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Research article

Physicochemical characterization of sangorache natural colorant extracts (A*maranthus quitensis* L.) prepared via spray- and freeze-drying

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Abstract: In this study, we sought to prepare sangorache encapsulate (*Amaranthus quitensis L.)* by spray- and freeze-drying, using three different concentrations (3, 5 and 7%) of the encapsulating agent, maltodextrin. To atomize the powder, we used a Buchi mini spray-dryer B-290 with three inlet temperatures (140, 160 and 180 ℃) and a laboratory freeze dryer with a −0.8 bar pressure for 4 days. During atomization, the powders presented lower moisture content and water activity with the addition of maltodextrin; however, there were no significant changes in the moisture of the freeze-dried powders. Colorimetric analysis revealed that the luminosity (*L*) and chroma (*C*) values changed as maltodextrin concentration increased. The structures of particles were examined by scanning electron microscopy (SEM) SEM, and very different morphological characteristics resulted from the two drying methods. Also, apparent density, hygroscopy and dissolution testing revealed significant differences between the powders. Sangorache antioxidant compounds were preserved to a greater extent by applying low concentrations of the encapsulating agent and by the freeze-drying technique. The natural colorant from sangorache inflorescences could serve as an additive to improve food color in the industry.

Keywords: antioxidant capacity; colorant; freeze drying; microstructure; sangorache; spray drying

1. Introduction

The genus *Amaranthus* contains different plant species that are often used in agriculture, food or pigments as a high-quality colorant source owing to their rich colorful leaves, inflorescences, stems and seeds [1,2]. Sangorache plants (*Amaranthus quitensis L.)* are typically found in the Andean region. The plant is purple or red, and its seed is black [3]. Sangorache inflorescence contains natural pigments called "betalains," which are responsible for the plant color and have important antioxidant properties [4]. Betalains are synthetized from the amino acid tyrosine, from compounds with two structural groups, betacyanins (red-purple) and betaxanthins (yellow-orange). They are beneficial to human health because of their antioxidant, anticancer, and antimicrobial activities [5]. Betalains could be used as a substitute for betanin from red beets in the food colorant industry [2,6]. Consumers consider artificial pigments to be harmful and undesirable. Owing to the current demand for products with natural pigments, food manufacturers have been focusing on the use of natural color substances [7].

The encapsulation of bioactive compounds has been proposed as a successful alternative to improve the stabilization of betalains and ensure their bioavailability when employed as food colorants [8]. Thus, microencapsulation can provide a physical barrier between the core compound and other product components. Only a few agents can be used with these compounds. However, the selected agent is dependent on the core material and the desired characteristics of the final product [9,10]. The most commonly used material for microencapsulation is maltodextrin (Ravichandran *et al.*, 2014). Maltodextrin is an effective phenolic compound used for encapsulation. Further, it is associated with a low cost and bland flavor. Maltodextrin has low viscosity with high solids ratio [11]. Other techniques, such as spray- and freeze-drying, have been developed to microencapsulate bioactive compounds. Spray-drying is the most widely used technique for preparing dry and stable food additives and flavors. This process is efficient and economical and could improve the stability of betalains [9,10]. Freeze-drying is used to dehydrate heat-sensitive materials and is the most ideal method for drying sensitive plant pigments [11]. However, the technology involved in the process is more expensive and demands higher energy than other drying techniques [9]. This study sought to obtain sangorache extracts by spray- and freeze-drying, with maltodextrin employed as the encapsulating agent.

2. Materials and methods

2.1. Raw material

Amaranthus quitensis L. inflorescences (INIAP-Rubí variety) were provided by the National Legumes Program and Andean Grains (National Institute of Agricultural Research), and the maltodextrin with 18 dextrose equivalent (DE) was provided by Quinhuangdao Lihua Starch Co., LTD., China).

2.2. Extract liquid preparation

Stalk and leaves were removed from the inflorescences, which were allowed to dry in a forced-air oven (Memmert, Büchenbach, Germany) at 50 ℃ for 2 h until approximately 30% humidity was

achieved. After the inflorescences were milled (Thomas Wiley, model 4, Swedesboro, NJ, USA), 1 L of distilled water was added to 1 kg of inflorescence powder to form a suspension (1:1 ratio). The extract was stirred in a shaker (KitchenAid, Michigan, USA) for 1 h, filtered and stored in plastic bottles at −10 ℃ until further analysis.

Analysis of extract was carried out to determinate total solid (AOAC method), the total phenolic content, betalain content and Trolox equivalent antioxidant capacity according to the methodology described in sections 2.6.1, 2.6.2 and 2.6.3.

2.3. Spray-drying

Twelve hours before the experiment, the extract was moved from −10 ℃ to 4 ℃ until fully defrosted. Maltodextrin (3, 5 and 7%) was added to the extract, and the mixture was shaken for 5 min (Cole Parmer agitator, Vernon Hills, USA). A mini spray-dryer, Buchi B-290 (Flawil, Switzerland), was used in the spray-drying process. The inlet temperatures were 140, 160 and 180 ℃, and the equipment was operated under the following conditions: 35 m^3/h aspiration rate, 5 mL/min flow rate and 473 L/h air flow. The powders were packed in polyethylene bottles and stored at −10 ℃.

2.4. Freeze-drying

Three concentrations of maltodextrin (3, 5 and 7%) were added to the extract, which was subsequently shaken for 5 min. The samples were placed in polypropylene trays and frozen at −20 ℃ for 24 h (LABCONCO, Kansas, USA) at −0.8 bar pressure. The powders produced were packed similarly to the spray-dried powders.

2.5. Determination of physical characteristics

2.5.1. Color measurements

Color measurements were recorded with a Portable Spectrophotometer DR LANGE, LZM 268 model (Chelmsford, United Kingdom), based on the CIE *L**, *a**, *b** color system.

2.5.2. Moisture and water activity

Moisture content was determined by the AOAC method (1996) [12]. The water activity was measured with an analyzer (Testo 650, Lenzkirch, Germany).

2.5.3. Apparent density

One gram of the sample was placed in a graduated centrifuge tube for centrifugation at 2500 rpm, 18 ℃ for 10 min. The volume was then recorded and used to calculate density [11].

2.5.4. Hygroscopic measurement

We used the hygroscopic methodology described by Cai and Corke (2000). Briefly, the samples (1 g) were placed in plastic hermetic cells with a saturated solution of Na_2SO_4 (81%). After three days, the hygroscopic moisture was measured and expressed as g of moisture per 100 g of dry solids (%).

2.5.5. Dissolution test

We added 1 mL of distilled water to 50 mg of samples at 25 ℃. The time required to reconstitute the powder was recorded using an electronic timer [13].

2.6. Determination of chemical characteristics

2.6.1. Total phenolic content

Total phenolic content was determined using Folin Ciocalteu 2N reagent. Absorbance was measured at 754 nm using an Evolution 201 spectrophotometer (Thermo Fisher Scientific, Massachusetts, USA), and the results are expressed in mg of gallic acid/100 g of dry sample [14].

2.6.2. Betalain content

Betalains extract was obtained with a 25% ethanol solution. The absorbance was measured at 540 and 480 nm to quantify betacyanins and betaxanthins, respectively, according to the methodology described by Ravichandran *et al.*, 2013. The contents of betacyanins and betaxanthins were calculated using the following equation and expressed as mg/100 g of dry sample.

$$BC;\ BX\ (\text{mg}/100\text{g}) = ((A \times DF \times Mw \times V)/\ (e \times l \times Pm)) \times 100 \quad (1)$$

A is the absorbance value at 540 or 480 nm, *DF* is the dilution factor, *Mw* is the molecular weight (550 g/mol for betacyanins and 308 g/mol for betaxanthins), *V* is the final extract volume, *e* is the extinction coefficient (60 000 L/(mol cm) for betacyanins and 48 000 L/(mol cm) for betaxanthins), *l* is the optical path (1 cm), and *Pm* is the sample weight.

2.6.3. Trolox equivalent antioxidant capacity

Trolox equivalent antioxidant capacity (TEAC) was determined using the 2´2´-azinobis (3-ethylbenzothiazoline-6-sulfonic acid) radical cation (ABTS) and ferric reducing antioxidant power (FRAP) methods. The compounds extraction was obtained with 50% methanol. We quantified them with ABTS solution. Simultaneously, a standard Trolox curve (2000 µM) was generated, and the absorbance was measured at 734 nm using an Evolution 201 spectrophotometer (Thermo Fisher Scientific, Massachusetts, USA). The results are expressed in µg Trolox Eq/g of dry sample (For the FRAP method, the extraction was performed with methanol solution (50%). Colorimetric quantification was performed with FRAP reagents (300 mM acetate buffer at pH 3,6 10 mM TPTZ and 20 mM ferric chloride). A standard Trolox curve (2000 µM) was generated with absorbance at 593 nm. The results are expressed in µg Trolox Eq/g of dry sample [6,15].

2.7. Morphological analysis by scanning electron microscope (SEM)

The surface morphology of the molecule was examined by a scanning electron microscope (TESCAN, S. A. Brno, Czech Republic). The powder was mounted on a stub with a double-sided carbon adhesive tape, coated with gold (20 nm) and imaged using a field emission gun (FEG). The accelerating voltage was 7 kV with magnifications of 1.67, 3.33, 16.7 and 33.3 kV. The SEM images were analyzed for spray dryer (3% maltodextrin, 140 ℃), freeze-dried (3% maltodextrin) and freeze-dried extract, without encapsulant. The particle size was determined by the estimation of the diameters of more than 800 particles using the software Image J and was reported in micrometers (μm), histogram graphs were made in the MATLAB program.

2.8. Statistical analysis

For the spray-drying statistical analysis, we used factorial analysis of variance (ANOVA). However, for the freeze-drying statistical analysis, we used a randomized design, with the INFOSTAT software package [16]. As a post hoc test, we applied Tukey's test. The results were significant if p was <0.05 and are expressed as the mean ± standard deviation.

3. Results and discussion

3.1. Chemical characteristics of sangorache extract

Table 1, shows chemical characteristics of sangorache extract. Inflorescences of *Amaranthus* contain natural pigments. These pigments present red and purple colors. Total phenols were 1897. 41 mg GAE/100 g. Also, betacyanins content prevailed in relation to betaxanthins. Other studies evaluated several species of *Amaranthus*; these researchers registered values between 42 and 199 mg/100 g of betacyanins. Variability can be attributed to the genetic conditions of crops, agricultural production systems and climatic conditions [4,6].

Table 1. Chemical properties of sangorache extract.

Total solids (%)		2.18 ± 0.16
Total phenols		
mg gallic acid/100 g		1897.41 ± 6.85
Betalains	Betacyanins	363.61 ± 4.33
(mg/100 g)	Betaxanthins	113.62 ± 3.21
Trolox equivalent antioxidant capacity	ABTS	70.64 ± 2.87
(μmol Trolox Eq/g)	FRAP	125.29 ± 3.98

mean ± standard deviation (n = 3).

3.2. Physical characteristics of powders

3.2.1. Color analysis

Color reflects the sensory attractiveness and the quality of powders produced in the spray- and freeze-drying processes [13]. Details of the color measurement results as well as the statistical analysis results are presented in Table 2. The luminosity, Chroma and *a** color system were statistically different between both powders. All treatments presented regions with red (+a) and blue (−b). When a high percentage of wall material was present, the powders appeared white, and saturation (Chroma) was recognized to increase with increasing maltodextrin concentration. The spray-dried powders were found to exhibit a higher luminosity than the freeze-dried powders. Spray-dried samples were lighter than freeze-dried powders; however, the latter provided higher color stability [17]. The hue angle (*H*), which indicates the tonality, was not significantly different between the two powders. However, a slight increase in *H* occurred with freeze drying. It can be seen in Figure 1.

Table 2. Color variations in the spray-dried and freeze-dried sangorache powders.

Drying process (°C)	MD (%)	Luminosity (*L*)	Chroma (*C*)	Hue angle (*H*)	*a*	*b*
SD-140	3	31.15 ± 1.78^{cd}	16.07 ± 0.78^{ab}	344.52 ± 2.77	15.46 ± 0.56^{ab}	-4.30 ± 0.94
SD-160	3	31.76 ± 2.49^{cd}	14.15 ± 3.10^{b}	345.51 ± 3.55	13.64 ± 2.93^{b}	-3.68 ± 1.52
SD-180	3	29.99 ± 3.95^{d}	16.67 ± 1.19^{ab}	341.57 ± 6.25	15.73 ± 0.87^{ab}	-5.30 ± 2.02
SD-140	5	36.24 ± 2.45^{bcd}	15.02 ± 0.72^{ab}	345.53 ± 2.07	14.54 ± 0.56^{ab}	-3.76 ± 0.69
SD-160	5	41.08 ± 3.13^{ab}	14.89 ± 1.66^{ab}	348.02 ± 1.59	14.55 ± 1.54^{ab}	-3.12 ± 0.75
SD-180	5	38.31 ± 3.95^{abc}	15.21 ± 1.57^{ab}	345.00 ± 2.03	14.69 ± 1.61^{ab}	-3.90 ± 0.41
SD-140	7	33.30 ± 1.31^{cd}	19.71 ± 2.18^{a}	340.06 ± 2.93	18.46 ± 1.80^{a}	-6.75 ± 1.60
SD-160	7	41.23 ± 2.06^{ab}	17.58 ± 1.84^{ab}	343.77 ± 3.10	16.83 ± 1.48^{ab}	-4.97 ± 1.45
SD-180	7	44.22 ± 1.17^{a}	19.03 ± 1.69^{ab}	344.11 ± 2.26	18.28 ± 1.42^{a}	-5.25 ± 1.20
FD	3	21.24 ± 0.51^{b}	5.36 ± 0.66^{b}	349.63 ± 3.01	5.23 ± 0.65^{b}	-0.97 ± 0.29
FD	5	24.31 ± 0.94^{a}	7.61 ± 1.02^{ab}	351.27 ± 4.89	7.50 ± 0.41^{ab}	-1.02 ± 0.41
FD	7	24.99 ± 0.45^{a}	10.26 ± 1.89^{a}	351.55 ± 3.16	10.13 ± 1.95^{a}	-1.45 ± 0.29

Notes: *Values associated with different letters within a column are significantly different ($P < 0.05$); mean ± standard deviation (n = 3). SD: spray-drying, FD: freeze-drying. MD: maltodextrin.

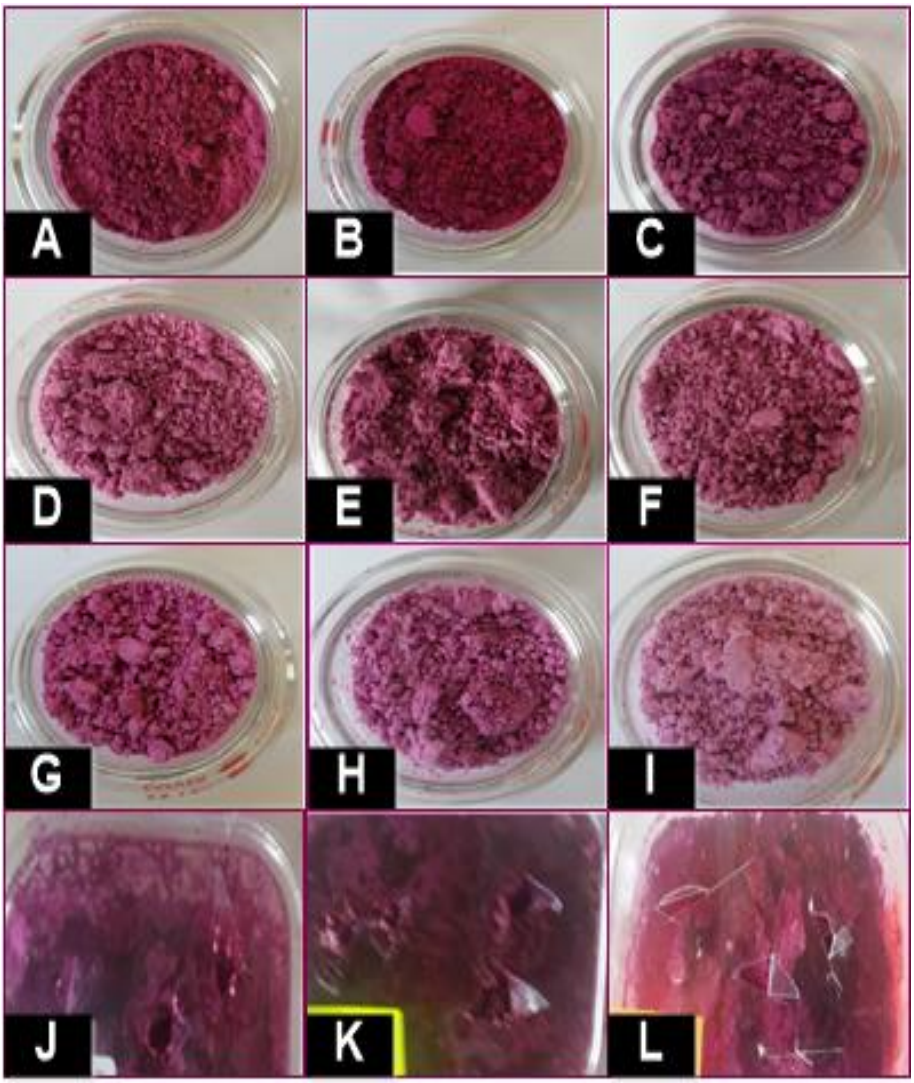

Figure 1. Color of the sangorache powders: spray-dried powders A) 140 ℃-3%, B) 160 ℃-3%, C) 180 ℃-3%, D) 140 ℃-5%, E) 160 ℃-5%, F) 180 ℃-5%, G) 140 ℃-7%, H) 160 ℃-7%, I) 180 ℃-7%; and freeze-dried powders J) 3%, K) 5%, L) 7%.

3.2.2. Moisture and water activity

Moisture content decreased as maltodextrin concentration increased. It is shown in Table 3. However, the inlet temperature did not significantly differ among the spray-dried powders. The application of 7% maltodextrin with inlet temperatures of 160 and 180 ℃ allowed us to obtain powders with 1.96 and 2.04% moisture, respectively. *Amaranthus* betacyanin pigments prepared via spray-drying reached 6.80 to 1.85% moisture using different inlet air temperatures and wall material [11]. The microencapsulates of betacyanin from colored organic quinoa ranged in moisture from 8.26 to 2.14%. This difference in moisture was attributed to the concentration of the encapsulating agent [18]. Nonetheless, the addition of maltodextrin increased the total solid content and reduced the amount of water lost through evaporation [13,19].

Table 3. Physical properties of spray- and freeze-dried sangorache powders.

Drying process	MD (%)	Moisture (%)	Water activity	Apparent density (g/mL)	Hygroscopy (%)	Dissolution Test (s)
SD-140	3	5.44 ± 1.02^{d}	0.31 ± 0.01^{c}	0.46 ± 0.05	12.68 ± 0.98^{bc}	50.67 ± 2.08^{a}
SD-160	3	5.08 ± 0.80^{d}	0.27 ± 0.02^{c}	0.46 ± 0.05	11.32 ± 0.39^{bc}	55.67 ± 1.53^{ab}
SD-180	3	4.97 ± 0.39^{cd}	0.26 ± 0.01^{c}	0.45 ± 0.01	12.43 ± 0.14^{bc}	61.80 ± 0.60^{b}
SD-140	5	3.44 ± 0.05^{b}	0.26 ± 0.04^{bc}	0.45 ± 0.01	9.08 ± 0.39^{a}	69.60 ± 1.20^{c}
SD-160	5	3.61 ± 0.16^{bc}	0.19 ± 0.04^{a}	0.44 ± 0.01	11.43 ± 0.71^{bc}	71.80 ± 1.83^{c}
SD-180	5	2.96 ± 0.34^{ab}	0.19 ± 0.004^{a}	0.44 ± 0.02	11.04 ± 0.33^{b}	72.80 ± 1.25^{c}
SD-140	7	2.46 ± 0.13^{ab}	0.20 ± 0.01^{ab}	0.47 ± 0.01	12.18 ± 0.58^{bc}	86.40 ± 5.23^{d}
SD-160	7	1.96 ± 0.15^{a}	0.16 ± 0.02^{a}	0.47 ± 0.03	12.76 ± 0.71^{c}	89.90 ± 2.74^{d}
SD-180	7	2.04 ± 0.32^{a}	0.14 ± 0.02^{a}	0.48 ± 0.02	11.83 ± 0.65^{bc}	86.80 ± 2.84^{d}
FD	3	11.42 ± 0.39	0.27 ± 0.01^{ab}	0.20 ± 0.001	16.50 ± 2.19^{b}	29.67 ± 2.52^{a}
FD	5	11.33 ± 0.62	0.30 ± 0.02^{b}	0.23 ± 0.03	14.19 ± 0.05^{ab}	44.33 ± 1.53^{b}
FD	7	10.91 ± 0.41	0.26 ± 0.01^{a}	0.24 ± 0.02	12.49 ± 0.49^{a}	55.00 ± 3.00^{c}

Note: *Values associated with different letters within a column are significantly different ($P < 0.05$); mean ± standard deviation (n = 3). SD: spray drying, FD: freeze-drying, MD: maltodextrin.

The freeze-dried powders presented no significant differences. However, when 7% maltodextrin was applied, sample moisture decreased to 10.91%. Anthocyanins of black glutinous rice reached 8.73% moisture when different proportions of maltodextrin were employed [19]. In *Aloe vera* powders, moisture content was 3.4% using 20% maltodextrin [20]. As the process conditions could influence dehydration, the type of wall material can rapidly absorb surrounding moisture due to the presence of more hydrophilic groups [19]. The spray-dried powders had the highest water activity at 0.31 with 3% maltodextrin at 140 ℃ and the lowest with 7% wall material at 180 ℃ (0.14). Therefore, the increase in both maltodextrin concentration and temperature reduced water activity. Such findings align with those of other studies [18,21].

Freeze-dried powders displayed a range of 0.26–0.30 for water activity. The water activity values of *Aloe vera* powders were in the range of 0.14–0.17, and black glutinous rice powders reached values between 0.42 and 0.52 [19]. The spray-dried powders had a greater shelf life than freeze-dried powders; however, freeze-dried sangorache had the highest water activity and moisture, with high hygroscopicity and water absorbance [22].

3.2.3. Apparent density

The apparent density did not significantly differ between the spray- and freeze-dried powders ($P > 0.05$). However, with the application of 7% maltodextrin, a slight increase in density was observed. The inlet temperature of the spray-dried samples had no influence on this parameter. Similar values were reported for microencapsulated quinoa betacyanin, (0.41–0.55 g/mL, Aguilar-Tuesta *et al.*, 2018); however, these results were slightly lower than those described for encapsulated *Amaranthus* betacyanin (0.52–0.67 g/mL) [11].

The apparent density of the freeze-dried powders ranged from 0.20 to 0.24 g/mL. The density of freeze-dried sumac extract powders was 0.27–0.28 g/mL [23]; however, in *Aloe vera* powders, the density was higher, at 0.31–0.38 g/mL [20]. The density of freeze-dried powders was lower than that of spray-dried powders. Products with low density often have poor storage stability, resulting in oxidative degradation of bioactive compounds [11,19]. Thus, the wall material was found to

influence the density. As maltodextrin has a high density, it easily fits within the spaces between the particles, occupying less volume [20].

3.2.4. Hygroscopy

The hygroscopicity values of the spray-dried powders did not present significantly different effects with temperature (P >0.05). With the encapsulation of eggplant anthocyanins, treatments with lower maltodextrin content (15%) presented higher hygroscopicity [21]. Cai and Corke (2000) reported an average value of 47.5% with maltodextrin (15 DE) at various drying temperatures. However, the opposite effect was found in acai powders. The lowest hygroscopic values were obtained with the highest maltodextrin concentration [24]. Freeze-dried powders produced with 7% maltodextrin had a lower hygroscopicity value than those produced with 3% maltodextrin. Some studies suggest that maltodextrin is a material with low hygroscopicity and have confirmed its efficiency as a carrier agent [11,24].

3.2.5. Dissolution test

The dissolution test revealed significant differences between the spray- and freeze-dried powders. The treatments prepared with 7% encapsulating agent dissolved faster than those prepared with 3% of this agent. In spray-dried powders, the inlet temperature was found to influence the dissolution. In fact, the wettability time was found to be shorter at 140 ℃. Thus, in spray-dried watermelon powders, the time taken for the powders to fully reconstitute was relatively shorter. This phenomenon might be related to the moisture in the powder [13]. Quinoa betacyanin powders were dissolved within 35.50 s; however, with an increase in maltodextrin concentration, the reconstitution time was 537 s. The powders with maltodextrin as a material wall were found to have better solubility and dispersibility [18].

Furthermore, with freeze-dried powders, higher concentration of the encapsulating agent increased dissolution time. In *Aloe vera* powders, the dissolution time was 16.33 s (20% maltodextrin), and the lowest time of 11.67 s was achieved with the 10% concentration [20]. The high porosity developed in the freeze-dried powders played a significant role in the reconstitution properties [19,23].

3.3. Chemical characteristics of powders

3.3.1. Total phenolic content

The chemical functional characterization of sangorache powders revealed significant differences between spray- and freeze-dried powders. It is shown in Table 4. In spray-drying, the content of maltodextrin and inlet temperature affected the total phenol content. The treatments prepared with 3% maltodextrin were found to preserve these compounds compared to those prepared with 5% and 7% concentrations. Furthermore, phenolic compounds degraded owing to the effect of temperature. For sumac powder, total phenols ranged between 1 165 and 1 450 mg GAE/100 g, when different drying conditions and amounts of maltodextrin are applied [25]. These values were lower than those of eggplant peel powder (503 and 522 mg GAE/100 g) [26]. Freeze-dried powders showed a higher content of phenolics than spray-dried powders, with the application of 3% maltodextrin preserving a greater phenolic proportion than the other maltodextrin concentrations. This drying process preserved bioactive compounds based on the sublimation phenomena [27].

Table 4. Chemical properties of spray-dried and freeze-dried sangorache powders.

Drying process	MD (%)	Total phenols mg gallic acid/100 g	Betalains mg/100 g		Antioxidant Capacity µmol Trolox Eq/g	
			Betacyanins	Betaxanthins	ABTS	FRAP
SD-140	3	1873.72 ± 4.13^{a}	474.18 ± 3.90^{a}	157.76 ± 4.24^{a}	377.15 ± 4.03^{a}	87.18 ± 3.71^{a}
SD-160	3	1244.98 ± 4.64^{c}	429.76 ± 3.62^{b}	149.57 ± 3.38^{b}	41.15 ± 4.17^{e}	58.18 ± 3.98^{b}
SD-180	3	1287.04 ± 2.06^{b}	243.57 ± 2.41 c	66.81 ± 3.59^{c}	40.06 ± 2.49^{e}	51.51 ± 4.14^{b}
SD-140	5	709.34 ± 5.06^{d}	129.97 ± 3.92^{de}	37.85 ± 1.22^{e}	67.76 ± 2.21^{b}	25.06 ± 2.12^{cd}
SD-160	5	629.90 ± 4.22 ^{g}	121.19 ± 4.58^{ef}	36.81 ± 2.33 ^{e}	64.72 ± 2.62^{bc}	20.88 ± 1.01^{d}
SD-180	5	685.75 ± 3.02 ^{e}	113.71 ± 4.25 ^{f}	36.45 ± 1.82 ^{e}	52.54 ± 5.64^{d}	23.50 ± 0.48^{cd}
SD-140	7	659.20 ± 4.00^{f}	136.74 ± 5.08^{d}	55.63 ± 3.08^{d}	61.61 ± 1.89^{bcd}	28.92 ± 2.12^{c}
SD-160	7	556.20 ± 3.45^{i}	80.70 ± 2.86^{h}	33.22 ± 3.35^{e}	59.88 ± 0.89^{bcd}	19.69 ± 2.95^{d}
SD-180	7	575.95 ± 1.15^{h}	98.22 ± 3.41^{g}	32.71 ± 0.46^{e}	57.99 ± 3.75^{cd}	11.09 ± 0.23^{e}
FD	3	3292.22 ± 6.61^{a}	743.76 ± 6.72^{a}	260.31 ± 6.79^{a}	328.93 ± 4.87^{a}	160.27 ± 2.98^{a}
FD	5	2997.62 ± 5.09^{b}	708.59 ± 4.15^{b}	192.07 ± 3.18^{b}	242.16 ± 3.40^{b}	132.55 ± 4.86^{b}
FD	7	1507.22 ± 7.05^{c}	341.67 ± 2.97^{c}	94.95 ± 2.48^{c}	137.24 ± 3.38^{c}	82.611 ± 3.87^{c}

Note: *Values associated with different letters within a column are significantly different ($P < 0.05$); means ± standard deviations (n = 3). SD: spray-drying, FD: freeze-drying, MD: maltodextrin.

3.3.2. Betalain content

Betalains are nitrogenous pigments that have two structural groups, betacyanins (red-violet) and betaxanthins (yellow-orange) [5]. Sangorache inflorescences extract contains more betacyanins than betaxanthins. The application of spray-drying with 140 ℃ and 3% maltodextrin preserved these compounds compared to higher temperatures and concentrations. Moreover, quinoa encapsulated powders had 19.95 mg/100 g of betacyanin [18], and microencapsulated products from purple cactus pear maintained betalain levels between 11.33 and 35.93 mg/100 g when different concentrations of maltodextrin and gelatin were employed [28].

The content of betalains in freeze-dried powders was preserved more than that in spray-dried powder. During spray-drying, an increase in inlet temperature occurred, which might have reduced the betalain content. Freeze-drying is a technique that permits efficient stabilization of thermo-sensitive substances; with lower temperatures, it was better for the stability of betalain than spray-drying with high temperatures [17].

3.3.3. Trolox equivalent antioxidant capacity

Encapsulated pigments obtained by spray- and freeze-drying displayed antioxidant capacities, with marked variations observed between the ABTS and FRAP methods. The antioxidant capacity of a substance is a function of in its capacity to inhibit oxidation. The antioxidant reacts with free radicals and prevents oxidation of the substrate. The antioxidant capacity of a substance should be evaluated with different methods to derive its lipophilic and hydrophilic characteristics, which reflect its different mechanisms and reactivity [29,30].

The application of 3% maltodextrin and an inlet temperature of 140 ℃ allowed a more suitable preservation of the antioxidant capacity than higher maltodextrin concentrations and temperature. Eggplant powder research had 252.8 µmol Trolox Eq/g (ABTS) with 15% of maltodextrin and 180 ℃ [21].

The application of different concentrations of encapsulating agents could influence the antioxidant capacity. However, the antioxidant compounds were better preserved via freeze-drying than spray-drying.

3.4. Morphological analysis by scanning electron microscope (SEM)

The SEM images of spray-dried and freeze-dried sangorache powders and freeze-dried extract without encapsulant are shown in Figure 2. Spray-dried sangorache powder showed spherical particles and particles with irregular, concave and wrinkled surfaces. The concavity and wrinkled particles permit the rapid evaporation of moisture during the spray dryer process [31], although wrinkled particles are more sensitive to oxidation reactions. In the same way, these particles could lead to the formation of agglomerates that hinder the dispersion of the dust when reconstituted in a solvent [32,33]. Also, blackberry and tamarillo powders presented rough surfaces, and some particles presented deformation on the surface [34].

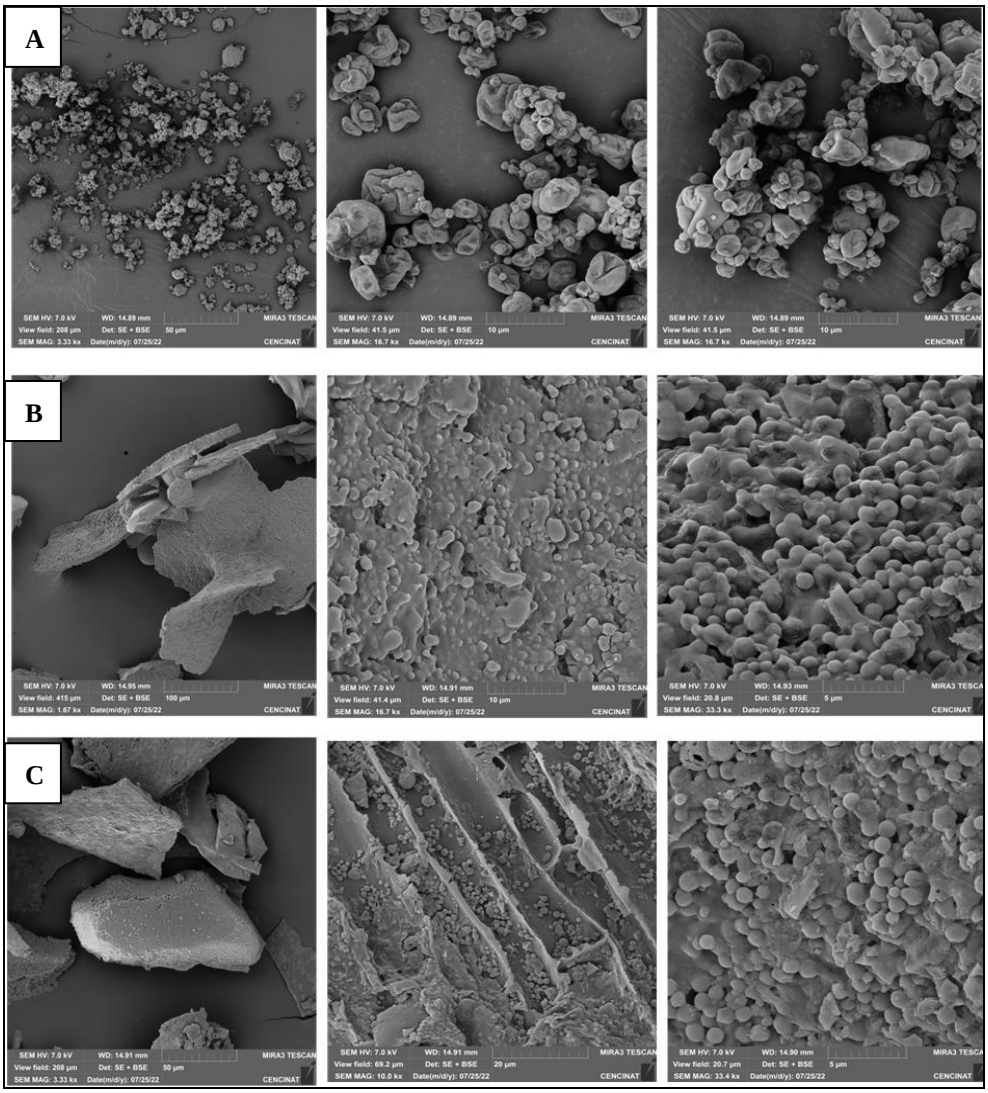

Figure 2. SEM images of the sangorache powders: A) Spray drying process (140 °C, 3%), B) Freeze drying process (3%) and C) Freeze drying process (without encapsulant).

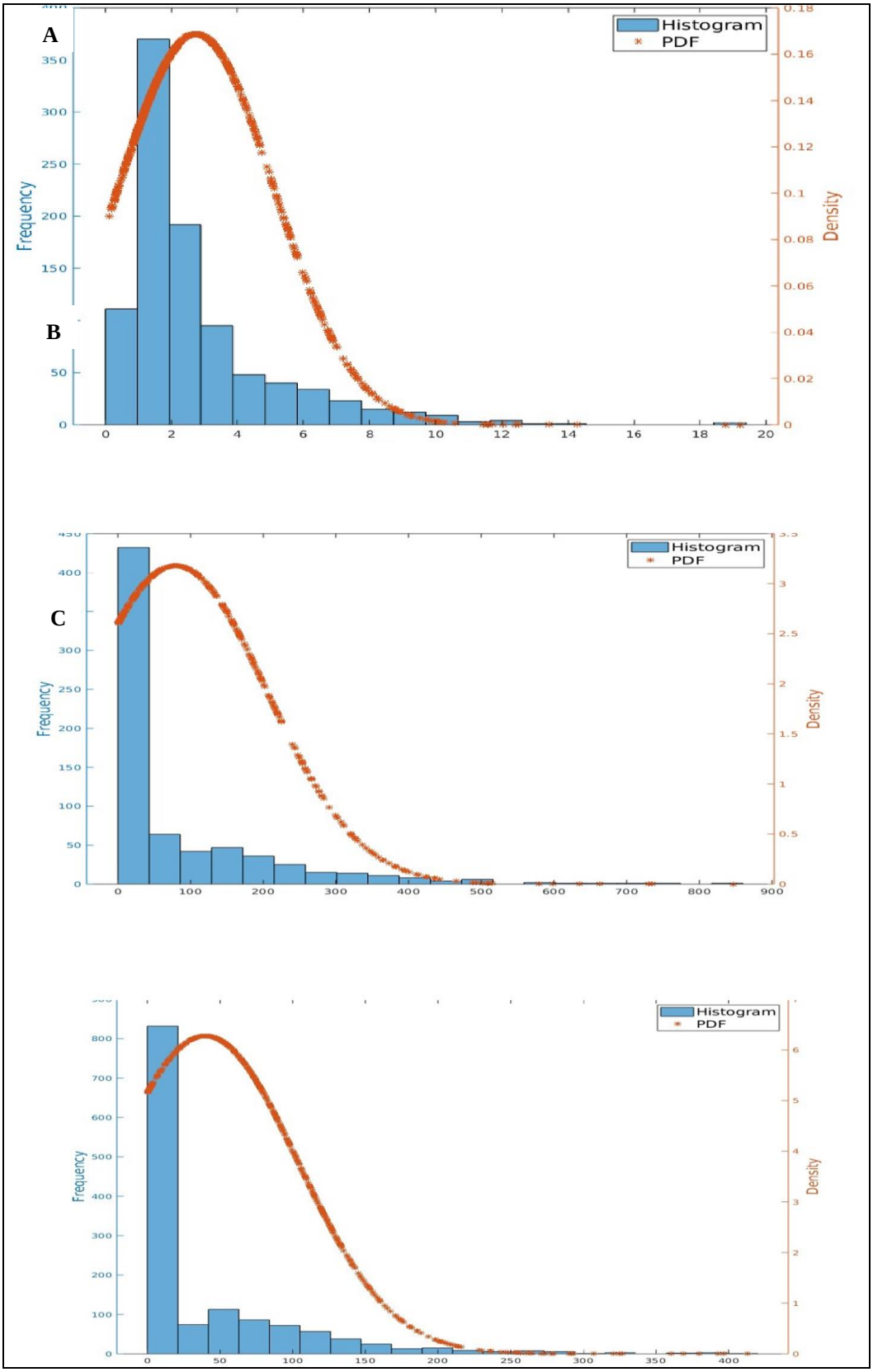

Figure 3. Particle size distribution of the sangorache powders: A) Spray drying process (140 ℃, 3%), B) Freeze drying process (3%) and C) Freeze drying process (without encapsulant).

However, freeze-dried sangorache powder (3% maltodextrin and without encapsulant) exhibited different morphological structure. The particles were not homogenous; they presented flakes or crystal shapes. Freeze-drying process Freeze-dried process, water is a support surface. This is removed by sublimation, and the encapsulant contains a porous structure. Pressure and temperature influence powder structures [35,36]. The type of encapsulant and its concentration help cracks on the surface. Maltodextrin with a low dextrose equivalent has greater irregularity and cracks in the particles [34].

The particle size distribution of sangorache is presented in Figure 3. Spray drying presented smaller particles than freeze drying. Spray dried particles were distributed around 10 μm, while freeze drying particles (3% maltodextrin and without encapsulant) were distributed around 800 μm. The difference in particle size distribution, especially affected by the two methods, was because of the materials and processes involved [36]. The drying temperature is determinant since it significantly influences the conservation of thermosensitive bioactive compounds, as well as the sizes, shapes and surfaces of the particles. Spray drying applies powerful atomization, whereas in freeze drying, the final particle size only depended on the grinding procedure instead of the drying process [33,37].

4. Conclusions

Sangorache powders were produced using spray- and freeze-drying technologies and three maltodextrin concentrations (3, 5 and 7%). The application of these conditions was found to affect the physicochemical characteristics and morphological structure of the produced powders. Maltodextrin resulted in a suitable solubility and could thus be recommended for the encapsulation of food ingredients. Possible further uses of sangorache powders and their stability conditions should be assessed in future projects.

Conflict of interest

All authors declare that have no conflict of interest.

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