

Revista Mexicana de Ingeniería Química

Vol. 14, No. 3 (2015) 579-588



STABILIZATION OF PHENOLIC COMPOUNDS FROM Opuntia oligacantha Först BY MICROENCAPSULATION WITH AGAVE SAP (AGUAMIEL)

ESTABILIZACIÓN DE COMPUESTOS FENÓLICOS DE Opuntia oligacantha Först POR MICROENCAPSULACIÓN CON AGAVE SAP (AGUAMIEL)

C. Pérez-Alonso¹, R.G. Campos-Montiel², E. Morales-Luna², A. Reyes-Munguía³, G. Aguirre-Álvarez², D.J. Pimentel-González^{2,4}*

Received December 8, 2014; Accepted August 28, 2015

Abstract

The aim of this research was to determine the stability of phenolic compounds from *Opuntia oligacantha* Först (xoconostle) by microencapsulation with a blend of biopolymers (maltodextrin and gum arabic) and agave sap (aguamiel) as a thermoprotector. The particle size distribution, morphology, stability during storage at different temperatures and water activity of the microcapsules were determined. The results showed significant differences (P<0.05) among the microcapsules. Higher protection was found in the microcapsules containing aguamiel. The microcapsules had a spherical shape with an average diameter of $7.72~\mu m$. It was observed that the microcapsules containing aguamiel had a minor change in colour independent of the drying temperature and preserved the phenolic compounds for more than 1467 days at a storage temperature of 25 °C. These results suggest the application of microencapsulation with phenolic compounds from xoconostle for food products.

Keywords: biopolymers, xoconostle, stability, spray drying.

Resumen

En esta investigación se determinó la estabilidad de compuestos fenólicos de *Opuntia oligacantha* Först (xoconostle) mediante la microencapsulación con una mezcla de biopolímeros (maltodextrina y goma arábiga) y agave sap (aguamiel) como termoprotector. A los microencapsulados secados por aspersión se les determinó distribución de tamaño de partícula, microscopía electrónica de barrido y estabilidad durante el almacenamiento a diferentes temperaturas y actividades de agua. Los resultados mostraron diferencias significativas (P<0.05) entre las microcápsulas encontrando mayor protección en las microcápsulas con aguamiel. Las microcápsulas con formas esféricas tuvieron un diámetro promedio de 7.72 μ m. Se observó que las microcápsulas con aguamiel tuvieron el menor cambio de color independientemente de la temperatura de secado y preservan los compuestos fenólicos por más de 1467 días con temperaturas de almacenamiento de 25 °C. Estos resultados sugieren la aplicación de estos encapsulados con compuestos fenólicos del xoconostle en productos alimenticios.

Palabras clave: biopolímeros, xoconostle, estabilidad, secado por aspersión.

1 Introduction

Opuntia oligacantha Först, known as xoconostle, is a

 * Corresponding author. E-mail: dianajpg@gmail.com

sour fruit that is widely consumed fresh or processed into jams, sweetened appetizers, powders, juices, hot sauces and alcoholic beverages. There are different

¹Departamento de Ingeniería Química, Facultad de Química, Universidad Autónoma del Estado de México, Paseo Tollocan esq. Paseo Colón s/n, CP 50120 Toluca, Estado de México, México.

²Instituto de Ciencias Agropecuarias, Universidad Autónoma del Estado de Hidalgo, Av. Rancho Universitario S/N, Km. 1, CP 43600 Tulancingo, Hgo., México.

³ Facultad de Ciencias Químicas, Universidad Autónoma de San Luis Potosí. Av. Manuel Nava No. 6, Zona Universitaria, San Luis Potosí, SLP México, C.P. 78290

⁴Departamento de Biotecnología, Universidad Autónoma Metropolitana-Iztapalapa, San Rafael Atlixco 186, Vicentina, México DF 09340, México.

species and varieties of xoconostle. This fruit has three parts, namely, the pericarp (thin fruit peel, 1-2 mm thick), the mesocarp (xoconostle skin, 1-1.3 cm thick), and the endocarp (pulp composed by seeds), with the first and last parts usually discarded prior to consumption (Reyes-Agüero and Valiente-Banuet, 2006). This fruit has been studied for the presence of bioactive compounds such as betacyanines and phenolic compounds. The beneficial effects of this fruit are due to its antioxidant activity related to the composition and concentration of phenolic compounds (Zakharova and Petrova 1998; Osorio-Esquivel et al., 2011). The consumption of xoconostle, due to its content of polyphenols, can help improve human health; it may contribute to the prevention of chronic diseases and other problems in humans such as diabetes, obesity and respiratory illnesses (Zavaleta-Beckler et al., 2001; Morales et al., 2012). Pimienta-Barrios et al. (2008) reported that the pericarp of Opuntia joconostle helps to lower cholesterol levels in the blood and to increase the insulin.

Despite all of its benefits, xoconostle is a highly perishable fruit, and the stability of its bioactive compounds is affected by pH, water activity (aw), oxygen, exposure to light, enzymatic activity and temperature (Castellar et al., 2003; Herbach et al., 2004; Tesoriere et al., 2005), limiting its use in the food industry. Temperature is the most significant factor leading to bioactive compound decomposition. Long drying times caused severe degradation in the levels of carotenoids, ascorbic acid, total phenols and antioxidant activity in the red pepper (Castañeda-Pérez et al., 2013). Microencapsulation by spray drying is an economical method for the preservation of bioactive compounds and can be used to convert xoconostle pulp into a stable powder with new possibilities of industrial applications as a source of polyphenols for addition into food products (e.g., cake mixes, gelatine desserts, chewing gums, pet foods, and breakfast cereal).

The spray drying of fruit juices is a process through which the juice can be converted into a powder product. The powder formulation facilitates transport by reducing the weight and it also preserves the product from bacterial degradation and increases its shelf life by drastically reducing its moisture content. Powdered food products made from fruits and vegetables that provide good nourishment and hydrating properties are of interest in the food industry (García-Gutiérrez *et al.*, 2004). The moisture content is essential for the stability and storage of the powder (O'Hagan *et al.*, 2005).

The encapsulating agents that are most used in the spray drying of fruit juices are gum arabic and maltodextrins (Quek et al., 2007) due to their low viscosity and high solubility. These agents, which have high molecular weights, are used to avoid spray drying operational problems in sugar-rich products (fruit juices), such as stickiness on the dryer chamber wall, and structural transformations such as collapse and crystallization during the food processing and storage (Carrillo-Navas et al., 2011; Guadarrama-Lezama, et al., 2014). One interesting possible encapsulation agent is aguamiel (agave sap), which has been used in the spray drying of microcapsules as a thermoprotector of probiotics (Rodríguez-Huezo et al., 2007; Rodríguez-Huezo et al., 2014). Aguamiel is a sap obtained mainly from the species Agave atrovirens (Estrada-Godina et al., 2001) that contains a considerable amount of fructooligosaccharides (FOS) (Cruz-Guerrero et al., 2006). It is an interesting prebiotic due to its dietary fibre content.

The aim of this work was to study the feasibility of the spray drying of xoconostle pulp in three aspects: i) to determine the physicochemical properties of the microcapsules produced, ii) to determine the total colour change of the rehydrated microcapsules, and iii) to evaluate the stability of the polyphenols from xoconostle pulp after rehydration and conditioning at different water activities and temperatures.

2 Materials and methods

2.1 Materials

Opuntia oligacantha Först fruits were harvested from Tezontepec de Aldama, Hidalgo State, Mexico at the second stage of physiological maturity (medium colouration). The fruit was washed and stored at -70 °C until sample preparation. Aguamiel (agave sap) (pH 6.8, 10.7 °Brix, 86 % moisture content, $68 \text{ g L}^{-1} \text{ sucrose}, 9.2 \text{ g L}^{-1} \text{ fructose}, 4.6 \text{ g L}^{-1}$ glucose, 15 g L^{-1} fructooligosaccharides, from A. atrovierens Karb) was obtained from Singuilucan, Hidalgo State, Mexico. Gum arabic (Acacia senegal) (GA) and maltodextrin DE-10 (MD) were obtained from Industria Ragar, S.A. de C.V. (Mexico City, Mexico) and used as protective colloids. Ciocalteu reagent and gallic acid were purchased from Sigma (Sigma Chemical Co., St. Louis, MO, USA). All chemicals used in this study were reagent grade, and all of the water used was bidistilled.

2.2 Methods

2.2.1 Xoconostle pulp obtention

Opuntia oligacantha Först fruit (xoconostle) was thawed and weighed (W1). The pulp from the whole fruit was obtained by an enzymatic method. The whole xoconostle fruit was milled using an industrial blender (Waring HGBSSSS6, Torrington, Connecticut, USA). The pulp was filtered through a # 100 (0.149 mm) sieve to eliminate solids in suspension. The filtered pulp was treated with an enzymatic solution of pectinases with a high hemicellulolytic side activity (Rohapect®B1L, Paniplus, S.A., Querétaro, México) at a ratio of 0.5 mL of enzymatic solution per kg of pulp. enzymatic solution was used to hydrolyse the pectin, hemicelluloses and gum-like substances from the xoconostle pulp.

The xoconostle pulp obtained had similar physicochemical properties to those reported by Morales *et al.* (2012) and Guzmán-Maldonado *et al.* (2010) for *O. joconostle* and *O. matudae*. The total sugar content was 15.42 ± 0.16 mg glucose/mL pulp, soluble solids 5.30 ± 0.10 g/100 g, pH 3.43 ± 0.01 , moisture 93.02 ± 0.02 % and ash 0.86 ± 0.02 %.

2.2.2 Preparation of xoconostle pulp microcapsules by spray drying

Aguamiel was added in an aguamiel:pulp weight ratio of 1:10. A 1:1 w/w blend of gum arabic and maltodextrin DE-10 was dissolved into the solution in a weight ratio of 1:3.7 aguamiel-pulp:hydrocolloid blend to adjust it to a solid content of 30 % w/w, and it was stirred with a plinth stirrer at 200 r.p.m. to homogeneity for 24 h at room temperature (~20 °C). Another solution of the blend of biopolymers and xoconostle pulp was prepared without the addition of aguamiel as a control. The solutions were then fed at a rate of 20 mL/min to a spray dryer (Mini Spray Dryer Büchi model B-290, Switzerland) operated with two inlet temperatures of 150 °C and 160 °C. The outlet temperature was 68 °C, and compressed air was injected at 4 bar. The spray-dried microcapsules were collected and stored in amber dark plastic bags. The spray drying for the formulation was made in duplicate.

2.2.3. Properties of spray-dried microcapsules

2.2.3.1 Moisture content

The moisture content was determined according to the AOAC (1995). Triplicate samples of the microcapsules (1 g) were weighed and dried in an oven at 100 °C for 24 h. The samples were withdrawn from the oven, cooled in desiccators and weighed. The drying and weighing processes were repeated until a constant weight was obtained.

2.2.3.2 Microcapsule particle size

The microcapsule particle size was determined by measuring their volume fraction-length mean size (d_{4:3}) using a Mastersizer 2000 (Malvern Instruments, Ltd., Malvern, Worcestershire, England) with alcohol as the dispersant (Carrillo-Navas *et al.*, 2011).

2.2.3.3 Morphology of microcapsules by scanning electron microscopy (SEM)

The morphology of the microcapsules was examined by SEM. The microcapsules were placed into a metal die that had two-sided adhesive carbon tape attached to it, and then it was coated with gold using a Fine Coat Ion Sputter JFD1100 (Jeol LTD., Akishima, Japan) and observed in an SEM (JEOL Scanning Electron Microscope JMS-035, Jeol Ltd., Akishima, Japan) operated at 20 kV.

2.2.4 Properties of rehydrated microcapsules

The microcapsules were rehydrated to the same moisture content as the natural xoconostle pulp. The quantity of bidistilled water/g of microcapsules was 30 \pm 1 °Brix (2.33 g H₂O/g microcapsule).

2.2.4.1 Total colour change of the rehydrated microcapsules

The total colour change of the rehydrated microcapsules was measured using a Hunter Lab (MINOLTA CM-508d, Japan) colorimeter with a 10° observer. CIELAB values of lightness (L_0^*) , redness (a_0^*) , and yellowness (b_0^*) were determined. The total colour change (ΔE) of the rehydrated microcapsules was obtained with the following expression (Rodríguez-Hernández *et al.*, 2005):

$$\Delta E = (\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2})^{0.5} \tag{1}$$

where Δ indicates the difference between the microcapsules before drying and the rehydrated

microcapsules with respect to the L^* , a^* and b^* parameters.

2.3 2.2.4.2 Total phenolic content of rehydrated microcapsules

The water activity values of the microcapsules after spray drying were determined using an Aqualab water activity meter with temperature compensation, model series 3 TE (Decagon Devices, Inc., Pullman, Washington, USA).

For the stability test, the microcapsules were placed into glass Petri dishes of 50 x 10 mm, taking care that they completely and homogeneously covered the surface of the dishes. These dishes were then introduced into smoke-tinted glass desiccators under vacuum that contained saturated solutions of lithium chloride, magnesium chloride and magnesium nitrate, which provided water activities of 0.115, 0.329, 0.536 at 25 °C, and 0.108, 0.318, 0.515 at 35 °C, respectively, according to the methodology followed by Pitalua et al. (2010). The containers were stored at two different temperatures, 25 °C, representing room temperature and 35 °C, one of the temperatures recommended by Labuza and Schmidl (1985) for accelerated shelf life tests. The microcapsules were weighed with an Ohaus electronic balance model AP210 (Pine Brook, New Jersey, USA) every five days until equilibrium was achieved. Equilibrium was assumed when the difference between two consecutive weightings was less than 1 mg/g of solids. The time to reach equilibrium varied from 15 to 20 days. Then, the microcapsules were rehydrated according to section 2.2.4. The samples were stored for 12 weeks (84 days) and analyzed weekly.

Finally, the extraction of phenolic compounds was performed to determine the total phenolic content of the rehydrated microcapsules, according to Rojas-Barquera et al. (2008) with slight modifications. Two millilitres of the xoconostle pulp extracted from the microcapsules was mixed with 40 mL of an ethanol: water (1:1) solution and magnetically stirred for 35 min. Then, the mixture was centrifuged at 3280.5 g for 15 min at 8 °C (Hermle type Z36HK, Germany). The supernatant was carefully separated and maintained at 4 °C in darkness. The precipitate was dissolved in 40 mL of acetone: water (7:3) solution and mixed with a magnetic stirrer for 35 min. After that, the mixture was centrifuged at 3280.5 g for 15 min at 8 °C. The supernatant from the second extraction was carefully removed and mixed with the supernatant from the first extraction. The supernatant mixture was then centrifuged at 3280.5 g for 90 min at 4 °C before being recovered and used for determining the total phenolic content

The phenolic content was determined according to the Folin-Ciocalteu method as described by Singleton and Rossi (1965) with slight modifications. Folin-Ciocalteu reagent (0.5 mL) and 7.4 mL of bidistilled water were added to 600 μ L of xoconostle pulp rehydrated from the microcapsules and stirred. Then, 1.5 mL of sodium carbonate solution (20% w/v) was added. The samples were stored for two hours, and the absorbance at 760 nm was measured with a spectrophotometer (Varian CARY 100BIO, Italy). The results were expressed as gallic acid equivalents according to a calibration curve (0-20 μ g/mL, with 9 point and r^2 =0.9953).

The total phenolic content was determined each week for 9 weeks. The reaction rate constant k and half-life time $(t_{1/2})$ were calculated based on the equations described by Tonon *et al.* (2010)

$$\ln\left(\frac{C_t}{C_0}\right) = -kt \tag{2}$$

$$t_{1/2} = \frac{\ln 2}{k} \tag{3}$$

where C_o is the initial total phenolic content and C_t is the content at time t.

2.2.5 Statistical analyses

The data for different microcapsules were analysed using one-way analysis of variance (ANOVA), and when there were significant differences (P < 0.05), the Tukey technique was used. All experiments were performed in triplicate, and all data were analysed using NCSS 2007 software (Wireframe Graphics, Kaysville, UT, USA).

3 Results and discussion

3.1 Properties of spray-dried microcapsules

3.1.1. Moisture content of spray-dried microcapsules

Powders formulated with and without the addition of aguamiel showed significant differences (P<0.05) in moisture content, ranging from 1.61 ± 0.12 to 4.17 ± 0.02 % drying base (Table 1), as the aguamiel increased the solids content. The moisture content depends on the wall material; it is reported that when the wall material reaches a moisture content < 7%,

the diffusion coefficient of water is reduced, and this decreases its movement through the dry matrix (Reineccius, 2004). These values are in concordance with those obtained for microcapsules of açai juice with gum arabic and maltodextrin dried at 140, 170 and 200 °C, as reported by Tonon *et al.* (2011), who observed that the moisture content descended with the increase in temperature.

3.1.2. Size distribution of microcapsules

The size particle distribution is important in that it affects aspects including the processing, humidity and shelf life. The size distribution of the particles in the powder is shown in figure 1; the particles cover a range of sizes with diameters that vary from approximately 0.7 to 33 μ m. The volume fractionlength mean size $(d_{4,3})$ obtained in this work was 7.72 μ m, due to the utilization of the binary mixture in the wall materials (GA and MD). Gum arabic exhibits a highly branched spherical structure that tends to form fine, dense, two-dimensional skins immediately upon drying (Pérez-Alonso et al., 2003). Because maltodextrin is obtained by the acid and/or enzymatic hydrolysis of starch, it contains small oligosaccharides of low molecular weight. Either linear amylose chains or branched amylopectin chains can be contained in maltodextrin (Loret et al., 2004), but because it is not possible to differentiate amylose from amylopectin, the entire distribution has been approximated as being composed of only linear amylose chains (Avaltroni et al., 2004). The structure of amylose can only be disordered under specific conditions of temperature (~70 °C); hence, it exhibits a rigid molecular structure, as the conditions used during spray drying are not enough to modify its structure. The average size particle distribution in xoconostle microcapsules can be attributed to the presence of gum arabic. Tonon (2010) noted that the particles produced with maltodextrins showed a wider distribution than those produced with gum arabic, indicating that the size of the particles was slightly more homogeneous in the latter case. These same authors mentioned that when using gum arabic, the diameters of the particles were smaller (8.91 μ m) than those obtained using maltodextrin 10 DE (10.08 μ m). However, they observed the presence of larger particles at the beginning of the agglomeration process.

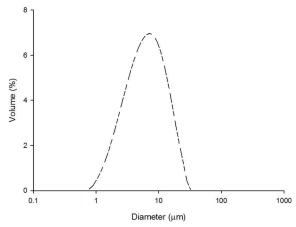


Fig. 1. Particle size distribution of powder produced at 150 °C with an aguamiel:pulp in ratio of 1:10.

3.1.3. Morphology of microcapsules

The microstructure is related to the functionality, stability and fluency of the powders (O'Hagan *et al.*, 2005). The xoconostle microcapsules obtained were examined to determine the presence of fractures, cracks, or any other possible defects that could expose the xoconostle pulp, as any fracture may lead to the degradation of the exposed encapsulated material.

The microcapsules obtained at inlet temperatures of 150 °C and 160 °C in the spray drying process showed the presence of semi-spherical microcapsules. In figure 2A, it is possible to observe that the particles obtained at 150 °C present corrugated surfaces, while those obtained at 160 °C (Fig. 2B) show smoother surfaces but with some fractures, which may lead the xoconostle pulp and its compounds to be degraded easily when they are exposed to the oxygen and light. However, agave sap (aguamiel) provided a thermoprotector effect because the total phenolic content was preserved in the microcapsules obtained at 160 °C, and no significant differences (P > 0.05) were obtained in comparison with the microcapsules without cracks/fractures obtained at 150 °C.

Many factors influence the morphological characteristics of the microcapsules, such as the wall material used for the microencapsulation, the droplet size in solution, and the temperature during the drying process. Any change in the spray drying process conditions could result in changes in the morphological characteristics of the microcapsules obtained.

In this study, the microcapsules obtained at the lowest temperature were observed to not have fractures, which may be due to the low drying

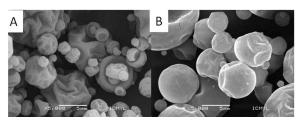


Fig. 2. Scanning electron microscopy images of microcapsules obtained from xoconostle pulp at (A) 150 °C, (B) 160 °C, with aguamiel, gum arabic and maltodextrin.

temperature as well as the viscoelasticity provided by GA (Jimenez-Avalos *et al.*, 2005) that allowed the surfaces to provide a better physical protection of the xoconostle pulp.

The morphology found in this research is in concordance with Oakley (1997), who reported that at low or medium inlet temperatures (74-150 °C), the microcapsules swelled and collapsed during processing, while at high inlet temperatures (153-200 °C), the particles obtained are rigid and porous, which determines the amount of fractured surfaces.

Conversely, it has been reported that most of the microcapsules show a corrugated surface, while the increase in the drying temperature leads to the production of microcapsules with smooth surfaces. This is related to the increase in the drying temperature, resulting in the quicker evaporation of water and leading to the formation of a hard and smooth crust (Tonon *et al.*, 2011).

However, it is possible that the increase in the drying temperature may increase the rate of drying, thus inducing changes in the size of the particles, fractures and broken shells due to the fast shrinkage of the microcapsules followed by an incipient expansion. According to Alamilla-Beltrán et al. (2005), the crust is more flexible and collapsed when low drying temperatures are used, while more rigid and porous crusts are the result of using higher temperatures, leading to a mixture of fractured spheres, broken layers and particles without breaking. When the drying temperature is high enough, the moisture evaporates very quickly and the surface becomes dry and hard. A hollow particle cannot deflate when the vapour condenses inside the vacuole when the particle enters cold regions of the dryer. Nonetheless, when the drying temperature is low, the surface stays wet and flexible for a longer time, so the hollow particle can deflate and wrinkle when it gets cold (Tonon et al., 2011).

3.2 Properties of rehydrated microcapsules

3.2.1 Effect of aguamiel and drying temperature on the properties of the rehydrated microcapsules

Significant differences (P<0.05) between the treatments with and without aguamiel are shown in figure 3. The preservation of total phenols after drying was higher in the case of the treatment with aguamiel $(2.09 \pm 0.01 \text{ mg GAE/mL})$, as the xoconostle pulp dried without aguamiel showed a decrease of 3.5%.

The drying temperature did not have a significant effect (P>0.05) in on the phenolic compounds. The aguamiel effect could be due to interactions with the mucilage of the xoconostle pulp, as Rodríguez-Huezo *et al.* (2007, 2014) reported on aguamiel acting as a thermoprotector in the spray drying of probiotic bacteria.

The colour change due to the treatments was measured (Table 1). The results showed significant differences (P<0.05) between the treatments. The control showed a higher change compared to treatments with the addition of aguamiel at both drying temperatures; it is suggested that aguamiel protects the compounds that give the xoconostle pulp its colour. The minor change of colour was present in the treatments with an aguamiel-pulp proportion of 1:10, independent of the drying temperature. Colour is an important indicator of quality that reflects the sensorial attractiveness and the quality of the powders produced in the spray drying process (Quek *et al.*, 2007).

Based on these results, it was determined that the treatment with an aguamiel-pulp ratio of 1:10 and drying at 150°C was better than the control due to its higher phenol content and because it confers colour protection. For this reason the following tests were performed on the treatment: particle size distribution, morphology, and stability during storage at different temperatures and water activities.

3.2.2 Stability during storage

The microcapsules obtained at 150 °C were stored at 25 and 35 °C for 84 days under three different activities of water; the zero time was when it was reached the desired water activity. The degradation of the total phenols in the particles produced with a mixture of gum arabic and maltodextrin as encapsulating agents showed two first-order kinetics,

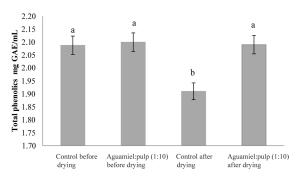


Fig. 3. Total phenolics in the treatments with and without aguamiel obtained before and after spray drying at 150 °C.

Table 1. Colour change and moisture content of xoconostle microencapsulated obtained with and without aguamiel at different temperatures.

	Temperature of drying	ΔE	% moisture
Control	150 °C	2.07 ± 0.05^a	4.17 ± 0.02^a
1:10 aguamiel	150 °C	1.57 ± 0.06^b	2.88 ± 0.03^{b}
Control	160 °C	1.56 ± 0.05^b	2.98 ± 0.08^{b}
1:10 aguamiel	160 °C	1.44 ± 0.04^{c}	1.61 ± 0.12^{c}

 $^{a-b,c}$ Means with different superscript letters in the same column indicate significant differences (P<0.05)

 ΔE =Total colour change between the before drying and rehydration

the first with a higher rate of reaction until day 22 of storage and the second one from day 23 on, with a lower rate that was constant during the test (Fig. 4). According to Desobry *et al.* (1997), the interval that presents a higher degradation speed corresponds to the degradation of compounds present on the surface of the microcapsule or compounds inside the microcapsule that are in contact with the oxygen present in the pores of the particle or trapped inside the microcapsule in the form of bubbles, which permits their oxidation.

It is also important to mention that after day 54, the microcapsules at water activities higher than 0.3 collapsed, although the content of total phenols was still perceptible. According to Tonon *et al.* (2010), this is due to the higher water adsorption at the beginning of the storage leading to a higher molecular mobility and thereby a higher degradation rate.

The particles showed two different first-order kinetics, so two values of k and two half-life times were calculated for each sample, as shown in table 2. The increase in temperature permits a faster degradation of total phenols. The influence of

the temperature on the stability of the antioxidant compounds has been reported by several authors (Tonon *et al.*, 2010; Pacheco-Palencia *et al.*, 2007). The half-life time $(t_{1/2})$ found for the rehydrated xoconostle pulp was 1467.51 days at 25 °C and a water activity of 0.115, which is higher than the value obtained by Jiménez-Aguilar *et al.* (2011), who reported a half-life of 300 days for blueberry extract stored at 25 °C in presence of light, and the value obtained by Ersus and Yurdagel (2007), who determined a half-life of 270 to 300 days for blueberry extract stored at 25 °C. It is similar to the values reported by Tonon *et al.* (2010), who

Table 2. Half-life time of the microcapsules with 1:10 aguamiel: xoconostle pulp at different temperatures and water activities of storage.

Storage condit	ions		
Temperature (°C)	aw	$k(\text{days}^{-1})$	$t_{1/2}$ (days)
25	0.115	0.0035	195.26
		0.0005	1467.51
25	0.329	0.0060	116.30
		0.0011	637.30
25	0.536	0.0126	54.96
		0.0059	117.44
35	0.108	0.0052	132.83
		0.0008	870.80
35	0.318	0.0069	100.87
	0.0015	475.58	
35	0.515	0.0160	43.44
		0.0090	76.87

aw= activities of water; k= reaction rate constant; $t_{1/2}$ = half-life time

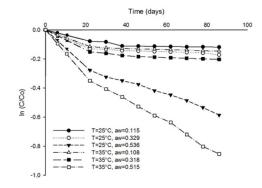


Fig. 4. Kinetics of total phenols degradation of the powder produced at 150 °C with an aguamiel: pulp ratio of 1:10 at different temperatures (25 and 35°C) and water activities (0.115, 0.329 and 0.556; 0.108, 0.318 and 0.515 respectively).

presented values of 1386.29 days for particles of açai juice stored at 25 °C at a water activity of 0.329. The higher degradation of phenolic compounds at higher temperatures can be related to the presence of sugars in addition to proteins that can carry out the Maillard reaction, which occurs during the processing at high temperature or during the long-term storage. This reaction is highly dependent on the temperature, is accelerated in the presence of oxygen and occurs more frequently in fruit juices (Tonon *et al.*, 2010).

The influence of the temperature on the degradation speed was higher for the samples stored at a higher relative humidity. This indicates that the water activity also plays an important role in the degradation of the phenolic compounds of xoconostle pulp.

At a higher water content, there is a higher molecular mobility inside the meal, which facilitates the physicochemical reactions of degradation (Tonon *et al.*, 2010).

The higher degradation at 84 days of storage was present at a temperature of 35 °C and water activity of 0.515, giving a 57 % a reduction relative to the initial content of phenols, while the lower one was at a temperature of 25 °C with a water activity of 0.115, yielding a final value 11 % less than the initial concentration (Fig. 4).

Conclusions

The results of this work show that using a blend of biopolymers (gum arabic and Maltodextrin 10DE) and agave sap (aguamiel) provides protection to the phenolic compounds contained in xoconostle (*Opuntia oligacantha* Först) pulp entrapped in synthetic matrices.

When the temperature of inlet was increased in the spray drying process, fractures were observed in the xoconostle microcapsules, but agave sap (aguamiel) had a thermoprotective effect, with the total phenolic content preserved and no significant differences obtained in comparison with microcapsules without cracks/fractures. The microcapsules containing agave sap (aguamiel) had the highest storage stability, a longer half-life storage at 25 °C and the lowest water activity (0.115). It also prevented changes in colour in the rehydrated xoconostle pulp.

This work contributes to the knowledge of improving the stability of microcapsules including agave sap (aguamiel) as a thermoprotector, and xoconostle microcapsules can be used as an instant

powder in the food industry for the pigmentation of foodstuffs.

Acknowledgements

The authors thank the Consejo Nacional de Ciencia y Tecnología (CONACyT) through project CB-2012-01-183807 for supporting this work, Biol. Yolanda Hornelas Orozco de Uribe from Instituto de Ciencias del Mar y Limnología of UNAM for the micrographs; and PANIPLUS, S.A. for the samples of enzymes.

References

- Alamilla-Beltrán, L., Chanona-Pérez, J.J., Jiménez-Aparicio, A.R., Gutiérrez-López, G.F. (2005). Description of morphological changes of particles along spray drying. *Journal of Food Engineering* 67, 179-184.
- A.O.A.C. (1995). *Official methods of analysis* of AOAC (16th Ed.). Arlington, VA: Association of Official Analytical Chemists, (pp. 12-14).
- Avaltroni, F., Bouquerand, P.E., Normand, V. (2004). Maltodextrin molecular weight distribution influence on the glass transition temperature and viscosity in aqueous solutions. *Carbohydrate Polymers* 58, 323-334.
- Carrillo-Navas, H., González-Rodea, D. A., Cruz-Olivares, J., Barrera-Pichardo, J.F., Román-Guerrero, A., Pérez-Alonso, C. (2011). Storage stability and physicochemical properties of passion fruit juice microcapsules by spraydrying. *Revista Mexicana de Ingeniería Química 10*, 421-430.
- Castañeda-Pérez, E., Osorio-Revilla, G. I., Gallardo-Velázquez, T., Proal-Nájera, J. B. (2013). Use of FTIR-HATR coupled to multivariate analysis to monitor the degradation of bioactive compounds during drying of red pepper. *Revista Mexicana de Ingeniería Química 12*, 193-204.
- Castellar, R., Obón, J. M., Alacid, M., Fernández-López, J. A. (2003). Color properties and stability of betacyanins from Opuntia fruits. *Journal of Agricultural and Food Chemistry* 51, 2772-2776.
- Cruz-Guerrero, A. E., Olvera, J. L., García-Garibay, M., Gómez-Ruiz, L. (2006). Inulinase-hyperproducing strains of *Kluyveromyces* sp.

- isolated from aguamiel (*Agave* sap) and pulque. *World Journal of Microbiology and Biotechnology* 22, 115-117.
- Desobry, S. A., Netto, F. M., Labuza, T. P. (1997). Comparison of Spray?drying, Drum?drying and Freeze-drying for β?Carotene Encapsulation and Preservation. *Journal of Food Science* 62, 1158-1162.
- Ersus, S., Yurdagel, U. (2007). Microencapsulation of anthocyanin pigments of black carrot (*Daucus carota* L.) by spray drier. *Journal of Food Engineering 80*, 805-812.
- Estrada-Godina, A. R., Cruz-Guerrero, A. E., Lappe, P., Ulloa, M., García-Garibay, M., Gómez-Ruiz, L. (2001). Isolation and identification of killer yeasts from *Agave* sap (aguamiel) and pulque. *World Journal of Microbiology and Biotechnology* 17, 557-560.
- García-Gutiérrez, C., González-Maldonado, M.B., Ochoa-Martínez, L.A., Medrano-Roldan. (2004). Microencapsulación de jugo de cebada verde mediante secado por aspersión. *Ciencia y Tecnología Alimentaria* 4, 262-266.
- Guadarrama-Lezama, A.Y., Cruz-Olivares, J., Martínez-Vargas, S.L., Carrillo-Navas, H., Román-Guerrero, A., Pérez-Alonso, C. (2014). Determination of the minimum integral entropy, water sorption and glass transition temperature to establishing critical storage conditions of beetroot juice microcapsules by spray drying. Revista Mexicana de Ingeniería Química 13, 405-416.
- Guzmán-Maldonado, S. H. Morales-Montelongo, A. L., Mondragón-Jacobo, C., Herrera-Hernández, G., Guevara-Lara, F., Reynoso-Camacho, R. (2010). Physicochemical, nutritional, and functional characterization of fruits xoconostle (*Opuntia matudae*) pears from central-Mexico region. *Journal of Food Science* 25, C485-C492.
- Herbach, K. M., Stintzing, F. C., Carle, R. (2004). Impact of thermal treatment on color and pigment pattern of red beet (Beta vulgaris L.) preparations. *Journal of Food Science* 69, C491-C498.
- Jiménez-Aguilar, D. M., Ortega-Regules, A. E., Lozada-Ramírez, J. D., Pérez-Pérez, M. C. I.,

- Vernon-Carter, E. J., Welti-Chanes, J. (2011). Color and chemical stability of spray-dried blueberry extract using mesquite gum as wall material. *Journal of Food Composition and Analysis* 24, 889-894.
- Jimenez-Avalos, H.A., Ramos-Ramirez, E.G., Salazar-Montoya, J.A. (2005). Viscoelastic characterization of gum arabic and maize starch mixture using the Maxwell model. Carbohydrate Polymers 62, 11-18.
- Labuza, T. P., Schmidl, M. K. (1985). Accelerated shelf-life testing of foods. *Food Technology 39*, 57-62.
- Loret, C., Meunier, V., Frith, W.J., Fryer, P.J. (2004). Rheological characterization of the gelation behavior of maltodextrin aqueous solutions. *Carbohydrate Polymers 57*, 153-163.
- Morales, P., Ramírez-Moreno, E., Sanchez-Mata, M.
 D. C., Carvalho, A. M., Ferreira, I. C. (2012).
 Nutritional and antioxidant properties of pulp and seeds of two xoconostle cultivars (*Opuntia joconostle* FAC Weber ex Diguet and *Opuntia matudae* Scheinvar) of high consumption in Mexico. Food Research International 46, 279-285.
- Oakley, D.E. (1997). Produce uniform particles by spray drying. *Chemical Engineering Progress* 93, 48-54.
- O'Hagan, P., Hasapidis, K., Coder, A., Helsing, H., Pokrajac, G. (2005). Particle size analysis of food powders. In: C. Onwulata (ed.), *Encapsulated and Powdered Foods*, Taylor & Francis, Food Science and Technology (pp. 215-245). New York: Marcel Dekker Inc.
- Osorio-Esquivel, O., Álvarez, V. B., Dorantes-Álvarez, L., Giusti, M. M. (2011). Phenolics, betacyanins and antioxidant activity in *Opuntia joconostle* fruits. *Food Research International* 44, 2160-2168.
- Pacheco-Palencia, L. A., Hawken, P., Talcott, S. T. (2007). Phytochemical, antioxidant and pigment stability of açai (*Euterpe oleracea* Mart.) as affected by clarification, ascorbic acid fortification and storage. *Food Research International* 40, 620-628.

- Pérez-Alonso, C., Báez-González, J. G., Beristain, C. I., Vernon-Carter, E. J., Vizcarra-Mendoza, M. G. (2003). Estimation of the activation energy of carbohydrate polymers blends as selection criteria for their use as wall material for spraydried microcapsules. *Carbohydrate Polymers* 53, 197-203.
- Pimienta-Barrios, E., Méndez-Moran, L., Ramírez-Hernández, B. C., García de Alba-García, J. E., Domínguez-Arias, R. M. (2008). Effect of xoconostle (*Opuntia joconostle* Web.) fruit consumption on glucose and seric lipids. *Agrociencia* 42, 645-653.
- Pitalua, E., Jiménez, M., Vernon-Carter, E. J., Beristain, C. I. (2010). Antioxidative activity of microcapsules with beetroot juice using gum Arabic as wall material. *Food and Bioproducts Processing* 88, 253-258.
- Quek, S. Y., Chok, N. K., Swedlund, P. (2007). The physicochemical properties of spray-dried watermelon powders. *Chemical Engineering* and Processing: Process Intensification 46, 386-392.
- Reineccius, G.A. (2004). The spray drying of food flavors. *Drying Technology* 22, 1289-1324.
- Reyes-Agüero, J. A., Aguirre R, J. R., Valiente-Banuet, A. (2006). Reproductive biology *Opuntia*: A review. *Journal of Arid Environments* 64, 549-585.
- Rodríguez-Hernández, G.R., González-García, R., Grajales-Lagunes, A. and Ruíz-Cabrera, M.A. (2005). Spray-drying of cactus pear juice (*Opuntia streptacantha*): Effect on the physicochemical properties of powder and reconstituted product. *Drying Technology 23*, 955-973.
- Rodríguez-Huezo, M. E., Durán-Lugo, R., Prado-Barragán, L. A., Cruz-Sosa, F., Lobato-Calleros, C., Alvarez-Ramírez, J., Vernon-Carter, E. J. (2007). Pre-selection of protective colloids for enhanced viability of *Bifidobacterium bifidum* following spray-drying and storage, and evaluation of aguamiel as thermoprotective prebiotic. *Food Research International* 40, 1299-1306.

- Rodríguez-Huezo, M. E., Estrada-Fernández, A. G., García-Almendárez, B. E., Ludeña-Urquizo, F., Campos-Montiel, R. G., Pimentel-González, D. J. (2014). Viability of *Lactobacillus plantarum* entrapped in double emulsion during Oaxaca cheese manufacture, melting and simulated intestinal conditions. *LWT-Food Science and Technology* 59, 768-773.
- Rojas-Barquera, D.R., Narváez-Cuenca, E.C., Restrepo-Sánchez, L.P. (2008). Evaluación del contenido de vitamina C, fenoles totales y actividad antioxidante en pulpa de guayaba (*Psidium guajava* L.) de las variedades pera, regional roja y regional blanca. *Memorias de Red-Alfa Lagrotech*, 49-60.
- Singleton, V. L., Rossi, J. A. (1965). Colorimetry of total phenolics with phosphomolybdic-phosphotungstic acid reagents. *American Journal of Enology and Viticulture 16*, 144-158.
- Tesoriere, L., Butera, D., Allegra, M., Fazzari, M., Livrea, M. A. (2005). Distribution of betalain pigments in red blood cells after consumption of cactus pear fruits and increased resistance of the cells to ex vivo induced oxidative hemolysis in humans. *Journal of Agricultural and Food Chemistry* 53, 1266-1270.
- Tonon, R. V., Brabet, C., Hubinger, M. D. (2010). Anthocyanin stability and antioxidant activity of spray-dried açai (*Euterpe oleracea* Mart.) juice produced with different carrier agents. *Food Research International* 43, 907-914.
- Tonon, R. V., Freitas, S. S., 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, 691-700.
- Zakharova, N. S., Petrova, T. A. (1998). Relationships between the structure and antioxidant activity of certain betalains. *Applied Biochemistry and Microbiology* 34, 182-185.
- Zavaleta-Beckler, P., Olivares-Orozco, L. J., Montiel-Salero, D., Chimal-Hernández, A., Scheinvar, L. (2001). Organic fertilization in sour prickly pear (*Opuntia joconostle* and *O. matudae*). *Agrociencia*.