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Encapsulation of cactus (*Opuntia megacantha*) betaxanthins by ionic gelation and spray drying: A comparative study

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ABSTRACT

The encapsulation of betaxanthins from orange *Opuntia megacantha* fruits was performed as a stabilisation strategy for these natural pigments. Betaxanthin-rich extract (BE) was encapsulated by two methodologies, spray drying (SD) and ionic gelation (IG). Encapsulates were obtained by using two encapsulating agents (EA), a mixture of maltodextrin-cactus cladode mucilage and sodium alginate (SA) for SD and IG, respectively. Their properties were compared with the freeze-dried cactus fruit pulp (CP) as control. Total betaxanthin content, moisture content, water activity (a_w), particle size, morphology by SEM, colour parameters (CIE*Lab*), and thermogravimetric properties were analysed in the encapsulates. Additionally, the antioxidant activity, total dietary fibre content, and pigment stability of encapsulates under two relative humidity conditions were also evaluated. The results showed that the encapsulation technique, EA concentration, and the BE/EA ratio affected the particle size and morphology, the glass transition temperature, and the mass loss of encapsulated materials. The best pigment stabilisation was obtained for the SD capsules, although both encapsulation techniques allowed improving the pigment stability in comparison with the CP taken as control. These results provided evidence that betaxanthins from *Opuntia megacantha* have the potential to be used as natural pigments, soluble in water, with functional properties for the food industry.

1. Introduction

Due to the increased health-promoting awareness, substitution of artificial colorants by their natural counterparts is a major goal of the food, pharmaceutical and cosmetic industries (Stintzing & Carle, 2004). In order to meet the growing demand for natural colorants, new pigment crops are being sought. Thus, orange cactus pear (*Opuntia megacantha*) belonging to the Cactaceae family has been suggested as a valuable source of betaxanthins (Coria-Cayupán & Nazareno, 2015). These pigments, such as indicaxanthin (proline-betaxanthin) and γ–aminobutyric acid-betaxanthin, provide a yellow-orange colour and exhibit antioxidant properties (Azeredo, 2009; Stintzing, Schieber, & Carle, 2003). Yahia and Mondragon-Jacobo (2011) evaluated the nutritional components and antioxidant capacity of ten cactus pear cultivars from Mexico finding that *O. megacantha* exhibited better nutritional characteristics (tocopherols, β-carotene, and total phenolic content) than those species most commonly consumed in Mexico.

However, the replacement of artificial colorants by natural ones, is often restricted due to their colour instability in the presence of oxygen, light, metals, pH, and high storage temperatures. To overcome this drawback, the encapsulation techniques of spray-drying and ionic gelation have been used as an effective way to protect food pigments against deterioration (Otálora, Carriazo, Iturriaga, Nazareno, & Osorio, 2015; Otálora, Carriazo, Iturriaga, Osorio, & Nazareno, 2016). Spray drying is the most commonly used microencapsulation technique for food products due to its versatility and efficiency. The final product quality and powder efficiency mainly depend on the operating conditions (Tonon, Brabet, & Hubinger, 2008). Another option of encapsulation is the ionic gelation with formation of alginate gels by ionic cross-linking with multivalent cations. This method uses very mild conditions avoiding harmful organic solvents. It is very simple, and the reversible physical crosslinking by electrostatic interactions excludes also the possible toxicity of chemical reagents and other undesirable effects in food products (Usmiati, Richana, Mangunwidjaja, Noor, & Prangdimurti, 2014). Alginate is an anionic biopolymer produced by brown algae and bacteria and consists of α-*L*-guluronic acid (G) and β-

Abbreviations: BE, betaxanthin-rich extract; CA, calcium alginate beads; CP, non-encapsulated lyophilised pulp of cactus pear; CM, cladode mucilage; DSC, differential scanning calorimetry; EA, encapsulating agents; MD, maltodextrin DE-20; RH, relative humidity; SA, sodium alginate; SEM, scanning electron microscopy; SD, spray drying; TGA, thermogravimetric analysis

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D-mannuronic acid (M) residues linearly linked by 1,4-glycosidic linkages (Yang, Xie, & He, 2011). The gelation of alginate occurs by exchange of sodium ions from the guluronic acids (G) blocks with multivalent cations and the stacking of these G blocks to form a characteristic "egg-box" structure (Lee & Mooney, 2012; Poojari & Srivastava, 2013).

There are scarce scientific studies using the encapsulation of cactus betalains by ionic gelation (Otálora et al., 2016); in contrast, there are several reports on betalain microencapsulation by spray-drying from cactus pear fruits (Robert, Torres, García, Vergara, & Sáenz, 2015; Saénz, Tapia, Chávez, & Robert, 2009; Vergara, Saavedra, Sáenz, García, & Robert, 2014). These literature reports the presence of small amounts of betaxanthins in *Opuntia ficus-indica* encapsulates; however, the research on the spray-drying encapsulation of betaxanthins from yellow-orange cactus pear (as indicaxanthin source) is too little (Gandía-Herrero, Cabanes, Escribano, García-Carmona, & Jiménez-Atiénzar, 2013; Gandía-Herrero, Jiménez-Atiénzar, Cabanes, García-Carmona, & Escribano, 2010; Krishnaiah, Nithyanandam, & Sarbatly, 2014).

Thus, the aim of this study was to encapsulate the orange betaxanthins from *Opuntia megacantha* fruit pulp by spray drying and ionic gelation, and comparatively evaluate the physicochemical properties, morphology, and antioxidant activity (DPPH· method) of the encapsulates. The pigment storage stability under two different humidity conditions was evaluated in terms of total betaxanthin content changes by UV–Vis spectrophotometry.

2. Materials and methods

2.1. Materials

Opuntia megacantha fruits (orange pulp) corresponding to the clone 1380 and cladodes of *Opuntia ficus-indica* from the clone 1279, were collected from the cactus collection belonging to the Faculty of Agronomy and Agroindustries (National University of Santiago del Estero) at the province of Santiago del Estero, located in the Argentinean region of the dry Chaco (27° 45′ S, 64° 18′ W, 170 m over sea level). The accession used in this study for the extract preparation (clone 1380) was identified following the proceedings from the cactus collection of UAA Antonio Narro from Saltillo, Mexico. As encapsulating agents (EA), maltodextrin DE-20 (MD) (Disproalquimicos, Bogotá, Colombia) and sodium alginate (SA) (Emulgel CP 3792, Saporiti, Buenos Aires, Argentina) were used as received without further purification. Cladode mucilage (CM) obtained by the method reported by Quinzio, Corvalán, Lopez, and Iturriaga (2009). Calcium chloride dihydrate ($CaCl_2.2H_2O$) was purchased from Cicarelli Laboratories (Buenos Aires, Argentina).

2.2. Preparation of betaxanthin-rich extract

Cactus fruit pulp from *Opuntia megacantha* was crushed in a homogeniser (Phillips, Buenos Aires, Argentina) and the seeds were removed by filtration. The homogenised cactus fruit pulp was lyophilised in a freeze-dryer Labconco Freezone 4.0 (Kansas City, MO, USA) to reach final moisture content between 1.9 and 2.3% (wet base). Then, the sample was kept in hermetic flasks with light protection and stored at −20 °C until be used. To obtain the betaxanthin-rich extract (BE), lyophilised cactus pulp was macerated with a phosphate buffer (pH 5.5), using 1:2 (*w*/w) or 1:10 (w/w) pulp/buffer ratio for ionic gelation and spray drying, respectively. Control samples were non-encapsulated lyophilised pulps of cactus pear (CP).

2.3. Microencapsulation by spray drying (SD)

The method described by Otálora et al. (2015) was followed with some modifications. The betaxanthin-rich extract (BE) was combined with maltodextrin DE-20 (MD), and cladode mucilage (CM) from *Opuntia ficus-indica,* in a ratio of 1:1:0.225 *w*/w/w, respectively. The spray-drying process was performed in a laboratory-scale LabPlant SD-06 spray-dryer (LabPlant, Huddersfield, England), with an 1110 × 825 × 600 mm main spray chamber. The feed-mixture was kept under constant magnetic stirring at 18 °C until homogeneity, and spray-dried immediately with an air flow of 100 m^3/h and a compressor air pressure of 4 bar. The nozzle internal diameter was 2.0 mm, feed flow was 485 ml/h, and the inlet and outlet air temperatures were 170 °C, and 77 °C, respectively. The obtained powder (SD) was collected in plastic containers, weighed, and stored in a desiccator containing silica gel at 18 °C.

2.4. Encapsulation by ionic gelation (IG)

The method described by Otálora et al., 2016 was followed. The BE was combined with sodium alginate (15 g/l) at pH 5.5 in a proportion of 1:0.015 *w*/w, respectively, using a homogeniser (Phillips, Buenos Aires, Argentina) for 2 min at low speed. Once the solution was homogenised, it was slowly added (drop-wise by using a 5 ml pipette with a 1.43 ± 0.03 mm internal diameter tip) on a 0.15 M calcium chloride solution, at 15 cm height between the tip and the calcium chloride solution. The hydrogel beads were maintained in the calcium chloride solution for 1 min (hardening time) and washed with distilled water. Subsequently, they were filtered and washed again with distilled water. Hydrogel beads dehydration was performed by air-drying at 30 ± 1 °C for 24 h in a forced-air circulating oven (Termo Dalvo SRL, Model TDC 30, Buenos Aires, Argentina).

2.5. Characterisation of encapsulated betaxanthins

2.5.1. Moisture content and water activity

Moisture content of the encapsulates was determined gravimetrically by drying 20 mg of each sample, in triplicate, at 70 °C until constant weight (method 934.06, AOAC, 1997). The water activity (a_w) of samples (1 g of each) was measured in triplicate by using a hygrometer HygroPalm AW1 (Rotronic Instruments, Huntington, NY, USA) at 18 °C.

2.5.2. Betaxanthin content

The encapsulates, SD (800 mg) and IG (300 mg) were accurately weighed and dispersed in 5 ml of distilled water, and 5 ml of methanol: water mixture (1,1, *v*/*v*), respectively, at 18 °C for 1 h and then centrifuged at 10000 rpm for 20 min. These suspensions were filtered through a Millipore membrane (0.45 μm). The concentration of betaxanthins was measured at λ = 480 nm by using a Jenway 7305 UV/visible spectrophotometer (Jenway, Staffordshire, UK) according to the method reported by Coria-Cayupán, Ochoa, and Nazareno (2011). The obtained values were expressed by using indicaxanthin as reference compound (ε = 48,000 l/mol cm; molecular weight = 308 g/mol, and λ = 480 nm). The same procedure was used for CP in order to obtain control measurements.

2.5.3. Colour measurement

The CIELAB parameters (L^*, a^*, b^*) of encapsulates were measured by using a Mini Scan EZ HunterLab, (Hunter Associated Laboratory, INC, Virginia, USA). The other colour parameters, chroma (C^*_{ab}) and hue (h_{ab}), were calculated as in Eqs. (1) and (2), respectively (Meléndez-Martínez, Vicario, & Heredia, 2003).

$$C^*_{ab} = [(a^*)^2 + (b^*)^2]^{\frac{1}{2}} \quad (1)$$

$$h_{ab} = \arctan\left(b^*/a^*\right) \quad (2)$$

2.5.4. Particle morphology

The morphology of encapsulates was evaluated by using a scanning electron microscope (SEM) FEI QUANTA 200 (operating at 20 kV), coating the samples by gold sputtering before their examination. A Quorum Q150R ES (Quorum Technologies, East Sussex, UK) sputter coater was used.

2.5.5. Thermal analysis

Analyses of encapsulating agents SA, MD, CM, and the encapsulates (SD and IG) were carried out using a differential scanning calorimeter (DSC)/thermogravimetric analyser (TGA) (STAR System DSC/TGA I, Mettler Toledo Instrument Ltd., Leicester, UK). The equipment was calibrated with high purity indium (T_m = 429.8 K, ΔH_m = 28.4 J g^{-1}). The experiments were performed under nitrogen flow (50 cm^3/min). The samples were heated from 20 to 300 °C for DSC and from 20 to 600 °C for TGA, in aluminium crucibles with a linear heating rate of 10 °C/min. An empty aluminium crucible was used as the reference material.

2.5.6. Dietary fibre content

Total dietary fibre (TDF) content was determined according to AOAC enzymatic–gravimetric procedure (method 985.29, AOAC, 2002).

2.5.7. Determination of antiradical activity by DPPH· method

A 500 µl aliquot of the BE were mixed with 2.5 ml of DPPH· methanol solution (31.6 mg DPPH·/L). The decrease in absorbance at 515 nm was monitored in 20 cycles every 30 s for 10 min using a UNICAM UV2 UV/Vis spectrophotometer (Cambridge, UK). The antiradical activity (ARA) was calculated according to the Eq. (3), as follows:

$$\%ARA = 100 \times (1 - A_{ss}/A_0) \quad (3)$$

where, A_{SS} is the absorbance of the solution at the steady state, and A_0 is the absorbance of DPPH· solution before adding the antioxidant. A_{SS} was estimated by the mathematical fitting of kinetic curves performed with Origin 7.0 software as indicated in Coria-Cayupán et al. (2011). The samples were analysed in triplicate.

2.6. Stability studies of encapsulated betaxanthins

Encapsulates (SD and IG) and CP samples were weighed (400 mg of each) in Petri dishes (4.5 cm diameter) and stored in the absence of light, at 18 ± 1 °C, and with controlled relative humidity. The samples were placed in sealed desiccators containing 200 ml of saturated solutions of KNO_3 and NaBr, to obtain constant humidity values of 90 ± 2%, and 57 ± 2%, respectively. Taking the initial concentration as reference of zero time, the samples were withdrawn every 5, 10, 15, 20, 25, and 30 days for the measurement of betaxanthin content (as described in section 2.5.2). All the measurements were done in triplicate. The observed release rate constant (k) and half-life time ($t_{1/2}$) were calculated by the method reported by Cai and Corke (2000) and adjusted to a pseudo-first order behaviour (Saénz et al., 2009). Half-life ($t_{1/2}$) for the betaxanthin retention was calculated from the rate constant as 0.693/k.

The pigment retention was calculated according to Eq. (4).

$$\text{Betaxanthin retention (\%)} = M_t/M_0 \times 100 \quad (4)$$

where M_t and M_0 are the absolute amount of betaxanthins dispersed in distilled water (SD) or methanol:water (1,1) (IG) at storage time and before starting the experiment (zero storage time), respectively.

2.7. Statistical analysis

The results were reported as the mean ± standard deviation (n = 3). Analysis of variance (ANOVA) was performed to identify differences among the means with the statistical package InfoStat/P version1.1 computing program (Di Rienzo et al., 2012) Differences at probability level $p < 0.05$ were considered significant.

Table 1
Physicochemical characterisation of lyophilised pulp of cactus pear (CP), and the encapsulates, SD and IG.

Parameter	Sample		
	SD	IG	CP
Aw at 18 °C	0.393 ± 0.007^b	0.509 ± 0.002^a	0.379 ± 0.001^b
Moisture (%)	1.18 ± 0.02^b	2.86 ± 0.01^a	1.96 ± 0.09^b
Betaxanthin content*	0.34 ± 0.01^b	0.32 ± 0.09^a	0.30 ± 0.04^a
Total dietary fibre content (TDF)**	< 4.0^c	15.0^a	8.0^b
Antiradical activity ***	31.01 ± 2.19^a	29.77 ± 1.24^a	28.85 ± 2.05^a
Colour parameters			
L^*	50.42 ± 0.54^a	7.83 ± 0.04^b	7.34 ± 0.07^b
a^*	1.18 ± 0.17^c	9.33 ± 0.02^b	15.32 ± 0.07^a
b^*	20.69 ± 0.23^b	21.90 ± 0.02^b	30.99 ± 0.94^a
C^*_{ab}	20.72 ± 0.29^b	23.81 ± 0.03^b	34.56 ± 0.95^a
h_{ab}	86.72 ± 1.32^a	66.91 ± 1.00^b	63.69 ± 1.34^b

All data are the mean of measurements (n = 3) ± standard deviation. Different letters in the same raw indicate a statistical difference ($p < .05$).

* Expressed as mg indicaxanthin equivalents/g solid.

** Expressed as g/100 g.

*** Expressed as µg of vitamin C/g of sample.

3. Results and discussion

3.1. Characterisation of the encapsulates

According to the obtained results shown in Table 1, the moisture content was lower than 4% in the two encapsulates, with the lowest value of 1.18% for SD sample. Accordingly, a_w value was also lower in SD sample; this result may be explained because a higher temperature was used for spray drying which gave a greater driving force in the water evaporation, resulting in an encapsulated material with lower moisture content and a_w (Laokuldilok & Kanha, 2015). The a_w of both encapsulates was < 0.6, which allows to consider these solids as biochemically and microbiologically stable.

Table 1 shows that the betaxanthin content in both encapsulates (SD and IG) are comparable, among them and with the sample control (CP). The presence of betaxanthins in *Opuntia megantha* fruit contributes to give biofunctional properties to this raw material, because there is previous evidence on the correlation between the betaxanthin content and the antiradical activity in this kind of plant material (Stintzing et al., 2005). The betaxanthins in CP have been identified as indicaxanthin, betanin, and isobetanin by Stintzing, Schieber, and Carle (2002).

The colour parameters in SD, IG and CP samples are also shown in Table 1. For the SD and IG, the L^* value increased, whereas the a^* and b^* values decreased with respect to CP, which could be explained by the influence of the encapsulating agent on the final colour of solids, since the pigments were incorporated into the matrix and protected. The value of b^* was higher in CP sample, indicating a tendency to more intense yellow colour due to a higher exposition of (barer) pigments. In general, the chroma parameter (C^*_{ab}) is lower for encapsulated materials as consequence of a decrease in both a^* and b^* values. On the other hand, the h_{ab} values mean that SD exhibits a yellowness grade, which is dependent on the encapsulating agent (BE/EA ratio was different for the two samples). It is important to point out that the particle size of fine powders has a significant influence on the colour hue of the materials. Thus, the lower particle size of SD powders could be contributing to the higher value of h_{ab} for this sample. In material science, it is clear that the lower particle size the higher surface area, and therefore a higher molecule-light interaction is possible to be occurred.

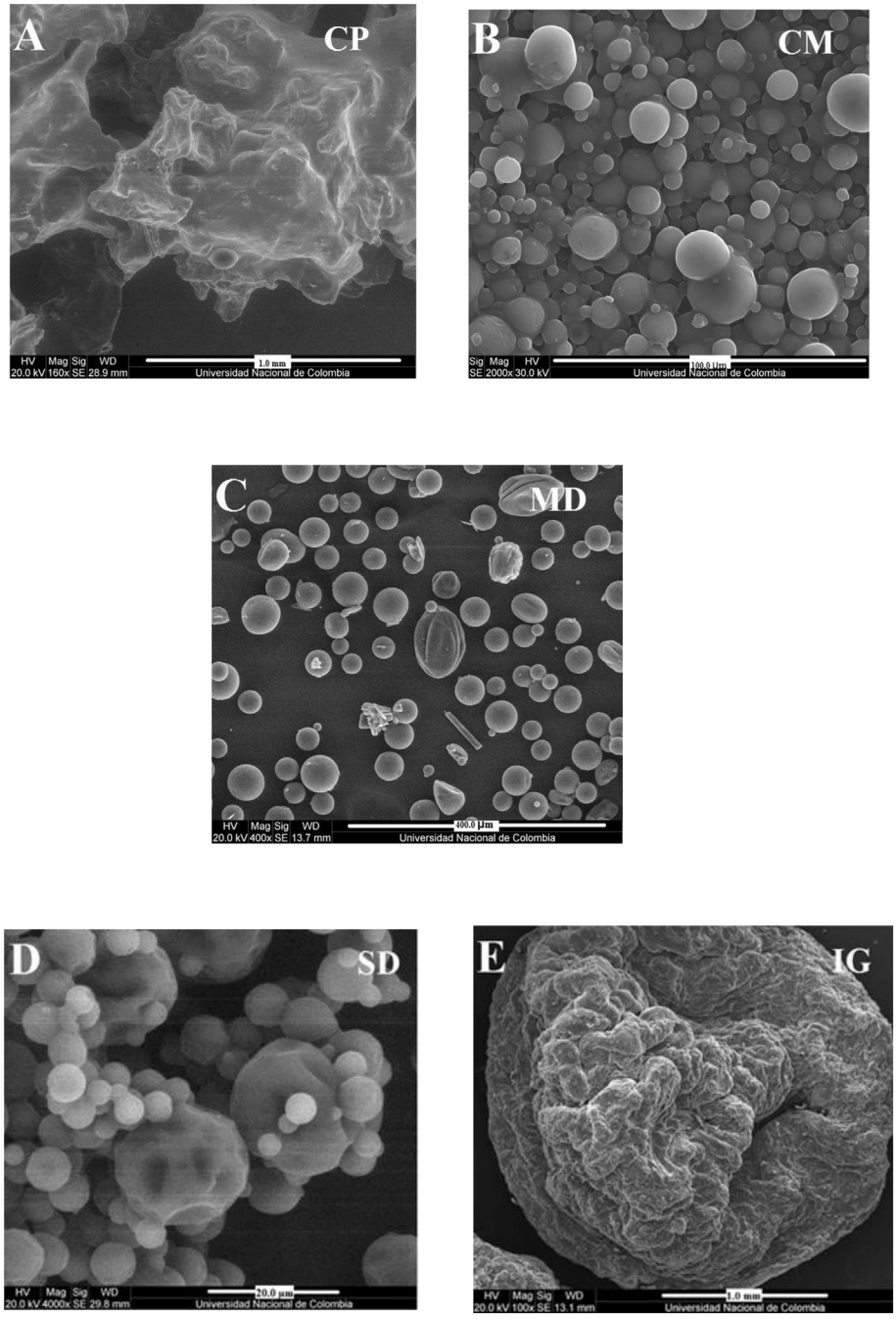

Fig. 1. Scanning electron micrographs of the starting materials and encapsulates. (A) Cactus pear lyophilised pulp (CP) at 160×, (B) cactus cladode mucilage (CM) at 2000×, (C) maltodextrin DE 20 (MD) at 400×, (D) SD sample at 4000×, and (E) IG sample at 100×.

Important differences were found in the particle size of all (encapsulates) powders analysed by SEM (Fig. 1), revealing smaller particles for SD encapsulates.

Morphological analysis of encapsulates is important because the shape and size of particles have strong effect on the fluidity of powders and their solubility. The spherical nature of the particles allows the powder to flow better, and smaller microparticles are more easily solubilised in a suitable solvent, compared to larger particles, due to their higher surface area. SEM analysis shows an extended matrix with irregular shape containing internal cavities for CP sample, as consequence of the lyophilisation process (Fig. 1A). This drying process allows removing water slowly at low temperature and pressure, typically without formation of micro-drop. In contrast, both (CM) cladode mucilage (Fig. 1B) and (MD) maltodextrin (Fig. 1C) samples show spherical microparticles with smooth surface, although more agglomerated particles were observed for the former sample. This agglomeration perhaps is a result of the water absorption of this material (more hygroscopic matrix). In general, this behaviour depends on the chemical properties of surface, and can be reduced through drying (Otálora et al., 2015). However, non-agglomerated particles with more uniform appearance were observed for MD sample (morphology probably acquired from the industrial spray drying of this commercial product), but in both cases the two matrices reveal their potential ability to form microcapsules. The SEM micrographs of SD and IG are shown in Fig. 1D and E, respectively. It is clear that the morphology of the products varied significantly depending on the applied drying method. As it is shown in Fig. 1D, the SD solid has microparticles with spherical or spheroidal shapes and non-smooth surface showing dents. This is likely

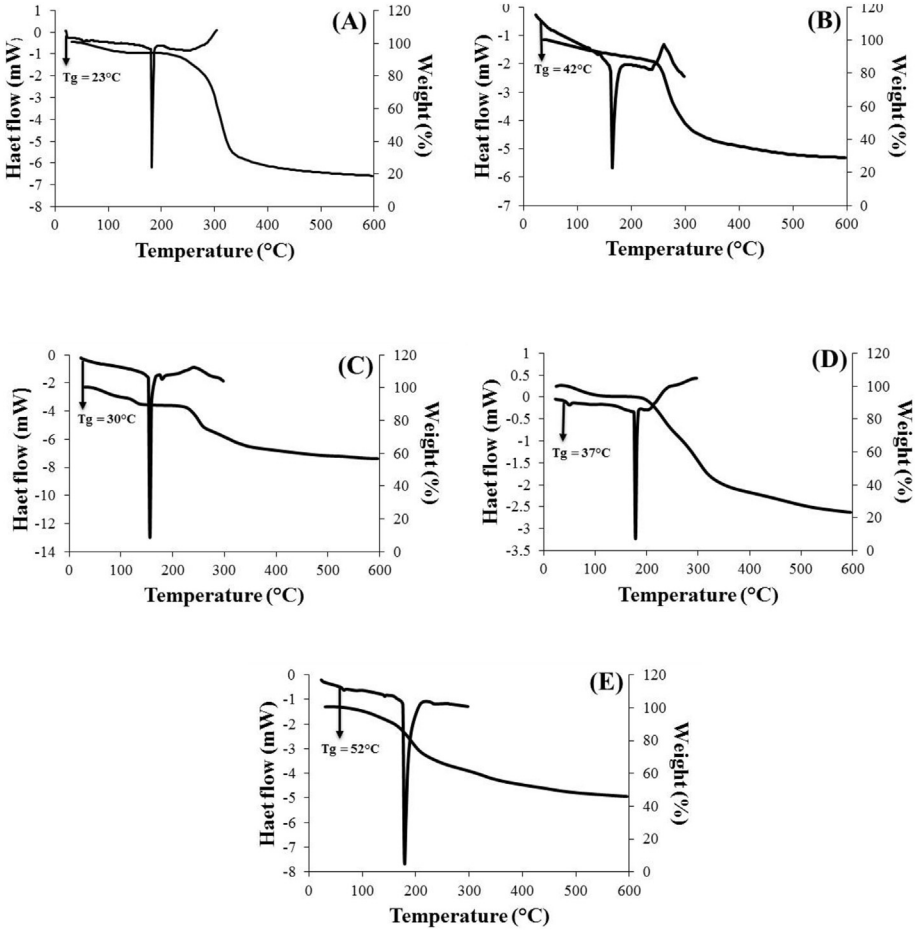

Fig. 2. TGA–DSC profiles for maltodextrin (A), cladode mucilage (B), sodium alginate (C), encapsulated by ionic gelation, IG (D) and microcapsulated by spray drying, SD (E). Glass transition temperature (T_g) is indicated in each graph.

due to the contribution of CM content with high molecular weight, which prevents the surface contraction avoiding the formation of regular spheres (Otálora et al., 2015). Particle morphology of the IG sample (Fig. 1E) showed collapsed irregular particles with abundant roughness and cracks on the surface. This suggests the cracking of the polymer network probably due to tensions promoted by shrinking during drying. Drying removes the water and causes the collapsing of bead walls (Otálora et al., 2016). Different particle sizes were observed for the encapsulate powders obtained by the two methods, being larger (1 to 3 mm) for IG than those of the SD powder (2.5 to 20 μm). This is important because of some chemical and physical properties may be sensitive to the particle size of powders.

Simultaneous thermal analyses (TGA–DSC) of MD, CM, SA, and the SD and IG encapsulates are shown in Fig. 2. The glass transition temperature (T_g) in MD, CM and SA were of 23, 42, and 30 °C, respectively (Fig. 2A, B, and C). In MD the T_g value could be attributed to the lower molecular weight of shorter chains contained in maltodextrins (Kasapis, 2005). It is well known that the larger chain of polymers the higher value of glass transition temperature, and consequently the T_g value increases with increasing molecular weight of encapsulating agents (Medina-Torres, Brito-De La Fuente, Torrestiana-Sánchez, & Katthain, 2000; Vicini, Castellano, Mauri, & Marsano, 2015). Thus, different values of T_g for the encapsulating agents are revealing different molecular weight with varied intermolecular interactions. For example, both mucilage and maltodextrin are different types of polysaccharides, whose structures are able to form interactions such as dipole–dipole and hydrogen bonding with molecules like the betaxanthins and water. These interactions cause disruption in polymeric aggregations, modifying the size of molecular aggregates of wall materials, and yielding slight variation in the glass transition temperatures (Otálora et al., 2015). In CM the Tg value perhaps can also be influenced by either the amorphous nature or vaporization of water, indicating the presence of hydrophilic groups (Rivera-Corona, Rodríguez-González, Rendón-Villalobos, García-Hernández, & Solorza-Feria, 2014). In sodium alginate (SA) the Tg value could also be affected by the complex desorption and dehydration processes. All the DSC profiles obtained for both raw materials and the encapsulates reveal narrow and pronounced endothermic peaks between 150 and 200 °C, which can be attributed to the melting and partial decomposition (dehydroxylation/depolymerisation) (Archana et al., 2013) followed by volatilisation of melted products (this final event can be observed by strong loss of mass after 300 °C in the TGA curves). Narrow peaks indicate that no gelatinisation process could have occurred in important amount, as result of well-drawn drying processes. The loss of very small quantities of mass (water) before 100 °C, according to the TGA curves, confirms the low content of water in all cases. Furthermore, before 100 °C no endothermic peak (different to that of the glass transition) was observed, which in other cases is frequently found for evaporation of adsorbed or free water. The specific DSC-endothermic peaks were 188 °C (MD), 167 °C (CM), and 156 °C (SA) for the encapsulating materials. An exothermic peak around 250 °C, observed for both SA and CM, perhaps is related to degradation of remained biopolymers that undergo depolymerisation reactions (Mimmo, Marzadori, Montecchio, & Gessa, 2005).

The thermal degradation behaviours of MD, CM, and SA were quantitatively recorded by TGA curves (Fig. 2A, B and C). For MD, 6% of mass was lost in the temperature range of 31–205 °C (small free-water loss, dehydroxylation and partial volatilisations), and 74% of mass was lost between 205 and 585 °C as result of a possible thermal

decomposition of the polysaccharides and the volatilisation of liquids after melting. The CM showed a 12% of mass loss between 37 and 223 °C (because small quantities of moisture, dehydroxylation, and partial volatilisation), and 59% of weight loss in the range of 223 to 580 °C corresponding to the structural decomposition of polymer and volatilisation of liquids from melting. The SA sample showed a similar behaviour, but with a lesser total loss of mass. For this sample, 11% of weight was lost between 30 and 205 °C. In this interval, additional to the above-described phenomena, specifically the breaking of segments of mannuronic acid and guluronic acid may have happened (Sand, Yadav, Mishra, & Behari, 2010). Finally, 33% of mass was lost in the range of 205 to 579 °C. The T_g values of SD and IG encapsulates were ca. 37 and 52 °C (Fig. 2D and E). The DSC curves for these solids also showed an endothermic peak corresponding to the melting of encapsulates, which was noted at 178.60 °C for SD and 179.22 °C for IG. This indicates that the melting temperature of IG was increased regarding SA, slightly improving the thermal stability of the encapsulated material. For SD and IG samples, a significant loss of mass (37 and 58%, respectively) was obtained about 150 to 400 °C (Fig. 2D and E). These mass losses involve all the processes above-described. In addition, the TGA curves indicate that the presence of sodium alginate in the matrix IG gives higher thermogravimetric stability (after 280 °C) than that provided by MD and CM in the SD powder.

The total dietary fibre content (TDF) was measured for the SD, IG and CP samples (Table 1). The value for CP was 8.0%, higher than those reported by Díaz-Medina, Rodríguez - Rodríguez, and Díaz-Romero (2007) for both *Opuntia ficus indica* and *Opuntia dillenii.* The obtained results reveal a significant contribution of sodium alginate, and to lesser extent of mucilage, in TDF contents of the samples IG and SD, respectively. This fact suggests that the TDF content of encapsulates can be controlled by the polymer composition. The inclusion of this dietary fibres in those solids represents a nutritional strategy for the development of health-promoting food ingredients.

3.2. Stability studies of encapsulates

Stability of pigments under different humidity conditions of SD, IG, and CP samples is shown in Fig. 3. These results clearly evidence that the betaxanthin stability decreased with the increase of moisture content. It is also noted that CP sample was less stable than SD and IG encapsulates, thus demonstrating the protective effect of encapsulation. Regarding both encapsulates, SD was more stable than IG, being more significant at high humidity environment. The more hydrophilic nature of sodium alginate facilitates the degradation of IG capsules by effect of hygroscopic environments (Olivas & Barbosa-Cánovas, 2008; Rhim, 2004).

The degradation of betaxanthin in IG encapsulate follows a pseudo first-order behaviour during storage at different values of relative humidity (%RH). According to the literature, two linear sections could be correlated. The first slope represented an initial step with superficial pigment degradation, and the second slope represented a second pseudo-first order step with a slower rate than that of the first stage, corresponding to the internal pigment degradation (Saénz et al., 2009). The degradation of the pigments in SD encapsulate showed only one slope, suggesting that the degradation of both superficial and internal betaxanthins occurred at the same rate.

Kinetic data under different storage conditions of humidity (Table 2) showed that $t_{1/2}$ of the betaxanthin in CP was shorter than those for SD and IG. The k values showed that for CP great losses of pigment were rapidly observed, indicating that SD and IG offers an important protection to the pigment. The reason is the effective encapsulation of betaxanthins within the encapsulating agent, which can avoid the damage induced by a_w, temperature, oxygen, light exposure, among others factors during storage (Herbach, Stintzing, & Carle, 2004). Furthermore, the pigment stability was greatly influenced by the type of encapsulating agent as well as the encapsulation technique.

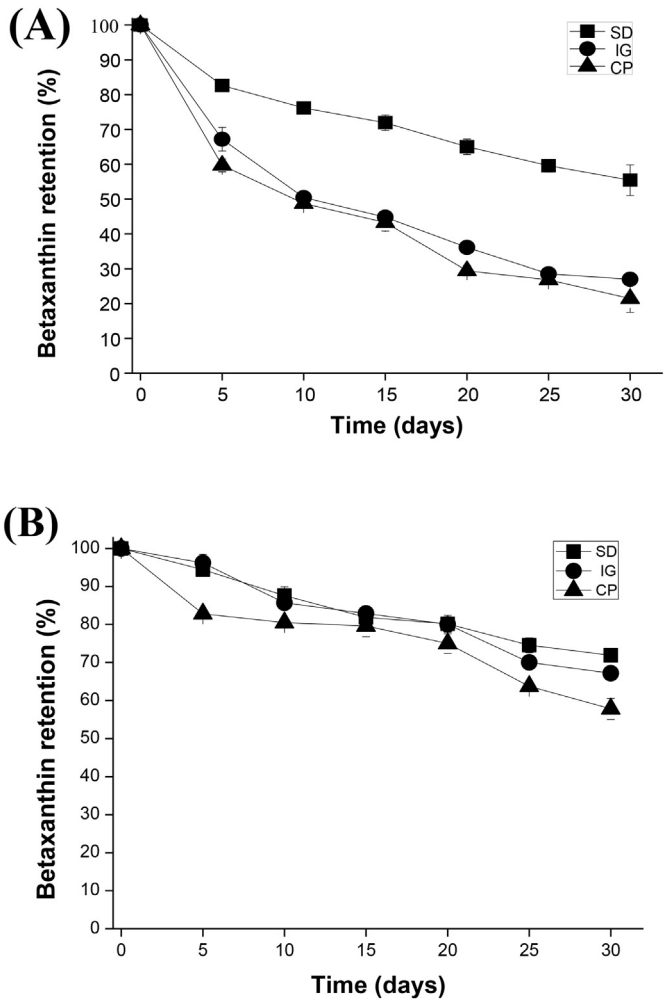

Fig. 3. Percentage of betaxanthin retention during storage (stability test) of the encapsulates (SD and IG) and CP at 18 °C, (A) 90%RH, and (B) 57%RH.

The betaxanthin/betacyanin content in the original extract of *Opuntia megacantha*, and in the control sample CP and the encapsulates SD and IG at initial time, were determined. The encapsulates SD and IG have better storage stability than CP, showing that incorporated encapsulating agent increased the betaxanthin stability because of the protective effect due to encapsulation. However, the betacyanins exhibited higher degradation than the betaxanthins. The orange pigments were more resistant to the conditions of storage in contrast with red-purple pigments. The indicaxanthin content of the samples decreased with the increase of relative humidity in the storage ambient, because of the betaxanthin degradation (Moßhammer, Stintzing, & Carle, 2005).

4. Conclusions

Physical, thermal, and microstructural properties of SD and IG were affected by the encapsulation technique, encapsulating agent, and BE/EA ratio. The encapsulation for spray drying and ionic gelation allowed greater pigment stability at low % RH in contrast to lyophilised pulp, used as a control. The encapsulates showed a high total dietary fibre content and anti-radical activity such giving an added-value to this yellow-orange natural colorant as biofunctional additive. These results are certainly encouraging to develop a cost-effective natural colorant that would be of more attraction to product developers.

Table 2
Kinetic parameters for the degradation of betaxanthins in lyophilised pulp of cactus pear (CP), and in the encapsulates SD and IG during the storage (30 days) at different relative humidity (% RH) conditions.

Sample		Storage conditions					
		$10^2 k_{obsd}$ ($days^{-1}$)*		$t_{1/2}$ (days)*		Correlation coefficients (r)	
		Superficial	Internal	Superficial	Internal	Superficial	Internal
18 °C	CP	–	6.92 ± 0.14[a]	–	10.01 ± 1.06[c]	–	0.93
90%RH	SD	–	1.85 ± 0.07[c]	–	37.48 ± 1.43[a]	–	0.96
	IG	6.84 ± 0.09	3.51 ± 0.06[b]	10.12 ± 0.14	19.74 ± 0.31[b]	0.97	0.95
18 °C	CP	–	1.76 ± 0.01[a]	–	39.37 ± 3.25[c]	–	0.94
57%RH	SD	–	1.11 ± 0.10[c]	–	62.68 ± 3.59[a]	–	0.95
	IG	1.54 ± 0.11	1.53 ± 0.30[b]	45.11 ± 3.31	51.16 ± 1.89[b]	0.99	0.96

– = Not determined.
Different letters in the same column indicate a significant difference between SD (spray dried) and IG (ionic gelation) and control sample (CP) at each condition storage ($p < .05$).
* Values were obtained from plots of the slopes of ln (% retention) vs. time.

Note

The authors declare that there is no any conflict of interests.

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