray Drying Handbook, Longman Scientific and Technical, Essex, England, (Ed. 5).

Moyers C.G. 1997 – Spray Dryers, Perry's Chemical Engineers Handbook, McGraw-Hill Book Co., (Ed. 7), 12-81 to 12-90.

Quek S.Y., Chok N.K. and Swedlund P. 2007 – The physicochemical properties of spray-dried watermelon powders Chemical Engineering and Processing: Process Intensification, 46(5): 386-392.

Rodríguez-Hernández G.R., González-García R., Grajales-Lagunés A., Ruiz-Cabrera M. A. and Abud-Archila M. 2005 –Spray-Drying of Cactus Pear Juice (Opuntia streptacantha): Effect on the physicochemical properties of powder and reconstituted product. Drying Technology, 23(4): 955–973.

Sánchez F.D., López E.M.S., Kerstupp S.F., Ibarra R.V. and Scheinvar L. 2006– Colorant extraction from red prickly pear (Opuntia lasiacantha) for food application. Journal of Environment Agricultural. Food Chemistry, 5: 1330-1337.

Tonon R.V., Freitas S.S. and Hubinger M.D. 2011– Spray drying of açai (*Euterpe oleraceae* mart.) juice: Effect of inlet air temperature and type of carrier agent. Journal of Food Processing and Preservation, 35(5): 691-700.

Tze N.L., Han C.P., Yusof Y.A., Ling C.N., Talib R.A., Taip F.S. and Aziz M.G. 2012– Physicochemical and nutritional properties of spray-dried pitaya fruit powder as natural colorant. Food Science and Biotechnology, 21(3): 675-682.

Yousefi S., Emam-Djomeh Z. And Mousavi S.M. 2011– Effect of carrier type and spray drying on the physicochemical properties of powdered and reconstituted pomegranate juice (*Punica Granatum* L.). Journal of food science and technology, 48(6): 677-684.