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Total phenolics, anthocyanin profile and antioxidant activity of maqui, *Aristotelia chilensis* (Mol.) Stuntz, berries extract in freeze-dried polysaccharides microcapsules

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ABSTRACT

The effect of different polysaccharides combinations on the stability of maqui extract was studied in order to design functional foods, dietary supplements or natural colorants. Encapsulation by freeze-drying using maltodextrin, gum Arabic and inulin at 10, 20 and 30% was performed and phenolics, anthocyanin, antioxidant capacity and color difference of the microcapsules were determined. The stability of the bioactives after 60 days storage at 25 °C was also evaluated, along with analysis of a_w, adsorption isotherm, and microstructure to characterize the powders. 10% encapsulating polysaccharide produced best results, with maltodextrin leading to highest process efficiency, while the mixture of maltodextrin/inulin in equal proportion led to highest retention of polyphenols (91.1%) and anthocyanin (98.8%) during storage. The inulin microcapsules retained 94.1% of its antioxidant capacity compared to 25.3% for the freeze-dried maqui powder. Concentration level and polysaccharide matrix of encapsulating agent significantly affect retention of bioactives in the microcapsules.

1. Introduction

Bioactive compounds known for their health-promoting properties are of utmost importance for the food and pharmaceutical industries, where methods and formulations with berries extracts to satisfy growing demands of health-conscious consumers are being developed (Vasconcelos, Garcia, Jimenez, & Ibrahim, 2013; Gironés et al., 2014; Nile & Park, 2014). However, the stability and the effective antioxidant capacity of these phenolic compounds from different sources are affected by many factors, like microstructure of the food matrix, pH value, temperature and presence of enzymes, inhibitors or potentiators of absorption process among others (Bouayed, Hoffmann, & Bohn, 2011). The anthocyanins from plants are hydrophilic phenolic pigments widely distributed in berries and have free radical scavenging activity that contributes in mitigating oxidative stress (Pojer, Mattivi, Johnson, & Stockley, 2013). However, their application as food additives is limited by their readiness to degradation after extraction (Betz & Kulozik, 2011). Encapsulation techniques, a kind of packing technique for solids, liquids or gases within a porous polymeric membrane (Mahdavi, Jafari, Ghorbani, & Assadpoor, 2014; Ray, Raychaudhuri, & Chakraborty, 2016), is an effective method to extend shelf-life of the bioactives and to protect natural extracts, polyphenolics, volatile compounds, enzymes and even probiotic bacteria from irreversible damage caused by oxidative effects of environmental factors, like water, oxygen, light and temperature (Nazzaro, Orlando, Fratianni, & Coppola, 2012; Fang & Bhandari, 2010). Stability studies of barberry's anthocyanin have been conducted and have shown that encapsulation with appropriately selected wall materials such as maltodextrin and gum Arabic can contribute to overcome instability issues that restrict commercial applications (Mahdavi, Jafari, Assadpoor, & Ghorbani, 2016).

The material used to encapsulate the bioactive agent must be of food grade quality, biodegradable and should act as a protective barrier against external damaging factors (Nedovic, Kalusevica, Manojlovic, Levic, & Bugarski, 2011). Such material may be maltodextrin to encapsulate phenolic compounds, protecting the bioactives from oxidation despite having poor emulsifying capacity (Joye & McClements, 2014). Another encapsulating material is gum Arabic, a heteroglucane

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complex with a branched structure of D-galactopyranose chain in 1–3 β-D-glucoside linkage. It has favorable properties for use in encapsulation by drying techniques and it increases the glass transition temperature of the encapsulated product, imparting high solubility, low viscosity and good emulsifying properties in food disperse systems. However, its use is rather limited due to product scarcity and high cost (Ramírez, Giraldo, & Orrego, 2015). Both maltodextrin and gum Arabic have proved to be suitable wall materials to stabilize anthocyanin from saffron petals, using freeze-drying as an effective microencapsulation method (Jafari, Mahdavi-Khazaei, & Hemmati-Kakhki, 2016). A combination of maltodextrin and gum Arabic in a core to wall ratio of 25% has proved to be more efficient than maltodextrin alone or maltodextrin mixed with gelatin to protect natural anthocyanin pigments (Mahdavi, Jafari, Assadpoor, & Dehnad, 2016).

Inulin, a β-(2-1)-oligo-D-fructose of plant origin well-known for its prebiotic properties, is also used as encapsulating polysaccharide. It is partially hydrolyzed in the stomach and small intestine, forming favorable matrix for transporting bioactive molecules to the colon; biopolymer matrix with inulin increases stability of bioactive particles to resist severe medium conditions and extreme temperatures. Inulin has been used to produce through precipitation and spray drying micro spheres enriched with α-tocopherol (Joye & McClements, 2014).

The encapsulation techniques used to preserve bioactive compounds through spray-drying at high inlet temperatures (Mahdavi, Jafari, Assadpoor, & Ghorbani, 2016; Mahdavi et al., 2016) or freeze-drying at low temperature and low absolute pressure (Jafari et al., 2016) are quite frequent. However, the freeze-drying technique is usually preferred for simple encapsulation of water-soluble materials, aromatic substances and pharmaceuticals (Desai & Park, 2005) and is appropriate for an easy-to-scale-up process from both a technical–economic and environmental point of view (Jafari et al., 2016). The bioactive agent is first homogenized in a solution with the encapsulating agent before freeze-drying to preserve most of the original characteristics of the bioactives, whereby the efficiency of the protective action or the controlled diffusion of the bioactives would depend on composition and microstructure of the encapsulating medium (Ceballos, Giraldo, & Orrego, 2012).

The berries of maqui (*Aristotelia chilensis* [Mol.] Stuntz), native to central and southern Chile (Gironés-Vilaplana, Mena, García, & Moreno, 2012), are known to have an outstanding content of bioactives, associated with high content of anthocyanins (Escribano-Bailón, Alcalde-Eon, Muñoz, Rivas-Gonzalo, & Santos-Buelga, 2006), flavonols, flavanols and phenolic acids (Céspedes et al., 2010) and to possess anti-inflammatory (Schreckinger, Lotton, Lila, & de Mejia, 2010), anti-adipogenic (Schreckinger et al., 2010), anti-atherogenic (Miranda-Rottmann et al., 2002) and cardioprotective activities (Céspedes, El-Hafidi, Pavon, & Alarcon, 2008). The bioactives are unfortunately prone to degradation once the berries are plucked. The aims of this study were to find out if retention and stabilization of bioactives increase with increasing amount of encapsulating agent, and how the process is affected by the polysaccharide matrix consisting of different proportions of maltodextrin, gum Arabic and inulin through freeze-drying compared to freeze-dried maqui berries powder, where the bioactives are protected by the natural supramolecular complexes, such as plasma membrane or cell wall tissues. The retention of the bioactives in the polysaccharide matrices has been evaluated through response surface methodology and analysis of phenolics and anthocyanin, antioxidant activity and color difference, expressing results as a linear multivariate function of the polysaccharides fraction. The morphology of the powders produced using different combinations of the polysaccharides was examined using optical microscopy and scanning electron microscopy (SEM). The adsorption isotherms of the maqui powders at 25 °C were also determined and modelled.

2. Materials and methods

2.1. Raw materials and freeze-drying process

Maqui berries were purchased directly from merchants, who recollected the berries from neighboring forests of Valdivia, Chile (latitude ≈39°S). Berries extracts for the freeze-drying process were prepared in triplicate for each assay, weighing each time in a Falcon tube 5 g berries that were crushed with a glass stab, before adding 20 mL distilled water acidified with 0.1% 1.5 M HCl. The berries were macerated refrigerated in the dark for 48 h. The extracts were then obtained after centrifugation at 12000 rpm for 20 min.

The freeze-drying process of the maqui extract mixed with the tested polysaccharides was performed following a procedure described by Rocha-Parra, Lanari, Zamora, and Chirife (2016) with some modifications. The mixture of polysaccharides (maltodextrin D15, inulin and gum Arabic) was first added to the aqueous extract of maqui berries in proportions of 10, 20 or 30% m/v to obtain a suspension that was then poured on an aluminium tray to a thickness of 1 cm and frozen at –20 °C for 24 h, before the freeze-drying process conducted in a freeze-dryer (VirTis, Benchtop K, USA) at –80 °C and 25 mmHg for 72 ± 1 h. The dried samples were then ground using mortar and pestle and stored in polyethylene bags, protected from sunlight, until further analysis.

2.2. Experimental design

A simplex centroid mixture design was applied to obtain the most appropriate combinations of the three polysaccharides (PS), namely maltodextrin D15 (M), inulin (I) and gum Arabic (G). Seven combinations for the formulation with the three different mixtures of polysaccharides were obtained (Table 1). In samples M, I and G, only pure polysaccharides were used. In samples MI, MG and GI, both polysaccharides were present in equal proportions, while in samples MGI, all three polysaccharides were in equal proportions. The combination of PS was added to the maqui extract in 3 different proportions (10, 20 or 30% m/v) as described in the preceded subsection.

2.3. Moisture content, water activity and adsorption isotherm

Moisture content was determined according to AOAC official methods (AOAC, 1990), using a vacuum oven (OV-11, JEIO Tech, Seoul, South Korea) at 70 °C for 72 h. Water activity was measured at 25 °C by means of a water activity instrument (Novasina, TH-500, Pfäffikon, Switzerland). All analyses were performed in triplicate. An adsorption isotherm was also determined at 25 °C, using a known mass of sample, prepared in triplicate, allowing it to equilibrate in an atmosphere of known water activity (a_w), inside a hermetically closed flask that contained a glass dish with a saturated salt solution. The salts used were: LiCl, CH_3COOK, $MgCl_2$, $Mg(NO_3)_2$, NaCl, $(NH_4)_2SO_4$, KCl and K_2SO_4. The sample mass (± 0.0001 g) was determined at the start of the assay and after 30 days when constant mass at equilibrium was achieved. Thymol was placed separately in a test tube inside the flasks with relative humidity above 75% to prevent mold growth.

$$x_{we} = \frac{x_m C K a_w}{(1 - K a_w)(1 + [C - 1] K a_w)} \quad (1)$$

The adsorption isotherm was modelled using the mathematical expression derived by Guggenheim, Anderson and de Boer (GAB) as given in Eq. (1), which is widely used for description of equilibrium moisture isotherms of biological materials and considers parameters based on physicochemical phenomena, such as monolayer moisture content x_m, first layer of heat of sorption (constant C) and multi-layer molecules (constant K) with respect to the bulk liquid. The coefficient of determination r^2 (Eq. (1a)) and percentage error Er% (Eq. (1b)) were the selected criteria to evaluate the fit quality of mathematical model.

Table 1
Water activity, adsorption isotherm and encapsulation efficiency.

Encapsulating polysaccharide	10%	20%	30%		
	Water activity				
M	0.223 ± 0.001^{b}	0.225 ± 0.002^{a}	0.167 ± 0.002^{c}		
I	0.185 ± 0.002^{d}	0.144 ± 0.002^{e}	0.010 ± 0.002^{d}		
G	0.141 ± 0.0010^{e}	0.149 ± 0.001^{d}	0.175 ± 0.002^{bc}		
MI	0.277 ± 0.001^{a}	0.206 ± 0.002^{b}	0.184 ± 0.002^{a}		
MG	0.227 ± 0.002^{b}	0.202 ± 0.001^{b}	0.176 ± 0.002^{b}		
GI	0.140 ± 0.001^{e}	0.147 ± 0.001^{d}	0.182 ± 0.002^{a}		
MGI	0.196 ± 0.001^{c}	0.183 ± 0.001^{c}	0.170 ± 0.002^{bc}		
Constants of GAB model for adsorption isotherm (Eq. (1)) with 10% EP					
	x_m	C	K	R^2	Er%
M	3.425	–23.113	0.810	0.981	6.370
I	4.636	−25.260	0.762	0.982	6.344
G	3.558	−40.780	0.801	0.976	5.941
MI	3.762	−598.745	0.765	0.961	8.152
MG	4.695	−45.752	0.763	0.985	4.372
GI	4.202	−40.639	0.781	0.978	5.642
MGI	4.293	−46.698	0.756	0.978	4.566
Encapsulating polysaccharide	10%	20%	30%		
	Encapsulation efficiency				
M	65.7 ± 1.53^{a}	29.5 ± 0.61^{b}	23.7 ± 0.36^{b}		
I	59.4 ± 0.99^{b}	32.3 ± 0.59^{a}	5.7 ± 0.19^{d}		
G	24.2 ± 0.59^{c}	5.9 ± 0.17^{c}	20.1 ± 0.89^{c}		
MI	67.2 ± 1.98^{a}	33.9 ± 1.25^{a}	5.9 ± 0.26^{d}		
MG	23.7 ± 0.53^{c}	6.2 ± 0.26^{c}	18.2 ± 0.32^{c}		
GI	27.2 ± 0.54^{c}	7.5 ± 0.18^{c}	30.3 ± 0.69^{a}		
MGI	24.3 ± 0.77^{c}	6.7 ± 0.23^{c}	22.3 ± 0.71^{b}		

Different letters in the same column indicate significant difference ($p < 0.05$).

$$r^2 = \frac{\sum_{i=1}^{n} (\hat{y}_i - \bar{y})^2}{\sum_{i=1}^{n} (y_i - \bar{y})^2} \quad (1a)$$

$$Er\% = \frac{100}{n} \sum_{i=1}^{n} \left| \frac{y_i - \hat{y}_i}{y_i} \right| \quad (1b)$$

Being $\hat{y}_i$: predicted value; y_i: experimental value; $\bar{y}$: mean value; i: terms (0, 1, 2…n); n: number of data.

2.4. Encapsulation efficiency of freeze-drying process

Encapsulation efficiency E of the freeze-drying process to obtain the maqui extract powder was determined using Eq. (2) based on dry matter content (Çam, Içyer, & Erdogan, 2014). In the equation $C_{A,E}$ and $C_{A,T}$ are concentrations of total monomeric anthocyanin determined in the powder samples after removal of surface anthocyanins and in the extract respectively (Kaushik & Roos, 2006).

$$E = \frac{c_{A,E}}{c_{A,T}} 100 \quad (2)$$

2.5. Microstructural analysis and color measurement of maqui extract powders

General characteristics of the microcapsules were evaluated by optical microscopy using a Zeiss Stemi DV 4 microscope. A sample portion was placed on a slide, which was sealed with a coverslip and transparent tape. The micrographs were acquired at 32X with a Canon EO5 Rebel T3 digital camera. Then, the images were analysed and processed with Axia Vision Rel 4.8 software. Also, a Scanning Electron Microscope (Auriga Dual Beam FIB SEM, Zeiss, Germany) was used to examine and evaluate the microstructure of the freeze-dried powder. The samples were mounted on the specimen holder and sputter-coated with gold (2 min, 2 mbar), and images were observed at 15 kV under vacuum.

Color of freeze-dried powdered maqui berries as control and the freeze-dried maqui extract powders was evaluated by the CIE Lab System using a spectrophotometer (PCE-CSM 8, PCE Instruments, UK). Three measurements were performed for each sample after instrument's calibration using a white standard reflectance plate. Color difference ΔE was determined according to Eq. (3).

$$\Delta E = \sqrt{\Delta L^2 + \Delta a^2 + \Delta b^2} \quad (3)$$

2.6. Determination of total phenolics, total anthocyanin and anthocyanin profiles

Total phenolics content (TPC) was determined using Folin-Ciocalteu (FC) reagent and absorbance was measured at 765 nm (Ramírez et al., 2015). TPC was obtained using gallic acid standard for calibration. Results were expressed as gallic acid equivalent per 100 g sample (mg GAE/100 g). All analyses were performed in triplicate and absorbance was measured using a spectrophotometer (Spectronic® 20 Genesys®, IL, USA).

Total anthocyanin content (TAC) was determined by pH differential method (Lee, Durst, & Wrolstad, 2005), after extraction of the dried maqui berries (500.0 ± 0.1 mg) following the procedure described by Fredes et al. (2014). The absorbance was measured at 520 and 700 nm and results were expressed in mg cyanidin-3-glucoside/100 g sample (mg cya-3-glu/100 g).

Anthocyanin profile in the microcapsules was determined using HPLC-DAD method performed on a Waters™ Alliance 2695 system, with a Waters™ 2996 photodiode array detector, a Thermo™ Scientific C18 BDS (100 mm × 4.6 mm; 2.4 mm) column, at an eluent flow rate of 0.7 mL/min, column temperature of 30 °C, injection volume of 20 µL and a gradient elution method with 0,3% trifluoroacetic acid (A) and acetonitrile (B): 0 – 4th min 95% A; 4.5th min 90% A; 27th min 85% A; 47th min 45% A; 48th min 10% A; 50th min 10% A; 51st min 95% A and 60th min 95% A.

2.7. Determination of antioxidant capacity by DPPH assays

The DPPH radical scavenging activity of maqui extracts was measured using the method of Brand-Williams, Cuvelier, and Berset (1995). The results were expressed as mg Trolox (6-hydroxy-2,5,7,8-tetramethylchroman-2-carboxylic acid) equivalent/100 g sample (mg TE/

100 g).

2.8. Statistical analysis

The parameters were obtained in triplicate and the software Statgraphics® Centurion XV.I (Statistical Graphics Corp., Herndon, VA, USA) was used for data analysis (ANOVA). Differences among the media were analyzed using the least significant difference (LSD) test at significance level of $\alpha = 0.05$ or $\alpha = 0.01$ at confidence interval of 95% ($P < 0.05$) or 99% ($P < 0.01$). A multiple range test (MRT) was also performed to demonstrate existence of homogeneous groups within each of the parameters.

3. Results and discussion

3.1. Efficiency of freeze-drying process

An average of 92.6 ± 5.2% of maqui extract powder was recovered as dried product with respect to the initial weight of the polysaccharides' mixture used. The efficiency of the freeze-drying process, as determined through evaluation of the anthocyanin profile of the maqui extract and the produced powders, was highest for the maltodextrin-inulin powder (67.2 ± 2.0%) but did not show highly significant difference ($P < 0.01$) compared to the maltodextrin powder (65.7 ± 1.5%). A lower efficiency was obtained for the inulin powder (59.4 ± 1.0%). In general, efficiency of the freeze-drying process for the powders containing gum Arabic did not show highly significant differences ($P < 0.01$), fluctuating between 23.7 ± 0.5% for the maltodextrin-gum Arabic powder and 27.2 ± 0.5% for the gum Arabic-inulin powder. Fredes, Osorio, Parada, and Robert (2018) found that efficiency values decreased at higher proportion of added polysaccharides during spray-draying of maqui juice. Fredes et al. (2018) also reported that encapsulation efficiency depended on the wall material used and obtained values between 44.0 and 51.2% for encapsulation of maqui juice in alginate, and values between 65.6 and 78.6% for encapsulation with inulin as wall material. It was assumed that in the case of inulin encapsulation efficiency was directly related to the number of hydroxyl groups, while in the case of alginate the hydroxyl groups showed no effect on encapsulation efficiency that was rather influenced by the type of polyphenol and its structural features. On the other hand, Mahdavi et al. (2016) showed that the type of wall material used is a relevant factor that affects encapsulation efficiency. It was also shown that for barberry's anthocyanin a ratio of core to wall materials of 25% led to highest efficiency in the combined state of wall materials; a mixture of maltodextrin with gum Arabic proved to be more efficient than pure maltodextrin for the encapsulation process, since a single encapsulating wall material presumably do not possess all required characteristics.

3.2. Characteristics of maqui extract powders

The anthocyanin identified in maqui extract powders were also found in the freeze-dried powders but at different levels. Delphinidin-3-sambubioside-5-glucoside predominated as anthocyanin in the maqui extract, being 80.4% of the anthocyanin delphinidin glucosides, while the remainder was cyanidin glucosides. Similar anthocyanin profiles have been reported for maqui berries with Delphinidin-3-sambubioside-5-glucoside being the leading anthocyanin (Céspedes et al., 2010; Gironés-Vilaplana et al., 2012; Fredes et al., 2014, 2018). Maltodextrin and inulin powders were found to have similar anthocyanin profile to the original maqui extract (Fig. 1). However, maltodextrin powders contained only around 66% of the anthocyanin compared to 60% in the inulin powders. After 60 days storage at 25 °C retention of anthocyanin in maltodextrin and inulin powders was reduced to 56 and 50% respectively. In gum Arabic powders a different profile of anthocyanin was obtained and retention of the anthocyanin after freeze-drying was only around 24% and this value dropped to 20% after 60 days storage at 25 °C. This low level of anthocyanin was also found in all powders that contained gum Arabic. As reported by Kaushik and Roos (2006), high degree of polymerization of the encapsulating agent may be the cause for the low retention of bioactive components, which may be the case for the highly branched molecule of gum Arabic.

The maqui extracts powders obtained using different proportions of polysaccharides had water activities (Table 1) ranging from 0.010 ± 0.002 with inulin as unique polysaccharide to 0.277 ± 0.001 with a mixture of maltodextrin and inulin, which corresponds to a stable dried material. Similar values of water activities between 0.02 ± 0.00 (using gum Arabic) and 0.289 ± 0.030 (using maltodextrin) have been reported by Mahdavee Khazaei, Jafari, Ghorbani, and Hemmati Kakhki (2014) for microencapsulated saffron petals' anthocyanin. As can be seen in Table 1, water activity showed a tendency to decrease as the proportion of encapsulating polysaccharide increased, except for the powders that contained gum Arabic (G) or a mixture of the same with inulin (GI). Similar decreasing behaviour of a_w from 0.410 ± 0.001 to 0.160 ± 0.001 was observed with increasing proportion of maltodextrin from 20 to 30% for freeze-dried sumac extract powders (Caliskan & Dirim, 2016). Adsorption isotherms at 25 °C (Fig. 2) of the powders with 10% polysaccharide also revealed higher equilibrium moisture contents for gum Arabic powders. At an a_w value of 0.225 the highest and lowest equilibrium moisture contents of 7.27 ± 0.21% and 5.25 ± 0.29% were observed for gum Arabic and inulin powders respectively. The experimental data for the adsorption isotherms showed a good fit to the GAB model (Eq. (1)) with high coefficients of determination ($R^2 > 0.96$) and low percentage error (Er % < 8.2%); Table 1 also shows the constants of the respective GAB equations for the maqui extract powders produced using 10% PS.

3.3. Total phenolic, total anthocyanin and antioxidant activity

The total phenolics content (TPC) found in the maqui extract powders immediately after freeze-drying process and after storage of the powders at 25 °C for 60 days can be seen in Fig. 3, as a function of the weight fractions of PS in the aqueous maqui extract at 3 different levels (10, 20 and 30%). TPC in the capsules showed that a proportion of 10% PS would be more favorable for encapsulating the bioactive phenolics, since increasing proportion of EP led to a consistent decrease of TPC, showing the importance of this parameter. The highest experimental value of 3421.0 ± 98.4 mg GAE/100 g sample for TPC was determined in the powders with a proportion of 10% PS consisting of a mixture of equal amount of maltodextrin and inulin (*MI*). TPC in the different microcapsules can be calculated using Eq. (4) that stands for a general model used to calculate the desired parameter as a function of the weight fraction of the encapsulating PS.

$$X = a(M) + b(I) + c(G) + d(MI) + e(MG) + f(GI) + g(MGI) \quad (4)$$

In the equation X is TPC and M, G and I are the respective weight fraction (values between 0 and 1) of maltodextrin, gum Arabic and inulin, with the letters a to g denoting constant coefficients given in Table 2 for TPC value on day 1 and after a 60-day storage at 25 °C. According to the model obtained (Fig. 3) the highest TPC will be achieved with weight fractions of 0.448 and 0.552 for M and I respectively. After 60 days storage at 25 °C a TPC of 3092.0 ± 66.5 mg GAE/100 g sample was determined, which corresponded to 91% retention. Compared to the freeze-dried maqui berries a retention of 86% was achieved under similar storage conditions; however, TPC changed from a higher value of 4226.4 ± 45.9 to 3653.1 ± 34.6 mg GAE/100 g sample. As reported by Çam et al. (2014), total phenolics from pomegranate peel encapsulated in maltodextrin showed no significant loss during storage at 25 °C; a change from 129.1 to 119.3 mg GAE/g sample was observed within 60 days, while the non-encapsulated sample showed only a significant loss during the first 15 days of storage from 369.4 to 279.3 mg GAE /g sample and a TPC of 248.4 mg GAE /g

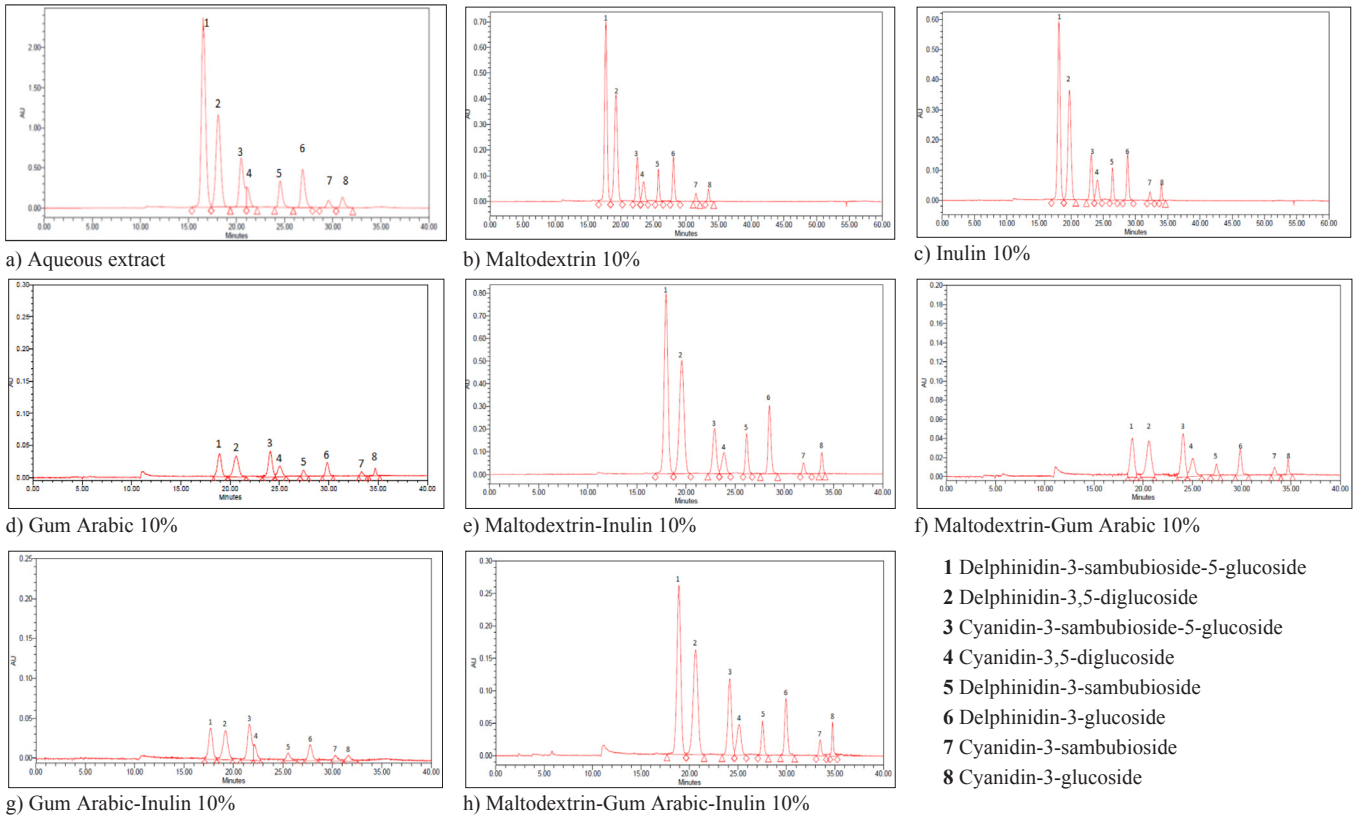

Fig. 1. Anthocyanin profile in maqui extract powders with 10% PS determined by HPLC-DAD.

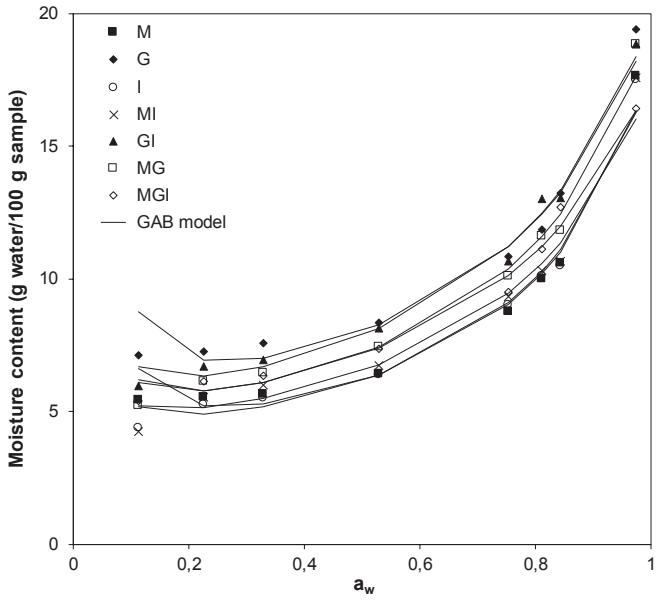

Fig. 2. Isotherm of maqui microcapsules powder at 25 °C.

sample after 60 days storage. It has also been reported by Sanchez et al.[34] that polyphenols from cherry encapsulated through freeze-drying in maltodextrin and gum Arabic can be retained at a level of 90% for 33 days at 38 °C.

The retention of total anthocyanin content (TAC) in the maqui extract powders was most favorable at 10% PS, either in maltodextrin alone or in a mixture of maltodextrin with inulin. A higher proportion of PS led to a lower concentration of anthocyanin in the freeze-dried powder (Table 3), which can be compared to the results reported by Fredes et al. (2018), however for spray-drying, where inulin and sodium alginate were used as carriers in proportion over 30% m/v at a ratio of carrier to juice of 6.6:1 and 3.4:1 respectively. This led to anthocyanin content between 280 and 590 mg cya-3-glu/100 g in the resulting maqui juice powder. In this work, mathematical models (Eq. (4) and Table 2) were developed from the experimental data to calculate the anthocyanin content as a function of the weight fraction of PS. A maximum value of 1185.73 mg cya-3-glu/100 g can be calculated for the powder with 100% maltodextrin as PS on day 1 and a maximum value of 1112.2 mg cya-3-glu/100 g after a 60-day storage at 25 °C for the powder produced with weight fractions of maltodextrin and inulin of 0.610 and 0.390 respectively. The retention of anthocyanin in maltodextrin powders after 60 days storage at 25 °C was around 82%, while in the mixture of maltodextrin and inulin, retention was around 99%. In the freeze-dried maqui berries that contained its own polysaccharides TAC changed from 2188.1 ± 50.6 to 1714.7 ± 55.2 mg cya-3-glu/100 g, achieving a retention of only 78.4%, but with an initially higher content of anthocyanin. Sanchez, Baeza, and Chirife (2015) have reported that 90% of the anthocyanin from cherry juice could be retained in the samples freeze-dried with maltodextrin and gum Arabic. Anthocyanin extracted from saffron petals were also found to be more stable when the extract was freeze-dried with than without maltodextrin and gum Arabic (Mahdavee Khazaei et al., 2014). It is presumed that moisture reduction during freeze-drying would reduce molecular mobility of water in the high molecular weight encapsulating polysaccharides, leading to confinement of the dispersed bioactive molecules that are then better protected from external degradation factors such as air, light or temperature (Fang & Bhandari, 2010; Jafari, Assadpoor, He, & Bhandari, 2008; Saavedra-Leos et al., 2014).

The antioxidant activity of the maqui extract powders was also determined and modelled as a function of weight fraction of PS. Antioxidant activity on day 1 and day 60 can be calculated using Eq. (4) together with the constant coefficients a to g given in Table 2. The highest antioxidant activity of 2644.4 ± 67.7 mg TE/100 g was

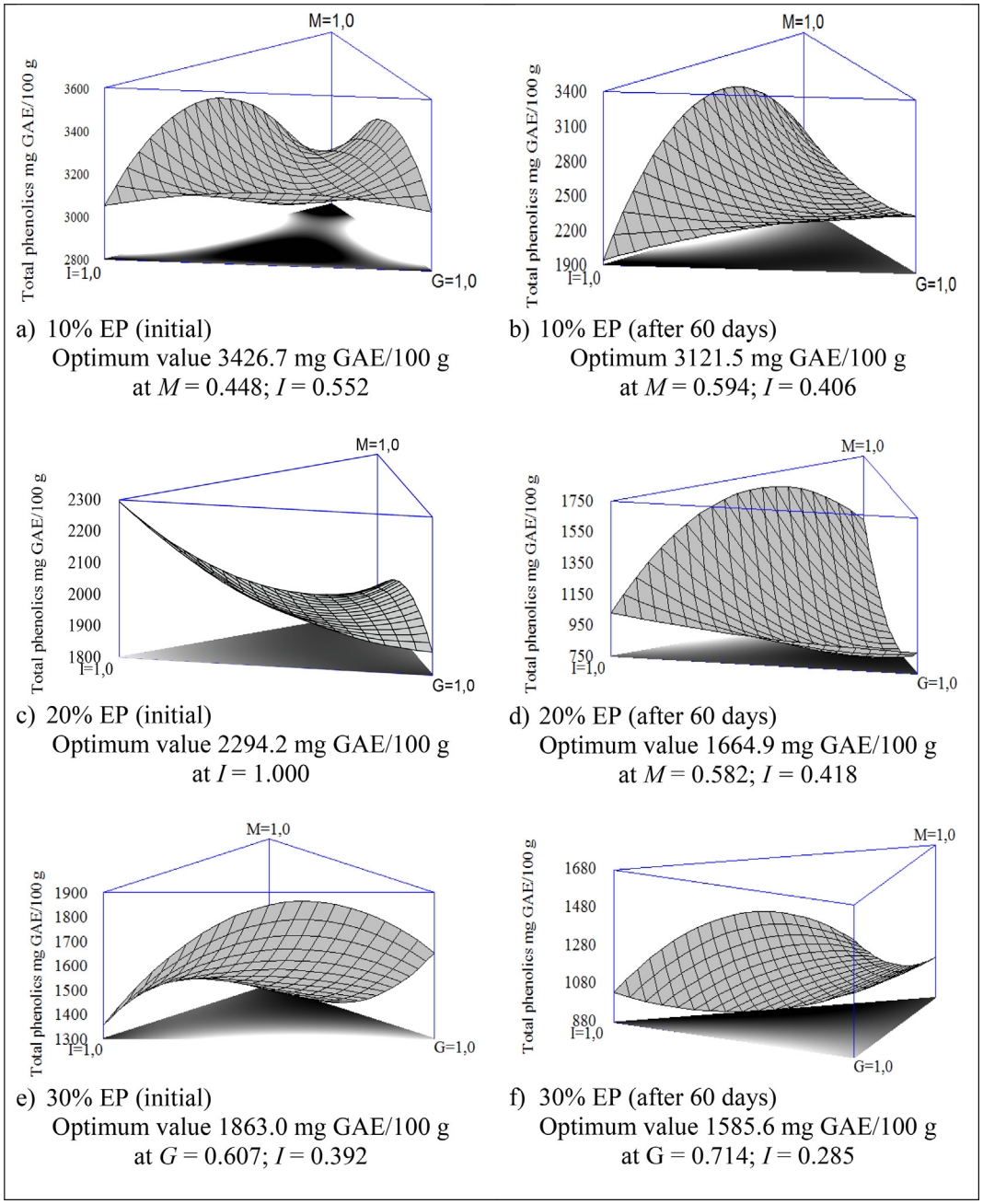

Fig. 3. TPC in maqui powders after freeze-drying process and after 60 days storage at 25 °C.

determined in the powder produced using pure inulin at 10%. After 60 days storage at 25 °C the antioxidant activity was reduced to 2489.5 ± 67.2 mg TE/100 g, which represented only a loss of 6%. In comparison, the antioxidant activity determined in the freeze-dried berries changed from 4525.5 ± 56.9 to 1145.9 ± 20.7 mg TE/100 g, meaning a loss of almost 75% with respect to initial value. As can be seen in Table 3 the powder obtained through freeze-drying of the maqui extract with 10% of a mixture of maltodextrin and inulin could also

Table 2
Coefficients for mathematical models used to determine *TPC*, *TAC*, *AOX* and *ΔE*.

X	*a*	*b*	*c*	*d*	*e*	*f*	*g*	R^2
TPC_{01}	2852.7	3049.5	3074.2	1881.7	1538.4	296.9	−9670.4	0.897
TPC_{60}	2571.4	1944.1	2395.8	3337.0	−564.5	395.7	−8076.9	0.973
TAC_{01}	1185.7	1021.4	481.3	60.4	−1210.8	−1100.1	−5171.0	0.998
TAC_{60}	968.6	761.0	620.2	944.1	−763.7	−305.1	−5354.4	0.993
$AOX1_0$	1864.2	2644.5	1171.6	619.6	−1549.2	−3275.8	−8248.2	0.998
AOX_{60}	1997.8	2489.5	2145.3	−143.6	−811.1	−2000.7	−3867.6	0.989
ΔE_{01}	16.1	23.6	19.6	−0.83	7.1	−4.6	938	0.824
ΔE_{60}	20.8	25.3	18.4	−19.3	−8.7	−6.0	123.1	0.887

Table 3
Total anthocyanin content, antioxidant activity and color difference in maqui powders.

	Initial			After 60 days storage at 25 °C		
Encapsulating polysaccharide	10%	20%	30%	10%	20%	30%
	Total anthocyanin (mg Cya-3-glu/100 g sample). In powdered freeze-dried berries, initially: 2188.1 ± 50.6 and after 60 days: 1714.7 ± 55.2					
M	1185.7 ± 4.2^{a}	662.4 ± 18.8^{c}	419.4 ± 18.3^{bc}	968.6 ± 33.4^{b}	570.1 ± 5.6^{c}	355.9 ± 10.7^{c}
I	1021.4 ± 8.4^{c}	835.3 ± 11.2^{a}	446.2 ± 20.1^{b}	761.0 ± 15.4^{c}	564.0 ± 22.4^{d}	124.0 ± 5.9^{d}
G	481.3 ± 17.7^{de}	309.3 ± 9.3^{d}	466.0 ± 9.4^{b}	620.2 ± 15.7^{d}	133.6 ± 5.2^{b}	472.3 ± 10.4^{b}
MI	1118.7 ± 21.0^{b}	773.5 ± 22.3^{b}	237.7 ± 7.3^{d}	1100.8 ± 6.0^{a}	580.7 ± 4.3^{d}	110.6 ± 4.7^{d}
MG	530.8 ± 9.8^{d}	312.6 ± 14.9^{d}	367.4 ± 1.1^{c}	603.5 ± 11.1^{d}	156.4 ± 5.0^{b}	449.0 ± 16.0^{b}
GI	476.4 ± 18.1^{de}	341.3 ± 4.0^{d}	565.1 ± 24.9^{a}	614.3 ± 20.5^{d}	161.2 ± 3.4^{b}	616.4 ± 11.3^{a}
MGI	454.6 ± 18.1^{e}	356.0 ± 11.6^{d}	418.1 ± 6.4^{bc}	571.1 ± 17.0^{d}	157.2 ± 5.1^{a}	656.5 ± 30.0^{a}
	Antioxidant capacity (mg TE/100 g sample). In powdered freeze-dried berries, initially: 4525.5 ± 56.9 and after 60 days: 1145.9 ± 20.7					
M	1864.2 ± 22.6^{c}	1563.3 ± 45.2^{c}	1136.5 ± 40.5 ^{cd}	1997.8 ± 7.7^{c}	1579.9 ± 7.4^{b}	1196.6 ± 14.9^{c}
I	2644.4 ± 67.7^{a}	2405.3 ± 45.7^{a}	1827.4 ± 75.1^{a}	2489.5 ± 67.2^{a}	1521.7 ± 3.9^{b}	682.5 ± 14.5^{d}
G	1171.6 ± 12.8^{d}	737.8 ± 35.6^{e}	1013.5 ± 22.1^{de}	2145.3 ± 26.2^{b}	1017.2 ± 1.8^{c}	1391.1 ± 18.9^{b}
MI	2409.2 ± 36.8^{b}	2202.4 ± 11.1^{b}	1502.1 ± 24.5^{b}	2207.8 ± 14.6^{b}	2173.4 ± 84.0^{a}	1510.0 ± 13.9^{a}
MG	1130.6 ± 12.8^{d}	940.9 ± 11.2^{d}	819.3 ± 39.8^{f}	1868.8 ± 25.7^{d}	1251.7 ± 4.0^{c}	1383.9 ± 20.9^{b}
GI	1089.1 ± 42.2^{d}	834.5 ± 9.4^{de}	1171.9 ± 7.3^{c}	1817.2 ± 11.2^{de}	1210.5 ± 21.8^{d}	1472.0 ± 11.3^{a}
MGI	1120.7 ± 3.7^{d}	846.7 ± 37.8^{de}	970.5 ± 31.4^{e}	1739.3 ± 24.0^{e}	1012.4 ± 11.9^{d}	1157.9 ± 24.3^{c}
	Color difference					
M	16.1 ± 0.4^{b}	20.4 ± 0.2^{de}	23.2 ± 0.5^{c}	20.8 ± 0.9^{bc}	19.5 ± 0.4^{f}	24.4 ± 0.4^{c}
I	23.6 ± 0.8^{a}	29.0 ± 0.2^{a}	36.6 ± 0.6^{a}	25.3 ± 0.4^{a}	32.3 ± 0.7^{a}	41.7 ± 0.5^{a}
G	19.6 ± 0.4^{ab}	20.1 ± 0.9^{e}	18.9 ± 0.2^{e}	17.3 ± 0.5^{bc}	23.5 ± 0.5 ^{cd}	19.8 ± 0.5^{d}
MI	19.6 ± 0.3^{ab}	21.7 ± 0.6 ^{cd}	28.5 ± 0.2^{b}	18.2 ± 0.5^{c}	22.0 ± 0.4^{de}	32.0 ± 0.1^{b}
MG	19.6 ± 0.2^{ab}	17.7 ± 0.2^{f}	20.7 ± 0.3^{d}	17.4 ± 0.8^{c}	20.7 ± 0.3^{ef}	21.9 ± 0.4 ^{cd}
GI	20.5 ± 0.7^{ab}	22.3 ± 0.1^{bc}	18.3 ± 0.2^{e}	20.4 ± 0.2^{bc}	25.5 ± 0.9^{bc}	20.4 ± 0.9^{d}
MGI	23.4 ± 0.1^{a}	23.5 ± 0.5^{b}	19.3 ± 0.6^{e}	22.3 ± 0.2^{ab}	27.4 ± 0.8^{b}	20.0 ± 0.7d

retained around 91% of the initial value of antioxidant activity. A retention of antioxidant activity over 88% in maqui juice freeze-dried with maltodextrin has also been reported (Brauch, Kroner, Schweiggert, & Carle, 2015). Similarly, it has been reported that extract of black currant spray-dried with inulin can maintain almost unchanged the antioxidant activity for 12 months during storage at 8 °C, while only slight changes would occur at 25 °C (Bakowska-Barczak & Kolodziejczyk, 2011). On the other hand, antioxidant activity of encapsulated grape polyphenols in maltodextrin and gum Arabic was found to decrease during spray-drying process due to the involved high temperature (Tolun, Altintas, & Artik, 2016). Microcapsules of polyphenols from green tea obtained through freeze-drying were also found to retain higher antioxidant activity than those obtained through spray drying (Pasrija, Ezhilarasi, Indrani, & Anandharamakrishnan, 2015), which implies that low temperature during freeze-drying contribute to maintaining constant antioxidant activity.

3.4. Microstructure and color difference

In the optical microscopy images taken at 32X (supplementary material), some differences in the characteristics of the powder are observed according to the encapsulating material and the concentration. The microcapsules have an irregular and indented aspect, similarity to the microcapsules obtained by Saikia, Mahnot, and Mahanta (2015), who encapsulated Averrhoa carambola pomace by lyophilization with maltodextrin. Likewise, Mahdavee Khazaei et al. (2014) reported that microcapsules of saffron petal's anthocyanin obtained by freeze-drying with maltodextrin and gum Arabic have similar characteristics, with amorphous glassy structure that could protect anthocyanins from the effect of heat and oxygen. On the other hand, Saavedra-Leos et al. (2014) showed that orange juice encapsulated with inulin through spray drying has a similar structure to that of the I-30 maqui powder. It was also observed that the MI-10 maqui powder with maltodextrin had glassy structure with dark and bright color, while the mixtures with inulin, showed a compact structure that was intensified with the increase in the proportion of wall material, moreover with weaker and matt coloration. The maqui powders with gum Arabic were of darker color. In the case of binary mixtures, the combined effects of the components could be seen, microcapsules with inulin, like the MI powders showed a lack of gloss, while MG microcapsules were dark and glossy. The different colors of the samples may suggest that there was an effect of the encapsulating material on the appearance of the powders, and these effects could be related to the molecular weight, polymerization degree and structure of encapsulating materials.

Fig. 4 shows the differences at microstructural level of the freeze-dried maqui extract powders that are granules rather than microspheres obtained during spray-drying (Fredes et al., 2018). Maltodextrin as encapsulating PS provides an irregular broken porous structure of very thin sheet-like particles with rather smooth surfaces (Fig. 4a). On the other hand, gum Arabic powder (Fig. 4b) had a continuous compact structure with very smooth surfaces, while the inulin powder (Fig. 4c) albeit compact and continuous had a rugged porous surface that was probably responsible for the matt coloration of the microcapsules. The powders from the mixture of polysaccharides showed microstructures with combined characteristics derived from the pure PS. Maltodextrin tended to formation of smaller structures causing disruption in the continuous structure of either the gum Arabic (Fig. 4d) or the inulin (Fig. 4f) powders. The SEM images confirmed the undefined indented glassy structures of the freeze-dried maqui powders, in accordance with the description of Fang and Bhandari (2010), that encapsulation method as well as the grinding process directly affects shape and size of the produced powders.

The color difference (ΔE), compared to freeze-dried maqui powder, was lowest for the maltodextrin powder and highest for the inulin powder prepared with 10% PS. This could be related to the fine smooth microstructure of the maltodextrin powder and the rugged surface of the inulin powder. ΔE can also be calculated using Eq. (4) with the constant coefficients *a* to *g* given in Table 2 for any combinations of PS at 10, 20 or 30% level. After 60 days storage at 25 °C the least color difference of 17.28 would be obtained for the maqui extract powders with weight fraction of 0.365 and 0.635 for maltodextrin and gum Arabic respectively. In general, the powder produced with 10% PS maintained a value of $\Delta E < 27$, even after 60 days storage at 25 °C. This color stability has been related to the low water activity of the

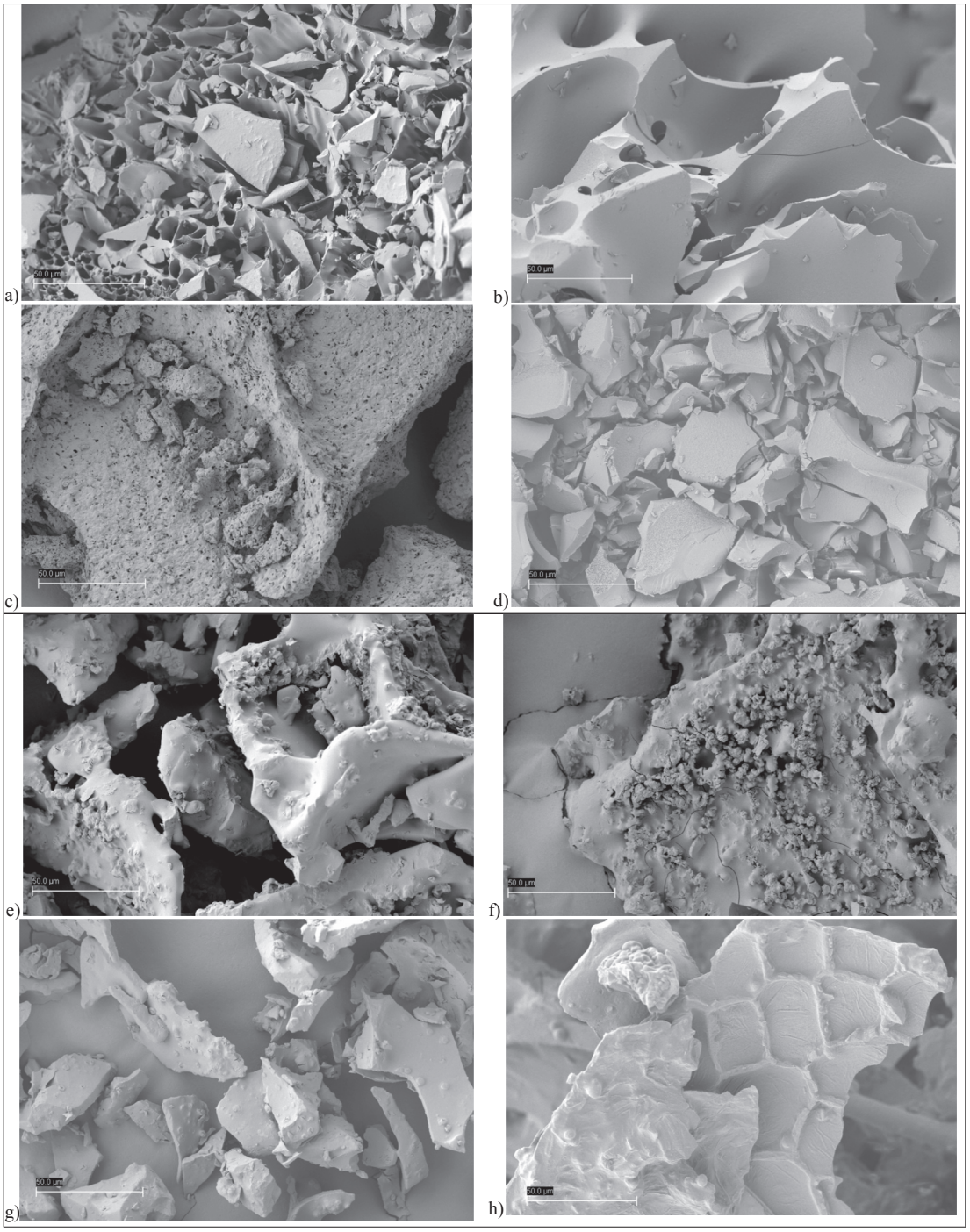

Fig. 4. Microstructure of the freeze-dried powder by scanning electron microscope.

powder. According to Rocha-Parra et al. (2016) encapsulated polyphenols of red wine through freeze-drying with maltodextrin and gum Arabic had stable CIE-Lab color values at a_w of 0.11, while at a_w of 0.33 significant changes in color values were observed. In this study, a_w did not exceed 0.3 and maltodextrin or gum Arabic powders would be the best PS for color preservation.

4. Conclusions

The retention of bioactives from maqui berries extracts depend on the type and concentration of the encapsulating agent used. The polysaccharides maltodextrin and inulin are more appropriate than gum Arabic for use as a protective agent in producing maqui extract powders by freeze-drying. A suspension of 10% polysaccharides produced the best results with a high retention of the bioactive properties (TPC, TAC and antioxidant activity) that may be obtained even after 60 days storage at 25 °C. Compared to powdered freeze-dried maqui, inulin powders produced better retention of antioxidant activity, while maltodextrin powders showed better retention of phenolics. Color of maltodextrin and gum Arabic powders were not significantly different to original color of powdered freeze-dried maqui berries, as opposed to the inulin powders. The simplex centroid mixture design enabled construction of an easy model to calculate quality parameters of the maqui powders during storage. The maqui extract powders are stable products suitable for potential nutraceutical applications and can be used in formulations as natural colorant in cosmetic or in foods products, like

dairy products and beverages.

CRediT authorship contribution statement

Jorge Romero-González: Conceptualization, Investigation, Methodology. **Kong Shun Ah-Hen:** Data curation, Methodology, Writing - original draft, Supervision. **Roberto Lemus-Mondaca:** Software, Writing - review & editing. **Ociel Muñoz-Fariña:** Methodology, Supervision.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.foodchem.2019.126115.

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