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FACULTAD DE INGENIERIA DE PROCESOS

ESCUELA PROFESIONAL DE INGENIERÍA DE INDUSTRIAS ALIMENTARIAS

TESIS FORMATO ARTÍCULO

Stabilisation of betalains and phenolic compounds extracted from red cactus pear *(Opuntia ficus-indica)* by spray and freeze-drying using oca *(Oxalis tuberosa)* starch as drying aid

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RESUMEN

El objetivo de este trabajo fue evaluar la estabilización de betalaínas y compuestos fenólicos extraídos de tuna roja por pulverización y liofilización. Después de la extracción hidroetanólica y la eliminación parcial del disolvente bajo presión reducida, los extractos de color se enriquecieron con almidón de oca y maltodextrina como materiales auxiliares de secado en diferentes proporciones (100:0, 70:30 y 50:50, almidón de oca:maltodextrina) antes de pulverizar y liofilizar. Los extractos en polvo microencapsulados obtenidos se caracterizaron por su contenido de humedad, higroscopicidad, solubilidad y morfología. Además, la estabilidad de las betalaínas encapsuladas y los compuestos fenólicos fueron evaluados durante el almacenamiento a temperatura ambiente durante 105 días. Todas las microcápsulas mostraron altas retenciones de betacianinas (69,9 a 86,5% después de 105 días), betaxantinas (72,2 a 81,9%), compuestos fenólicos (46,5 a 63,5%) y capacidad antioxidante (60,1-64,9%, método FRAP; 49,7-57,5%, método ABTS). El sistema con 70:30 almidón: maltodextrina como materiales auxiliares de secado, mostró los valores más altos de retención con respecto al contenido de polifenoles (63,5%), capacidad antioxidante (64,9% según método FRAP) y contenido de betacianina (86,5%), así como una tasa de degradación baja de las betacianinas (1,23 x 10^{-3} $dias^{-1}$) y una vida media prolongada (563 días). Utilizando solo almidón de oca o en combinación con maltodextrina ha demostrado funcionar adecuadamente como agente microencapsulante y estabilizador de pigmentos y antioxidantes derivados de la tuna roja.

Palabras clave: *Tuna roja, Oxalis Tuberosa, compuestos fenólicos, betalaínas, estabilización, métodos de microencapsulación*.

ABSTRACT

The objective of this work was to evaluate the stabilisation of betalains and phenolic compounds extracted from red cactus pear by spray and freeze-drying. After hydroethanolic extraction and partial solvent removal under reduced pressure, the highly coloured extracts were enriched with oca starch and maltodextrin as drying aids in different ratios (100:0, 70:30, and 50:50, oca starch: maltodextrin) prior to spray and freezedrying. The obtained microencapsulated extract powders were characterised by moisture content, hygroscopicity, solubility and morphology. In addition, the stability of the encapsulated betalains and phenolics was evaluated during storage at room temperature for 105 days. All microcapsules showed high retentions of betacyanins (69.9–86.5% after 105 days), betaxanthins (72.2–81.9%), phenolic compounds (46.5–63.5%) and antioxidant capacity (60.1–64.9%, FRAP method; 49.7–57.5%, ABTS method). The system with 70:30 starch:maltodextrin ratio as drying aids showed the highest values of retention regarding the polyphenol content (63.5%), antioxidant capacity (64.9% to FRAP method) and betacyanin content (86.5%), as well as a low degradation rate constant of betacyanins (1,23 x 10^{-3} $days^{-1}$) and a long half-life (563 days). Oca starch used alone or in combination with maltodextrin has been shown to work adequately as a microencapsulating agent and stabilizer of pigments and antioxidants derived from red cactus pear.

Keywords: *Red cactus pear, Oxalis Tuberosa, phenolic compounds, betalains, stabilisation, microencapsulating methods.*

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"Estabilización de betalaínas y compuestos fenólicos extraídos de la tuna roja (*Opuntia Ficus-Indica*) por pulverización y liofilización usando almidón de oca (O*xalis Tuberosa*) como material auxiliar de secado"

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3. Planteamiento del problema

EFECTOS: - La biodiversidad del país no está aprovechada por la industria alimentaria. - La poca utilización de antioxidantes naturales, debido a su baja estabilidad; la cual se ve ampliamente mejorada con la tecnología de microencapsulación.
PROBLEMA: La oca es un cultivo andino nativo de Perú cuyo almidón posee características especiales que son desaprovechadas por la industria alimentaria, específicamente en la tecnología de microencapsulación.
CAUSAS: - Estudios del almidón de oca tienen poco alcance en cuanto al aprovechamiento de sus propiedades a nivel pre-industrial e industrial. - Los almidones que actualmente son empleados por la industria alimentaria pertenecen mayormente a cultivos comerciales. - La tecnología de la microencapsulación en la industria alimentaria del Perú no está muy desarrollada.

La naturaleza fisicoquímica de los antioxidantes de la tuna provoca que estos sean inestables a las condiciones del medio en que se encuentran, lo que genera su pérdida durante los procesos de transformación del alimento, ocasionando que estos sean reemplazados por antioxidantes de origen sintético. ¿Es posible usar el almidón de la oca como material encapsulante para la protección de los antioxidantes extraídos de la pulpa de la tuna?

4. Objetivo general

Evaluación de la estabilidad de betalainas y compuestos fenólicos extraídos de la tuna roja por pulverización y liofilización usando almidón de oca como material auxiliar de secado.

5. Objetivos específicos

- Extraer y caracterizar reológica y morfológicamente el almidón de la oca para su uso como microencapsulante.
- Extraer y medir el contenido total de compuestos fenolicos y la capacidad antioxidante del extracto etanólico de antioxidantes de la pulpa de tuna roja.
- Microencapsular los antioxidantes extraídos de la tuna con el almidón de oca.
- Evaluar el contenido de compuestos fenólicos de las microcapsulas durante 105 días.
- Evaluar la capacidad antioxidante de las microcápsulas durante 105 días.
- Evaluar el contenido de betalainas de las microcapsulas durante 105 dias.
- Identificar la formulación adecuada de polisacáridos (almidón de oca: maltodextrina) que ofrece mejor estabilidad a las microcapsulas.
- Evaluar el rendimiento de la elaboración de las microcápsulas mediante los dos métodos: spray drying y freeze drying.
- Evaluar las características físicas de las microcapsulas: humedad, grado de solubilidad, higroscopicidad, morfología.

6. Descripción del Proyecto

El objetivo de este trabajo fue evaluar la estabilización de betalaínas y compuestos fenólicos extraídos de tuna roja por pulverización y liofilización. Después de la extracción hidroetanólica y la eliminación parcial del disolvente bajo presión reducida, los extractos de color se enriquecieron con almidón de oca y maltodextrina como materiales auxiliares de secado en diferentes proporciones (100:0, 70:30 y 50:50, almidón de oca:maltodextrina) antes de pulverizar y liofilizar. Los extractos en polvo microencapsulados obtenidos se caracterizaron por su contenido de humedad, higroscopicidad, solubilidad y morfología. Además, la estabilidad de las betalaínas encapsuladas y los compuestos fenólicos fueron evaluados durante el almacenamiento a temperatura ambiente durante 105 días.

7. Justificación

Arequipa es uno de los mayores productores de tuna en el Perú (Agroarequipa, 2017; Minagri, 2011). Este fruto se encuentra en el grupo de alimentos funcionales por su contenido de compuestos antioxidantes como flavonoides y polifenoles (Alba et al., 2014) y por ser una de las pocas fuentes naturales de betalaínas que existe (Sáenz et al., 2008). Sin embargo, la producción y el procesamiento de este fruto se mantiene a bajos niveles debido a su corta vida en anaquel, su susceptibilidad a temperaturas bajas y la presencia de espinas en su cáscara (Rodríguez 2002, citada por Alba et al., 2014), lo que conduce al desaprovechamiento no solo de la fruta, sino de los compuestos bioactivos que este contiene.

Los antioxidantes son inestables frente a condiciones adversas de temperatura, luz, pH y oxígeno (Castellar et al., 2003, citado por Sáenz et al., 2008), lo que provoca la pérdida y degradación del compuesto y, consecuentemente, una baja ingesta de antioxidantes entre la población peruana. En este punto, es necesario mencionar el importante rol que los antioxidantes cumplen en la salud humana, ayudando a prevenir enfermedades crónico-degenerativas (Coronado et al., 2015).

Estudios anteriores han reportado el uso de la microencapsulación como una técnica capaz de mejorar la conservación y disminuir la pérdida de la capacidad antioxidante. En efecto, la microencapsulación consiste en encapsular compuestos bioactivos con un biopolímero (Desai et al., 2005), como la maltodextrina y el almidón. Diversos autores han reportado resultados positivos en la microencapsulación empleando almidones de cultivos comunes como la papa y el arroz (Tapia, 2017; Arteaga, 2016 y Trindade, 1999), no obstante, siendo la oca un cultivo andino sub-utilizado, de precio similar al de la papa y con el potencial de generar desarrollo para sus productores (Tapia y Fries, 2007), este trabajo se enfoca en la evaluación del almidón de oca como material microencapsulante para la conservación de antioxidantes extraídos de la pulpa de tuna morada.

En el Perú son pocos los avances en el uso de la tecnología de microencapsulación (Silva et.al, 2018), la cual permite el desarrollo de alimentos con propiedades nutricionales mejoradas, con una incidencia directa en la salud y calidad de vida de los consumidores. Con los resultados del presente estudio se llenará un vacío de conocimiento que hasta el momento existe respecto al comportamiento del almidón de este tubérculo como material de barrera en la microencapsulación, además de generar un impacto a nivel social y económico al estimular el cultivo y procesamiento industrial de materias primas que se encuentran en el marco de biodiversidad del país.

8. Delimitación

Para el presente trabajo se trabajará con polifenoles extraídos de la pulpa de tuna, sin embargo se pueden hacer pruebas con otro tipo de fruta u otras moléculas nutracéuticas. Por otro lado, solo se evaluará la capacidad antioxidante del fruto y de las microcápsulas, mas no se evaluará su aplicación en producto alimentario.

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Article

FOOD SCIENCE AND TECHNOLOGY INTERNATIONAL

Stabilisation of betalains and phenolic compounds extracted from red cactus pear (*Opuntia ficus-indica*) by spray and freeze-drying using oca (*Oxalis tuberosa*) starch as drying aid

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Abstract

The objective of this work was to evaluate the stabilisation of betalains and phenolic compounds extracted from red cactus pear by spray and freeze-drying. After hydroethanolic extraction and partial solvent removal under reduced pressure, the highly coloured extracts were enriched with oca starch and maltodextrin as drying aids in different ratios (100:0, 70:30, and 50:50, oca starch: maltodextrin) prior to spray and freeze-drying. The obtained microencapsulated extract powders were characterised by moisture content, hygroscopicity, solubility and morphology. In addition, the stability of the encapsulated betalains and phenolics was evaluated during storage at room temperature for 105 days. All microcapsules showed high retentions of betacyanins (69.9–86.5% after 105 days), betaxanthins (72.2–81.9%), phenolic compounds (46.5–63.5%) and antioxidant capacity (60.1–64.9%, FRAP method; 49.7–57.5%, ABTS method). The system with 70:30 starch:maltodextrin ratio as drying aids showed the highest values of retention regarding the polyphenol content (63.5%), antioxidant capacity (64.9% to FRAP method) and betacyanin content (86.5%), as well as a low degradation rate constant of betacyanins (1.23×10^{-3} days^{-1}) and a long half-life (563 days). Oca starch used alone or in combination with maltodextrin has been shown to work adequately as a microencapsulating agent and stabilizer of pigments and antioxidants derived from red cactus pear.

Keywords

Red cactus pear, *Oxalis tuberosa*, phenolic compounds, betalains, stabilisation, microencapsulating methods

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INTRODUCTION

Peru is home to a spectacular number of species of flora and fauna. The wide variability of altitude, topography, climate and soils render Peru an ideal place for cultivation of numerous crops (Scott, 2011). However, most of the potential crops that find their ideal habitats in the Peruvian Andes are yet mostly underexploited, even though they are a rich source of nutrients and bioactive compounds. The lack of processing technology and the post-harvest perishability of many fruits results in the underutilisation of natural resources and, consequently, a huge loss for the development of Andean communities.

Opuntia ficus-indica (L.) Mill., commonly called prickly pear, is a species of cactus that has been cultivated in arid and semiarid regions worldwide. Its fruit is a rich natural source of betalains, a class of red and yellow coloured plant pigments (Sáenz et al., 2009).

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In addition, these compounds are potentially health-promoting antioxidants and of increasing interest in the food industry, as they represent an alternative to synthetic red colourants (Kuti, 2004; Mobhammer et al., 2006; Obón et al., 2009; Sáenz et al., 2009; Stintzing and Carle, 2004). However, betalains are unstable when exposed to a series of detrimental factors, such as temperature, pH, water activity and exposure to light and oxygen (Castellar et al., 2003; Castro et al., 2014; Kuck and Noreña, 2015; Mobhammer et al., 2006; Vergara, 2013). This instability hinders the storability and, thus, usability of betalain-rich foodstuff or extracts for the food industry, highlighting the need to protect them by efficient techniques.

At respect, microencapsulation is a method for preserving and stabilising a substance of interest, to eventually release it when opportune (Desai and Park, 2005). This can be achieved by different treatments, such as spray-drying which has long been used for encapsulating antioxidants, being advantageous for its rapid, simple, economic and easy-to-scale up characteristics; another treatment is freeze-drying, also addressed as lyophilisation, which uses freezing, sublimation and desorption for dehydrating in order to stabilize the substance of interest, having the use of low temperatures as its main advantage (Ozkan et al., 2019). In both drying approaches, the choice of appropriate drying aids is of utmost importance, as it will strongly influence the drying behaviour, the release profile and ultimately its capacity for retaining the bioactive compounds within the matrix (Ballesteros et al., 2017; Obón et al., 2009).

In order to cope with undesired product properties such as an often fructose-related low glass transition temperature or product hygroscopicity, mainly carbohydrate polymers have been tested as drying aids for spray and freeze-drying for microencapsulation of natural extracts. Maltodextrin represents one of the most common, among dextrose, Arabic gum, modified starches, proteins and others (Ballesteros et al., 2017; Cai and Corke, 2000; Ezhilarasi et al., 2013). Nonetheless, to the best of the authors' knowledge, detailed studies on the stabilisation of betalain extract from *Opuntia* spp. as depending on the drying aid, particularly including native starches, are scarce.

In this study, usage of starch derived from *Oxalis tuberosa* for the microencapsulation of betalainic extract from red fleshed *O. ficus-indica* fruit is proposed, mainly due to earlier reports on particularly favourable rheological and physicochemical properties (Bellido et al., 2017; Cruz et al., 2016; Zhu and Cui, 2020). Therefore, the aim was to evaluate the stability of the compounds of interest formulated into a microcapsular powder by spray and freeze-drying, using different mixtures of *O. tuberosa* starch and maltodextrin as drying aids.

MATERIALS AND METHODS

Materials

Cactus pear fruits (*O. ficus-indica*) were obtained from a plantation located in Santa Rita de Siguas (Arequipa, Peru). *O. tuberosa* tubers were purchased from a local market (Arequipa, Peru), and maltodextrin (DE 10) was purchased from Zhucheng Dongxiao Biotechnology (Shanghai, China).

All other chemicals and reagents were of analytical grade from Sigma Chemical Co.

Extraction and characterisation of *Oxalis tuberosa* starch

The mechanical extraction of oca starch was based on the methodology developed by the International Potato Center (CIP) and as mentioned by Paulet et al. (2009) and Tarazona (1995), with some modifications. In brief, the tubers were manually washed, cut into small pieces and blended with distilled water (ratio 1:2 w/v) using a blender (Osterizer, Milwaukee, USA). Larger solid particles were separated with a mesh, while the filtrate was allowed to stand for 4 h prior to decanting the supernatant. The remaining solids were washed with distilled water (ratio 1:1 solids:water) and left to stand for 1 h. The washing procedure was repeated three times, obtaining the starch as a white wet paste. The wet starch was dried at 45 °C for 24 h into a drying oven (Binder, ED56, Tuttlingen, Germany) until obtaining a moisture of ca. 10–13%. The dried starch was homogenized by crushing it in a mortar and sifting it using a mechanical sieve (106 μm mesh, Tyler, ASTM E-11, USA). The *O. tuberosa* starch was packed in a polythene bag.

Solubility and swelling power (SP) were determined according to the method described by Leach et al. (1959) and Valcárcel et al. (2013). Aqueous suspensions of 2% of starch (w/v) were placed in a centrifuge tube, at room temperature (20 °C) and in a water bath at 40 °C for 30 min, constantly shaking. The tubes containing the suspensions were centrifuged at 3000 r/min for 15 min. Supernatants were transferred to Petri dishes, weighted, then put in the oven at 105 °C for 24 h. Samples were cooled in the desiccator and weighted again. The difference between the initial and final weight of the Petri dish was considered as the weight of soluble starch expressed as percentage. The SP was calculated using equation (1), considering the weight of the settled paste obtained after centrifugation

$$\%\ Swelling\ Power = \frac{Weight\ of\ wet\ paste \times 100}{Weight\ of\ sample \times (100\% - \%\,Solubility)} \quad (1)$$

The morphological characterisation of oca starch was performed by scanning electron microscopy (SEM; Zeiss Evo, MA10, Oberkochen, Germany), following the method described by Cruz et al. (2016). Samples were observed at 2060 × magnification at 12 kV.

Gelatinisation temperature was determined by making up 10 g of starch to 100 mL with water. An aliquot of 50 mL of the resulting dispersion was transferred to a beaker and then placed in a water bath at 85 °C to allow slow heating. Stirring constantly, the temperature was monitored during the whole process. Gelatinisation temperature was the temperature range in which the starch formed a paste (Aristizábal and Sánchez, 2007).

Paste clarity was determined based on the method described by Aristizábal and Sánchez (2007) and Craig et al. (1989). Samples of 200 mg of starch were suspended in 20 mL of distilled water and placed in a boiling water bath for 30 min, manually shaking the suspension every 5 min. The suspension was then transferred to spectrophotometer cuvettes and allowed to cool to room temperature for 5 min. The transmittance (%) was read at a wavelength of 650 nm, using distilled water as a blank.

Betalain extract preparation

The betalain-rich prickly pear extract was obtained by using the method described by Sáenz et al. (2009). Some cactus pear fruits were selected by visual inspection and the spines were removed. A batch of 2 kg of fruit was then manually peeled and the obtained pulp was cut into cubes of ca. 1 cm^3 (yield of fruit pulp: 63% wet basis rel. to whole fruit). In order to improve the degree of cell membrane permeabilisation and extraction yield, the cubes were frozen at −20 °C (Azeredo, 2009; Robert et al., 2015). Afterwards, the pulp cubes were carefully crushed, without tearing the seeds, and ethanol 80% v/v was added until reaching 1:1 w/v pulp-to-ethanol ratio. Maceration with continuous stirring was carried out for 1 h. The reddened liquid was separated from the seeds and pulp remnants by using a simple kitchen sieve. The extraction procedure was repeated twice until the remaining solids were only faintly red. Subsequently, an aliquot of 500 mL of the extract was concentrated in a rotary evaporator (Buchi, RII, Flawil, Switzerland) at 45 °C for 30 min under reduced pressure (−480 mm Hg). Concentration was performed until 5% to 10% of alcohol content (v/v) was reached in the extract. The extract was then centrifuged (Centurion Scientific, Pro-Analytical C2004, Chichester, UK) at 4000 r/min for 15 min. The supernatant was separated and then stored at 4 °C until further processing.

Analyses of cactus pear pulp and the betalain extract

The moisture content, soluble solids (°Brix), pH and % titratable acidity were determined according to the methods of the Association of Official Analytical Chemists (AOAC, 1996). The total phenolic content was determined by following the Folin-Ciocalteau method (Obanda et al., 1997; Rufino et al., 2010). The antioxidant capacity was determined by the ferric reducing ability of plasma (FRAP) and 2,2′-azino-bis-(3-ethylbenzthiazoline-6-sulfonic acid) (ABTS) methods (Loizzo et al., 2012). The betalain content was quantified according to the method proposed by Stintzing et al. (2005). Betacyanins and indicaxanthins were monitored at 535 and 484 nm, respectively.

Microencapsulation

Oca starch and maltodextrin were mixed in dry form in proportions of 100:0, 70:30 and 50:50, respectively. Maltodextrin was added as a processing aid to modulate the properties of oca starch, mainly for reducing the viscosity of the solution fed to the dryers while simultaneously maintaining the desired targeted dry matter. Gelatinisation was carried out by dispersing 20 g of the polysaccharides mixture in deionized water at a ratio of 1:15 w/v under heating (85 °C) using a water bath (Lab. Companion, BW-20 G, MA, USA). The gelatinised paste was cooled down to a temperature of 45 °C prior to adding 80 mL of the cactus pear extract and homogenising the resulting mixture with a blender (Recco, RMIN-989 W, China). Subsequently, microencapsulation by spray drying was performed with a B290 spray dryer (Büchi, Flawil, Switzerland). The inlet temperature was 130 °C at a feed rate of approximately 6 mL/min, 100% aspiration and 80 °C outlet temperature. The spray dryer feed vessel was continuously stirred using a magnetic stirrer (CAT, M20, Staufen, Germany). The spray dried powder with the microencapsulated betalain extract was collected and stored at room temperature in dark, hermetically sealed, tri-laminated bags until future analyses.

Additionally to spray drying, microencapsulation was performed by freeze-drying. The betalain extracts were enriched with the aforementioned drying aids as described above, in this case the ratio of dissolution of polysaccharides mixture in deionized water was 1:7 w/v, then placed in the freeze dryer (Labconco, FreeZone Triad, Kansas, USA) after pre-freezing at −40 °C. Sublimation temperatures ranged from −25 °C to −5 °C at a ramp of 0.65 °C/min. The process lasted approximately 24 h. The freeze dried powder with the microencapsulated betalain extract was collected and stored at room temperature in the dark in hermetically sealed, tri-laminated bags until future analyses.

Each microencapsulation method was repeated three times. The abbreviations used in the following are ''SD'' for spray-drying and ''FD'' for freeze-drying. The given ratios always name oca starch before maltodextrin (100:0, 70:30 and 50:50).

Encapsulation yield

The yield of the spray-dried and freeze-dried microcapsule powder was determined as suggested by Otálora et al. (2015) and was expressed as the percentages of the mass of collected microcapsules after drying relative to the mass of the solids in the extract fed to the system (dry-matter-base).

Characterisation of the microcapsules' powders

Moisture content. The moisture content of the microcapsules was determined gravimetrically by drying 1 g of product, in duplicate, at 105 °C until constant weight (AOAC, 1990).

Solubility. The solubility of the particles was determined according to the method described by Leach et al. (1959). In brief, aqueous suspensions of 2% powder (w/v) were placed in centrifuge tubes at room temperature (20 °C) and constantly stirred for 30 min. The samples were then centrifuged at 3000 r/min for 15 min at 20 °C. Subsequently, the supernatant was decanted into Petri dishes and dried in a drying oven (Binder, ED56, Tuttlingen, Germany) at 105 °C for 24 h, cooled in the desiccator and then weighed. The weight of soluble microcapsules is considered to be the difference between the final and the initial weight of the Petri dish with the sample, divided by the weight of the initial powdered sample, expressed as a percentage.

Hygroscopicity. Hygroscopicity was determined following the method described by Cai and Corke (2000), including some modifications as reported by Rodriguez (2011). Samples of 1 g of the obtained reddish powder were placed in Petri dishes at 25 °C, which were placed inside a desiccator containing a vessel with a saturated solution of NaCl to achieve a relative humidity of 76% within the desiccator. Samples were stored for seven days, weighing them every day. Hygroscopicity was expressed as grams of moisture absorbed by 100 g of dry solids (g/100 g).

Particle morphology. The morphology of microcapsules was evaluated by using a SEM (Zeiss Evo, MA10, Oberkochen, Germany) operating at 12 kV, coating the samples by silver sputtering before their examination. The morphology of the particles was observed at $515\times$ magnification for freeze-dried powder, and at $3060\times$ for spray-dried powder. The sample must not have humidity greater than 10% (Cruz et al., 2016).

Evaluation of the stability of microcapsules

Storage stability was tested during 105 days. Samples were taken after 1, 8, 12, 18, 24, 32, 41, 50, 68, 88 and 105 days. The microcapsules were stored in the dark in hermetically sealed, tri-laminated bags at room temperature. Triplicate samples were analysed for each time point, in order to evaluate total polyphenol content (TPC), antioxidant activity and betalain content. The following assays were performed under dimmed light:

Total polyphenol content. The TPC was quantified by following the Folin–Ciocalteau method, described by Obanda et al. (1997) and Rufino et al. (2010). A sample of 1 ± 0.05 g of the microcapsules was dissolved in 20 mL of distilled water by stirring at room temperature for 5 min. The solution was then filtered with a 0.45 μm OlimPeak filter with a polyvinylidene difluoride membrane (Teknokroma Barcelona, Spain). An aliquot of 1 mL of the filtrate was mixed with 1 mL of Folin–Ciocalteau reagent (1:3), 2 mL of a 20% w/v sodium carbonate solution and 2 mL of ultrapure water. The samples were allowed to stand for 1 h. Then, the absorbance at 700 nm was read on a spectrophotometer (Silogex, SP-UV1100, Rocky Hill, USA). A standard curve of gallic acid in a range of 50–550 mg/L was built for quantification.

Antioxidant power iron reduction (FRAP). The FRAP method measures the change in absorbance that occurs when the 2,4,6-tripyridyl-*s*-triazine (TPTZ)–Fe3 + complex is reduced to the TPTZ–Fe2 + form in the presence of antioxidant compounds (Benzie and Strains, 1996). The FRAP assay was conducted as reported by Loizzo et al. (2012). The FRAP reagent was prepared by mixing 2.5 mL of 10 mM TPTZ dissolved in 40 mM HCl plus 2.5 mL of 20 mM Iron trichloride hexahydrate and 25 mL of 0.3 M acetate buffer (pH 3.6). An aliquot of 1.8 mL of FRAP reagent was then mixed with 0.2 mL of the sample, consisting of 1 ± 0.05 g of the microcapsules dissolved in 20 mL of distilled water. This solution was placed in a heating bath at 37 °C for 30 min prior to reading the absorbance at 595 nm. Aqueous solutions of known $FeSO_4$ concentration, in the range of 500–3000 μmol/L, were used for building a standard curve.

$ABTS^+$ free radical scavenging capacity. The method proposed by Re et al. (1999) was followed, including some modifications (Loizzo et al., 2012; Rodriguez, 2011). Briefly, the ABTS radical cation ($ABTS^+$) was

prepared by mixing 10 mL of a 7 mM ABTS solution with 166 μL of a 150 mM potassium persulfate solution. This mixture was stored in the dark at room temperature for 12 h before using. The $ABTS^+$ solution was diluted with ethanol to yield an absorbance of 0.70 ± 0.05 at 734 nm. An aliquot of 20 μL of the aforementioned sample solution was mixed with 2 mL of the diluted ethanolic $ABTS^+$ solution and then placed in a heating bath at 30 °C for 6 min, prior to reading the absorbance at 734 nm. A standard curve of Trolox, in a range of 100–2000 μmol/L, was used for calibration and quantification.

Betalain content. Betalains were quantified according to the method proposed by Stintzing et al. (2005). The betacyanins and betaxanthins were monitored at 535 and 484 nm, respectively. An aqueous solution of the microcapsule powder was dissolved with McIlvaine buffer (pH 6.5) to yield absorbance values of $0.9 \leq A \leq 1.0$ in their respective maximum absorbance. The betalains content (BC), including betaxanthins and betacyanins, was calculated using equation (2) as suggested by Castellanos Santiago and Yahia (2008)

$$\mathrm{BC\,(mg/L)} = ((A \times \mathrm{DF} \times MW \times Vd/\varepsilon \times L \times Wd)) \quad (2)$$

where A is the absorbance value, DF is the dilution factor, Vd is the sample volume (mL), L is the length of the cuvette reading (1 cm) and Wd is the sample weight (g), MW is the molecular weight and ε is the molar extension coefficient (for betanins, $MW = 550$ g/mol, $\varepsilon = 60{,}000$ L/(mol.cm) in water; $\lambda = 538$ nm; and for indicaxanthins, $MW = 308$ g/mol; $\varepsilon = 48{,}000$ L/(mol.cm) in water; $\lambda = 480$ nm).

Betalain retention. The pigment retention was calculated by using equation (3), as proposed by Otálora et al. (2015), considering the initial content of betalain in the sample and the final content after a determined amount of time. This was done in order to assess the loss of pigment over time

$$\textit{Betalain Retention}\ (\%) = \frac{\textit{Amount of betalain over time}}{\textit{Initial amount of betalains}} \times 100 \quad (3)$$

Betacyanin degradation rate and half-life. The data obtained on Betalain retention were used to derive the reaction rate constant (k) as well as the half-life ($t½$) of the respective compounds using a first-order kinetic model according to the following equations (4 and 5)

$$\ln C = \ln C_0 - kt \quad (4)$$

$$t^{1/2} = 0.693/k \quad (5)$$

where C is the amount of betacyanins at time t, C_0 is the initial amount of betacyanins in the microcapsules and k is the first-order degradation rate constant (Vergara, 2013).

Statistical analysis

Data are expressed as means ± standard deviation for triplicate ($n = 3$) and duplicate ($n = 2$) determinations, using StatGraphic Centurion XVI version 16.1.11 (StatGraphics Technologies, Inc., Virginia, USA). Significant differences among means were evaluated by analysis of variance and the Tukey multiple range test, considering the probability level $p < 0.05$ as a significant difference. Comparisons between two groups were evaluated by the Student's t-test, using SPSS software (IBM SPSS statistics 21).

RESULTS AND DISCUSSION

Starch extraction and characterisation

A total yield of 8.3% in w.b. of starch was obtained from *O. tuberosa* tubers, being within a range reported by Tapia and Fries (2007), who found yield values between 6.7% and 9.1%. The authors stated that these values are largely influenced by factors such as the variety and maturity of the tuber. Some physical characteristics of oca starch are shown in Table 1. The gelatinisation temperature of *O. tuberosa* starch was found to be between 61.7 °C and 63.4 °C, in agreement with Torres et al. (2011) and Zhu and Cui (2020). In a general view, the temperature of gelatinisation of oca starch is lower than the temperature reported for starches extracted from maize (67.7 °C), rice (67.4 °C) and tapioca (65.3 °C; Jenkins and Donald, 1998), and from some other Andean tubers, such as potato (65.5 °C), mashua (65.7 °C) and ulluco (63.1 °C; Torres et al., 2011). Solubility and SP of starch granules are directly proportional to the temperature at which the aqueous suspension of starch is subjected (Rondán and Finardi, 2009). According to Hoover (2001), when a suspension of starch

Table 1. Characterisation of the physical properties of *Oxalis tuberosa* starch.

Analysis	Value
Solubility (%; at 60 °C)	13.9 ± 0.5
Swelling capacity (%; at 60 °C)	11.9 ± 0.5
Morphology	Oval, elliptic
Gelatinisation temperature (°C)	61.7–63.4
Pasta clarity (%*T*)	13.2

is heated in water, the crystalline structure is broken up due to the thermal and water-driven disruption of starch-to-starch hydrogen bonds, causing an increase in swelling and solubility of the granules.

Oca starch paste clarity was measured to be at 13.2% *T* (transmittance). This is a low value when compared to potato starch paste, whose clarity is 96% *T*, and wheat starch pasta, whose clarity is 71% *T* (Craig et al., 1989). According to Aristizábal and Sánchez (2007), starch pastes whose transmittance values are less than 40% are considered opaque or cloudy. Low values of clarity might be due to the presence of granular remnants in the paste, which consequently causes an inhomogeneous refraction (Craig et al., 1989).

Native oca starch granules were found to be oval and elliptical in shape, with rounded edges and apparently smooth surface (Figure 1; Bellido et al., 2017; Hernandez Lauzardo et al., 2004).

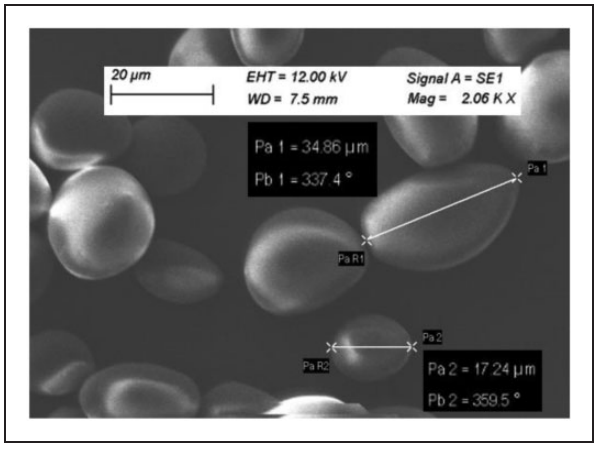

Figure 1. Morphology of starch granules from *Oxalis tuberosa* tubers. Pa: distance between points; Pb: angle of the line.

Extraction and characterisation of antioxidants from red cactus pear

Results on the physicochemical characterisation of red *O. ficus-indica* are shown in Table 2. Moisture content, soluble solids, pH and acidity values were consistent with those reported by Piga (2004), Reyes Munguia et al. (2014), Robert et al. (2015) and Sáenz et al. (2009). The TPC of cactus pear pulp and antioxidant extract are 719 and 693 mg of gallic acid/L, respectively. These values are similar to those reported by Morales et al. (2009) and Robert et al. (2015), but lower to TPCs reported by Sáenz et al. (2009). The chemical composition of the *Opuntia* fruits varies according to the plant species, cultivation area, degree of maturity and other factors (Andreu et al., 2017; Sáenz, 2000).

Regarding the antioxidant activity determined by the FRAP method, values of 20.5 and 18.0 µmol $FeSO_4$/g were recorded for the red cactus pear pulp and the ethanolic extract, respectively. These are higher values than those reported for the pineapple (14.50), cherry tomato (12.15), kiwi (10.75), but lower than guava (23.80) and pomegranate (25.57), all being expressed in µmol $FeSO_4$/g (Fu et al., 2011). The antioxidant activity was also evaluated by the ABTS method, obtaining values of 0.98 and 0.94 mmol of trolox/100 g for the red cactus pear pulp and the antioxidant ethanolic extract, respectively. These values are higher than those reported by Albano et al. (2015), i.e., 0.61 mmol of trolox/100 g.

The contents of betacyanins and betaxanthins of the cactus pear extract were similar to those reported by Stintzing et al. (2005), who obtained 431.0 BE mg/L and 195.8 mg IE mg/L respectively. On the other hand, Sáenz

Table 2. Characterisation of the pulp and the ethanolic antioxidant extract of red cactus pear (*Opuntia ficus-indica*).

Analysis	Purple cactus pear pulp	Ethanolic extract of red cactus pear
Moisture (%)	85.8 ± 0.1	–
pH	6.06 ± 0.01	–
Acidity (% citric acid)	0.04 ± 0.01	–
Soluble solids (°Brix)	13.4 ± 0.1 b	20.8 ± 0.2 a
Total polyphenol content (mg GAE/L)	719.0 ± 4.3 a	693.2 ± 4.7 b
Antioxidant capacity (FRAP; µmol $FeSO_4$/g)	20.5 ± 0.2 a	18.0 ± 0.7 b
Antioxidant capacity (ABTS; mmol Trolox/100 g)	0.97 ± 0.06 b	0.94 ± 0.06 b
Betacyanins (BE mg/L)	439.5 ± 3.1 a	426.6 ± 1.7 b
Betaxanthins (IE mg/L)	262.5 ± 3.6 a	240.2 ± 1.5 b

BE: betanin equivalent; IE: indicaxanthin equivalent.
Different letters indicate significantly different means between the pulp and the extract ($p < 0.05$).

et al. (2009) obtained lower values for red cactus pear pulp (280.9 BE mg/L and 99.6 mg IE mg/L). According to Cruz et al. (2019), betacyanins impart red and betaxanthins yellow colours. This influences the predominance in colour of the fruit's flesh. In fact, red cactus pear as used herein had a higher content of betacyanins, and this can be seen in Table 2, where betacyanins concentrations were approximately twice that of betaxanthins.

Degradation of betalains depends on several factors such as temperature, exposure to light and oxygen, pH, water activity and their inherent chemical structure (Azeredo, 2009; Castellar et al., 2003; Herbach et al., 2006). For instance, in the case of betacyanins, glycosylated structures have been shown to be more stable than the respective aglycons, probably due to the higher oxidation–reduction potential of the first (von Elbe and Attoe, 1985). Overall, the factor that is known to most heavily influence the stability of betalains during food processing is temperature: during heating processes, betalains are degraded by isomerisation, decarboxylation or cleavage by heat. Foreseeably, this results in a gradual reduction of the intensity of red colour (Huang and von Elbe, 1985).

The values reported in Table 2 are higher for cactus pear pulp than for the ethanolic extract, which may suggest either inefficiency or unfavourable conditions during the extraction process. At respect, it has been reported that an ultrasonic-assisted extraction enhances yield (Maran and Priya, 2016; Righi Pessoa da Silva et al., 2018), as well as microwave-assisted extraction of betalains (Singh et al., 2017). The solvent used during maceration extraction also influences yield, although both ethanol and water have been proved to be a good choice of a solvent for extraction of betalains (Castellar et al., 2003).

Microcapsules characterisation

Powder yield of the microencapsulation drying processes is shown in Table 3. Systems with a major content of oca starch (70% and 100%) as drying aids exhibited the highest yield, reaching values of 64.5%. Overall, the yield values obtained from both microencapsulation techniques are higher to those reported for cactus pear encapsulation by spray-drying when using different coating materials, such as cladode mucilage, maltodextrin, inulin, glucose syrup and gelatin (Castro et al., 2014; Obón et al., 2009; Otálora et al., 2015; Sáenz et al., 2009).

Moisture content of the microcapsules was less than 10% for all systems, which might favour the stability of the capsules during storage. Moisture and hygroscopicity of the powder obtained by spray-drying were inversely related: the lower the humidity, the greater the capacity of moisture absorption of the medium (Otálora et al., 2015). This is reflected in the higher hygroscopicity values of the particles whose drying aid material contains the highest percentages of maltodextrin (Table 3). As compared to starches, maltodextrin has a lower molecular weight, which favours hygroscopicity (Cai and Corke, 2000). In contrast, treatments whose unique material had been oca starch exhibited the lowest hygroscopicity values. Similar relationships between humidity and hygroscopicity have been seen in previous studies (Castro et al., 2014; Obón et al., 2009). Hygroscopicity is considered a critical factor in the stability of the microcapsules as well as in the hydrolysis paths followed by some compounds of interest (Vergara et al., 2014), but this will be addressed further below.

The solubility of spray dried powders with microencapsulated bioactives has been shown to be strongly related to the solubility of the encapsulation materials (Castro et al., 2014). Both the maltodextrin and the starch are soluble in heated media, allowing particularly the starch to gelatinize (Ezhilarasi et al., 2013). However, the solubility of the starch in cold media is low as compared to that of maltodextrin, even when the starch had been gelatinized before. This can be seen in the products with 100% oca starch as drying aid, where

Table 3. Yield of encapsulation, moisture, solubility and hygroscopicity of microcapsules powder obtained by spray-drying (SD) and freeze-drying (FD).

Spray (SD) or freeze (FD) drying	Oca starch to maltodextrin ratio	Yield (%)	Moisture ($g \times 100\,g^{-1}$)	Solubility (%)	Hygroscopicity (g water $\times 100\,g^{-1}$)
SD	100:0	42.2 ± 0.5 a	9.3 ± 0.2 e	53.1 ± 0.6 c	15.1 ± 0.8 b
	70:30	64.5 ± 1.6 e	7.0 ± 0.5 b	91.5 ± 0.8f	16.2 ± 1.2 c
	50:50	52.5 ± 1.1 c	8.4 ± 0.3 d	82.3 ± 0.8 e	15.4 ± 0.8 b
FD	100:0	56.0 ± 1.9 d	5.7 ± 0.1 a	24.9 ± 0.6 a	10.4 ± 0.1 a
	70:30	46.6 ± 1.9 b	7.0 ± 0.2 b	43.9 ± 0.5 b	11.6 ± 0.8 a
	50:50	44.3 ± 0.8 a	7.9 ± 0.1 c	56.1 ± 0.6 d	13.1 ± 0.3 a

Different letters show statistically significant differences of means among systems, i.e. within a column ($p < 0.05$).

the poorest solubility values were achieved (ranging from ca. 25% for freeze dried, to 53% for spray dried), as compared to the products with maltodextrin (Table 3). It was also notable that the spray-dried powder is significantly more soluble than that obtained by freeze-drying.

Figure 2 shows the external structure of the obtained powder particles of each of the three different systems obtained by spray drying and freeze-drying, being clear that the morphology of the particles greatly depends on the drying method. With regard to lyophilisation, the particles were amorphous and of irregular surfaces and sizes. These morphological features are characteristic of the lyophilisation process, as has been previously reported (Ballesteros et al., 2017; Kuck and Noreña, 2015; Otálora et al., 2015). Particles obtained by spray drying have regular shapes, with mostly smooth, rounded and indented surfaces, although also being irregular in size. Additionally, irregular spherical shapes, pervasive on spray-dried powder, might favour the retention of the compounds of interest within the coating materials (Ballesteros et al., 2017); while in the case of particles obtained by freeze-drying, irregular surface and bigger size – thus greater contact

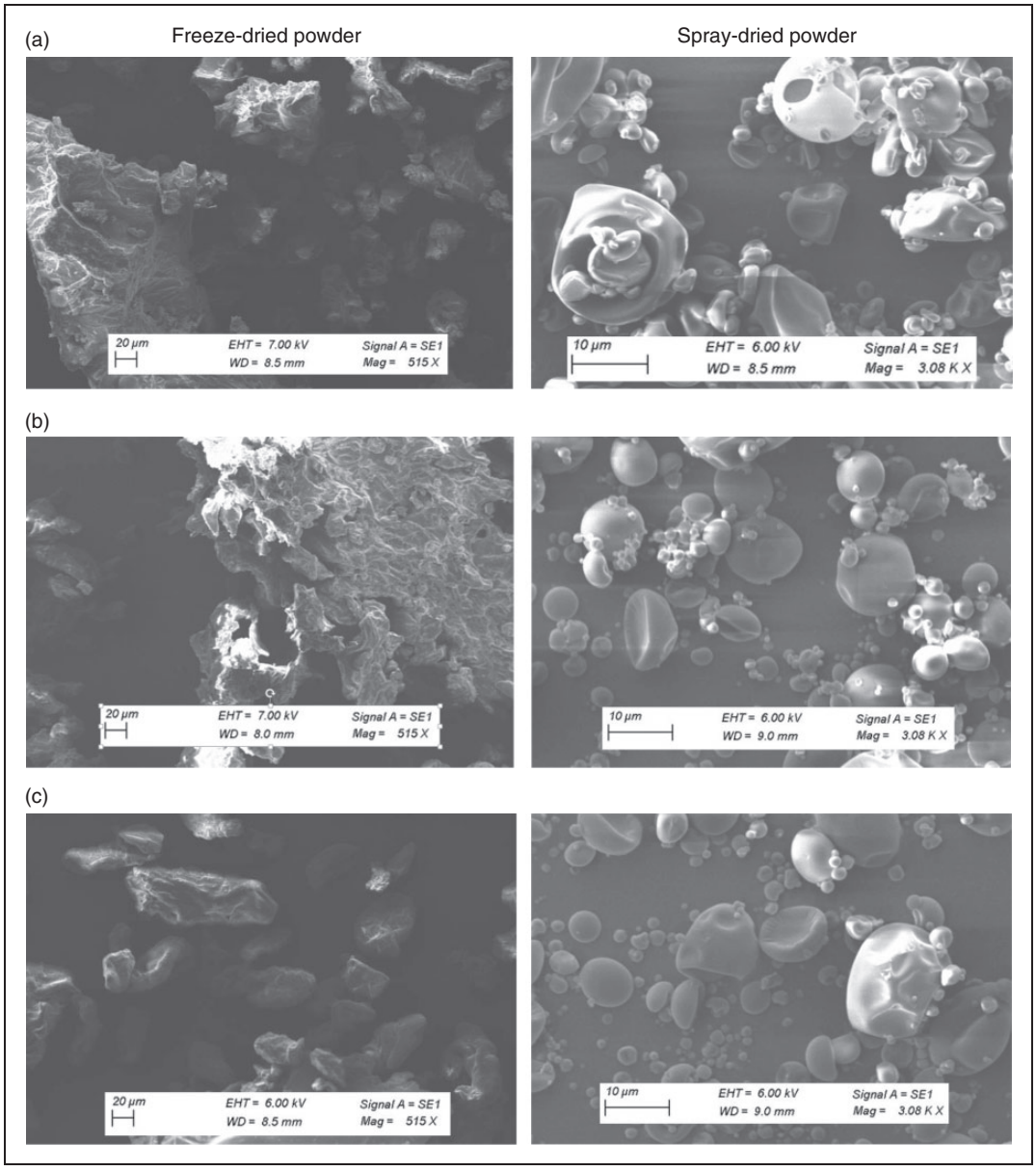

Figure 2. Scanning electron micrographs of cactus pear extract microencapsulated by spray-drying and freeze-drying using different ratios of oca starch:maltodextrin (100:0, 70:30, and 50:50, respectively).

surface – might allow a faster degradation (Ballesteros et al., 2017; Kuck and Noreña, 2015).

According to Walton and Mumford (1999), the use of starch as a coating material, due to its ability to form gels, could prevent the material of interest from evaporating and forming a crust on the surface, reducing the strength of the agglomerate. This can be seen in the SEM micrographs of the spray dried products with 100% and 70% oca starch, i.e. systems with high proportions of oca starch, whose shapes and surfaces are more uniform than those with the lowest starch content as a wall material (spray-dried powder with 50% of oca starch).

The morphology of the particles is often strongly related to some physical characteristics of the powder, as is the case of agglomeration (Otálora et al., 2015). As the particles obtained by spray-drying are smaller, in addition to their spherical pseudo-shape and absence of rough surfaces, this powder flows more easily and, therefore, reduces its tendency to form agglomerates, unlike the freeze-dried particles which, on the contrary, are larger particles, with greater agglomeration potential.

Stability of the microcapsules powder

The stability test shows a clear negative trend over time, which means a decrease in the TPC and antioxidant capacity (Figure 3). The antioxidant capacity (measured through the FRAP and ABTS methods, both yielding similar results) experienced an apparent increase between days 18 and 41. However, after these days, a continuous significant decrease was noted. Robert et al. (2015) reported a similar behaviour during storage at 60 °C of cactus pear microcapsules (with isolated soy protein, maltodextrin and inulin as coating materials). The increase recorded for TPC during storage could possibly be attributed to the hydrolysis of polyphenol glycosides and its subsequent conversion into more reactive aglycones (Robert et al., 2015; Sáenz et al., 2009).

The drying method had a substantial effect on the retention of polyphenols and antioxidant activity (Figure 4). The particles obtained by spray drying were able to retain polyphenols to a greater extent than those obtained by freeze-drying, this is in accordance to Gokhale and Lele (2012), and Kujala et al. (2000), who reported a total phenolic content increase linked to an increase in drying temperatures, due to the degradation of betacyanin forming other phenolic compounds. Although, on the contrary, freeze-dried powder exhibited the best retention values of antioxidant activity (between 62.9% and 64.1% by FRAP method and between 53.5% and 57.6% by ABTS

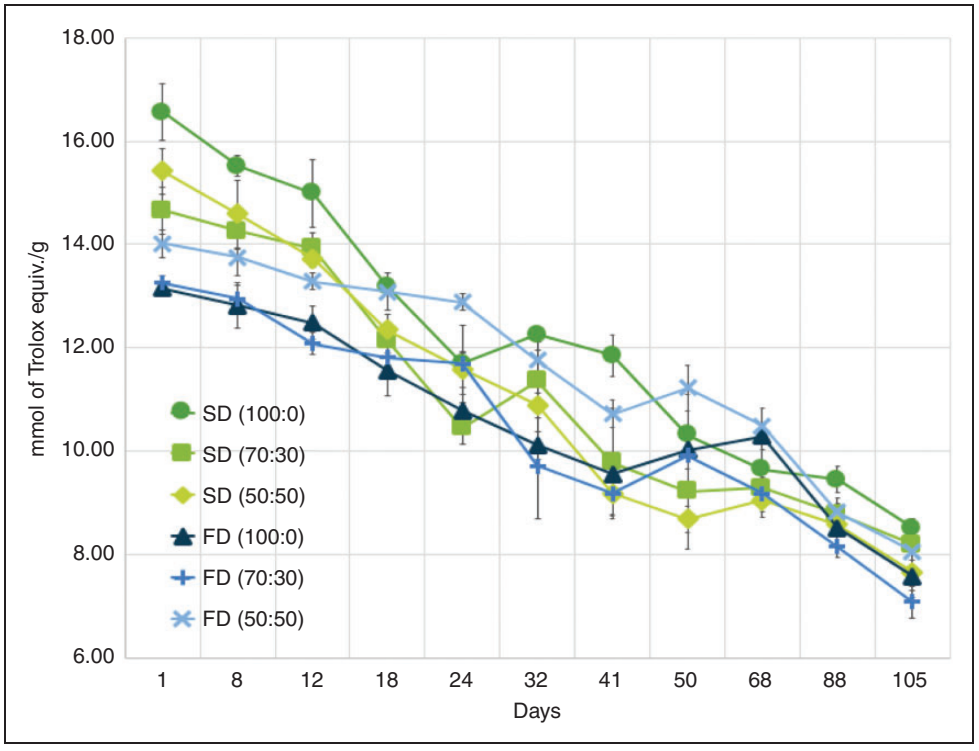

Figure 3. Antioxidant activity measured by the ABTS method during room temperature storage of spray-dried (SD) and freeze-dried (FD) powders microencapsulated with oca starch and maltodextrin at different ratios (100:0, 70:30, and 50:50, respectively).

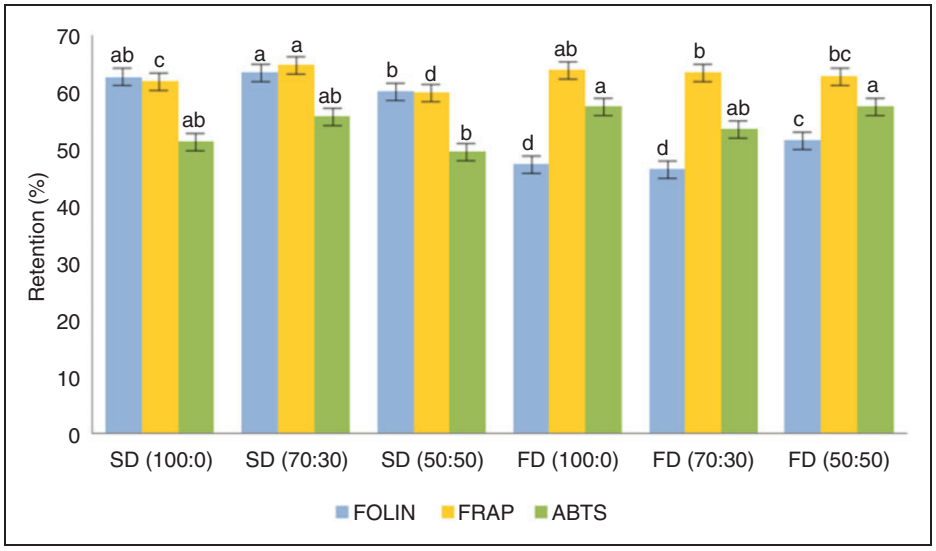

Figure 4. Influence of the oca starch:maltodextrin ratio (100:0, 70:30, 50:50) and drying method (SD: spray drying; FD: freeze-drying) on the relative retention of the total polyphenol content and antioxidant capacity (determined by FRAP and ABTS assays) after storage at room temperature for 105 days. Different letters indicate statistically significant differences ($p < 0.05$) between all values of each analysis across both SD and FD and all the ratios.

Table 4. Retention percentage of total polyphenol content, antioxidant activity and betalains content of spray-dried (SD) and freeze-dried (FD) microcapsules powder after 105 days of storage.

	% Retention after 105 days of storage				
		Antioxidant activity		Betalains	
System	Total polyphenol content	FRAP	ABTS	Betacyanins	Betaxanthins
SD (100:0)	62.7	62.1	51.5	76.5	72.2
SD (70:30)	63.5	64.9	55.9	86.5	75.1
SD (50:50)	60.2	60.1	49.7	83.0	74.7
FD (100:0)	47.4	64.1	57.6	78.8	72.6
FD (70:30)	46.5	63.6	53.5	69.9	79.4
FD (50:50)	51.7	62.9	57.5	81.9	81.9

method), attributable to the drying conditions, with temperature being a determining factor for the preservation of antioxidant activity (Soong and Barlow, 2004).

The ratio of oca starch to maltodextrin as drying aid did not show a significant effect on the protection of the betalains or the preservation of antioxidant activity during the stability test (Table 4). Figure 4 shows the retention percentages obtained for each of the analysis in each of the systems, thus the best system with respect to the conservation of total phenolic compounds and the antioxidant activity measured by FRAP was spray-dried powder with an oca starch-to-maltodextrin ratio of 70:30. This is in accordance with Moharram and Youssef (2014) who stated that the total phenolic content was often highly correlated with the antioxidant activity when this latter is determined by the FRAP method. Regarding the antioxidant activity values obtained by the ABTS method, these do not seem to be related to the values obtained by the FRAP method, which could be attributed to the different antioxidant mechanisms that each method measures (Moharram and Youssef, 2014; Soong and Barlow, 2004) and the expression of the results based on different substances.

The higher content of maltodextrin in the systems led to an increase in hygroscopicity (Table 3), which is considered a critical factor in the stability of the betalains within the powders during storage. According to Herbach et al. (2006), hydrolysis of the

aldimine bond of betalains produce *cyclo*-dopa-D-glucoside (colourless) and betalamic acid (yellow), which leads to a loss of colour intensity. Linear regression of the natural logarithm of the retention percentage of betalains versus storage time shows that the degradation of betacyanins follows a pseudo-first order kinetics (Figure 5). The same order was reported for cactus pear microcapsules with different encapsulating materials, such as capsul, a chemically modified corn starch (Vergara et al., 2014), maltodextrin and inulin (Sáenz et al., 2009), and soybean protein isolated and maltodextrin (Robert et al., 2015). In addition, Cai and Corke (2000) reported an analogous degradation trend for *Amaranthus* betacyanin microcapsules when using corn starch and maltodextrin as coating materials.

The values for betalain retention (Table 4) are higher for those systems with a smaller proportion of oca starch and, overall, obtained by spray-drying (SD3). This latter might seem to be in discordance to betalains being unstable under high temperatures; however, spray-drying uses a high temperature-short time principle and the air inlet temperature is not equal to the commonly drastically lower product temperature due to the evaporative cooling effect, therefore the exposure to high temperatures is minimal (Gharsallaoui et al., 2007). In contrast, sample preparation for freeze-drying, i.e., ''pre-freezing'', often also causes decay of valuable compounds. During freezing, commonly water freezes first, while residual compounds like betalains, but also fruit acids remain in solution. Thereby, acidity levels often increase and evoke drastic pH drops (Shishehgarha et al., 2002), which might have caused damage to betalains.

The betalain content values are similar to those reported by Cai and Corke (2000) and Vergara (2013), but higher than those presented by Robert et al. (2015), who performed an accelerated stability test at 60 °C in the absence of light, and obtained a retention rate of betacyanins of 31% to 53%, when testing soybean protein isolated and maltodextrin as encapsulating agent for cactus, pear pulp. This comparison therefore suggests a greater preservation of betacyanin when using oca starch as part of the formulation of the system.

Table 5 shows the degradation rate constant (k) and half-life *(t1/2)* of betacyanins. The SD (70:30) and FD (50:50) systems have the lowest values of k, at 1.23×10^{-3} days^{-1} and 1.86×10^{-3} days^{-1}, respectively, which indicates a lower degradation rate and a longer half-life. This could be attributable to the higher proportions of oca starch found in these systems. In agreement, the systems with higher proportions of maltodextrin had a greater degradation rate. According to Serris and Biliaderis (2001), low molecular weight polymers might facilitate the entry of oxygen into the microcapsules. On the other hand, it is notorious that spray-dried encapsulated systems have lower degradation rates than those obtained by freeze-drying, with the SD (70:30) system having the longest half-life (563 days). Previous research has shown that the degradation rate and half-time are affected by the storage relative humidity and temperature (Robert et al., 2015;

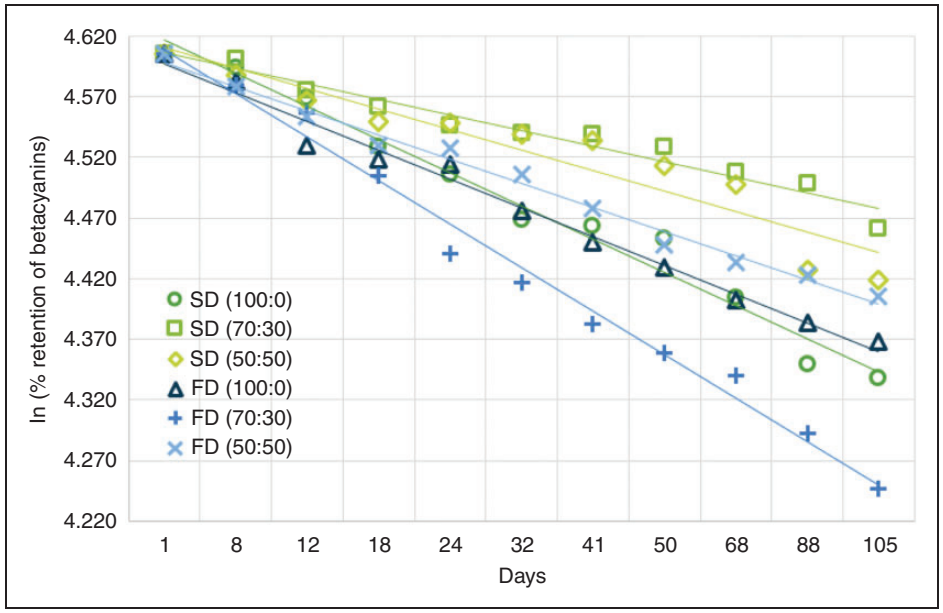

Figure 5. Degradation of betacyanins during room temperature storage of spray-dried (SD) and freeze-dried (FD) powders produced with oca starch and maltodextrin at different ratios (100:0, 70:30 and 50:50, respectively).

Table 5. Betacyanin degradation rate constant of spray-dried (SD) and freeze-dried (FD) powder under ambient storage conditions

System	k (days^{-1})	$t_{1/2}$	R^2
SD (100:0)	2.63×10^{-3}	263	0.95
SD (70:30)	1.23×10^{-3}	563	0.93
SD (50:50)	1.71×10^{-3}	405	0.96
FD (100:0)	2.21×10^{-3}	313	0.90
FD (70:30)	3.35×10^{-3}	206	0.91
FD (50:50)	1.86×10^{-3}	372	0.90

Values were obtained from plots of the slopes of ln (% retention) vs. time (days).

Sáenz et al., 2009; Vergara, 2013). The values obtained for the degradation rate constant (k) in this investigation (Table 5) are close to those reported by Vergara et al. (2014), when evaluating a cactus pear–capsul (modified corn starch) system at a storage temperature of 30 °C; and are smaller than those reported by Otálora et al. (2015) when testing the stability of microcapsules of betanin extract, maltodextrin and cladode mucilage system at 18 °C and 57% HR.

CONCLUSION

Oca starch was found to be suitable for microencapsulating and stabilising betalains and antioxidant compounds extracted from *O. ficus-indica* by spray and freeze-drying. The obtained powders exhibited good physical properties, such as low hygroscopicity, good solubility, high retention values for phenolic content and antioxidant activity, as well as a low degradation rate and great expected half-life regarding betalain content. Thermogravimetric analysis and differential scanning calorimetric are analyses that would complete the characterisation of the microcapsules in future research. The powders' properties were found to depend on the microencapsulation method, while the proportion of the coating materials was found to have a lower effect on the stabilisation of the compounds of interest. The system containing 70% of oca starch and 30% of maltodextrin as coating materials might be a suitable choice for scaling-up the production of oca-based microencapsulated betalain powders from red prickly pear. In future investigations, further applications such as the production of biodegradable films might be targeted, as previously by Daza et al. (2018) proposing starch of *Ullucus tuberosus* Caldas, another Andean starchy vegetable.

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