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Stabilization of natural antioxidants from Chillangua (Eryngium foetidum) by encapsulation

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Abstract

The Chillangua (Eryngium foetidum) is 4a species of significant scientific interest due to its history and traditional uses throughout the world. Therefore, the focus of this research was on stabilizing the functional and antioxidant components of Chillangua through encapsulation with maltodextrin and Arabic gum. A central composite experimental design was used to evaluate the variables: type of encapsulant (maltodextrin and Arabic gum), encapsulant concentration (3% and 10%), and atomization temperature (130 and 160°C). The results showed that the fresh plant contained 1.34% fat, 17.47% protein, 22.90% fiber, 14.24% ash, and a pH of 6.88. Additionally, concentrations of 3,883.77 µg/g of chlorophyll a, 1,761.72 µg/g of chlorophyll b, 889.19 µg/g of carotenoids, 61.21 mg/g of total polyphenols, 0.02 mg/g of flavonoids, 1.29 mg/g of ascorbic acid, 40 µmol Trolox Eq/g, and 91.60 µmol Trolox/g of antioxidant activity by 2´2´-azinobis (3-ethylbenzothiazoline-6-sulfonic acid) (ABTS) and 2,2-difenil-1-picrilhidrazilo (DPPH), respectively, were identified. When encapsulating Chillangua, it is identified that a reduction occurs in its functional components, as well as its antioxidant activity. The maximum process optimization was achieved at 160°C with 3% maltodextrin and 3% Arabic gum. The irregular morphology of the particles contained C, O, K, Zn, Cl, Al, Ca, and Na.

Keywords: biopolymers; encapsulation; morphology; antioxidant capacity.

Practical application: The practical application of encapsulated extracts extends to the pharmaceutical and food industries. Their inclusion in formulations for food production, as well as in the manufacturing of pharmaceutical and personal care products, provides an opportunity to harness the preservation of Chillangua's sensory attributes and antioxidant activity, delivering benefits for both health.

1 INTRODUCTION

Chillangua (*Eryngium foetidum*) is a species with astonishing potential, displaying high adaptability to different climates and conditions (Restrepo et al., 2005).

Its chemical composition testifies to its richness and versatility (Leitão et al., 2020). The roots, stem, and leaves harbor saponins, flavonoids, calcium, iron, carotenoids, ascorbic acid, ferulic acid, and alkaloids, among others (Blair & Madrigal, 2005), making it a promising candidate for the food and medicinal industry (Sharma & Singhvi, 2018). It has also garnered interest in health and well-being research (Bassolino et al., 2022).

This vegetable, which is rich in antioxidants, is sparking growing interest due to its remarkable health benefits for humans (Dalukdeniya & Rathnayaka, 2017). However, the stability of functional and antioxidant compounds poses a challenge (Chen et al., 2019). Antioxidants in plants act as defenders against pests and provide color and flavor (Aybastier, 2021). On the contrary, when consumed by humans, these compounds help prevent oxidative damage and contribute to overall health (Jideani et al., 2021).

Biopolymers, serving as thickeners, emulsifiers, and gelling agents, are ideal for encapsulating bioactive compounds (Vega & Montaño, 2020). Maltodextrin obtained from starch hydrolysis (Food and Drug Administration, 2021) and Arabic gum from Acacia Senegal (Badui, 2006) play a crucial role in retaining volatile substances and the permeability of encapsulated walls. They prevent caramelization, preserving flavors and colorants (Muhamad et al., 2018).

Encapsulation is an innovative technology aimed at preserving and protecting active compounds, preventing degradation due to environmental factors. Furthermore, it enables the controlled release of bioactive compounds (Sandoval-Peraza et al., 2017). However, this technique depends on the encapsulating material used (Ortiz-Romero et al., 2021), does not efficiently control particle size, and is not suitable for highly viscous solutions (Kalušević et al., 2017). The research objective focuses on achieving the stabilization of natural antioxidants from Chillangua (*E. foetidum*) through encapsulation.

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2 MATERIALS AND METHODS

2.1 Raw material

Eryngium foetidum leaves were provided by the Amazon Experimental Station of the National Institute of Agricultural Research, INIAP. Arabic gum and maltodextrin with 18 dextrose equivalent (DE) were provided by Quinhuangdao Lihua Starch Co., Ltd., China.

The clean leaves were processed in two different ways. One set of leaves underwent freeze-drying: 24 h of freezing followed by 5 days of lyophilization (LABCONCO, Kansas, USA) at –0.8 bar pressure. Another set of leaves was dried at 50°C with forced air (Memmert, Büchenbach, Germany) for 2 h. Once dried, the leaves were processed using the Retsch mill (Haan, Germany) to obtain a sample size of 0.5 μm.

2.2 Extract preparation

The extract was obtained by mixing 50 g of dried leaves with 600 mL of distilled water for 2 h at room temperature in a stainless-steel container (KitchenAid, Michigan, United States). Subsequently, a second extraction was performed using 100 g of the residues from the first extraction and 600 mL of distilled water. The resulting extract was filtered through different sieves and then combined and stored.

2.3 Atomization of the extracts

The portions of maltodextrin and Arabic gum were dissolved in 200 mL of the filtered extract and distilled water, respectively. The solutions were agitated separately and then mixed for atomization using mini Spray Dryer BUCHI B-290 equipment (Flawil, Switzerland).

The spray drying process was conducted as follows: once the equipment reached a pressure of 8 bar, the equipment and the refrigerant were turned on, operational parameters such as temperature and airspeed were adjusted, an initial cleaning step with distilled water was carried out, and then the extract was atomized (Sharma et al., 2003).

The collected powder from the process was measured for the following parameters: moisture content (Flores et al., 2021), color (DR LANGE spectrophotometer – colorimeter), and water activity (testo 650 – Lenzkirch, Germany). Additionally, a dissolution test was performed, involving the addition of 100 mg of encapsulated material into a test tube with 10 mL of distilled water, and the dissolution time was recorded (Quek et al., 2007).

2.4 Proximate composition

The contents of protein, fat, crude fiber, and ash were determined using standardized methods (AOAC, 1980, 2000). Carbohydrate content was calculated from the difference.

2.5 Color measurements

The color of the leaves was determined using the CIE L^* a^* b^* method with a portable Hach Lange spectro-color, LZM 268 (Chelmsford, United Kingdom) (Luna-Solano et al., 2019).

2.6 Morphological analysis by scanning electron microscope

The surface morphology of the molecule was examined by a scanning electron microscope (SEM) equipped with the JEOL EDS System (IT100LA). 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) (Sarabandi et al., 2019). The accelerating voltage was 20 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°C), freeze-dried (3% maltodextrin), and freeze-dried extract, without encapsulant. Additionally, an elemental analysis was conducted using energy-dispersive X-ray spectroscopy (EDS). 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).

2.7 Functional components and antioxidant activity in the encapsulates

The analysis of functional components and antioxidant activity of the encapsulates was carried out. Both the encapsulates and freeze-dried Chillangua were transformed into liquid extracts by mixing 0.8 g of the samples with 10 mL of 25% methanol while agitating for 10 min. Additionally, a second extraction was conducted using the residues from the first extraction with a similar methodology but subjecting them to a water bath (80°C) for 5 min. Subsequently, the two solutions were filtered, mixed, and stored in translucent containers.

2.7.1 Total phenolic content

The total phenolic content was determined using the Folin Ciocalteu 2N reagent. The absorbance was measured at 754 nm using an Evolution 201 spectrophotometer (Cachay-Morante et al., 2022) (Thermo Fisher Scientific, Massachusetts, USA), and the results are expressed in mg of gallic acid/100 g of dry sample (Waterhouse, 2002).

2.7.2 Flavonoid content

The flavonoid content was determined by mixing 3 mL of the extracts/blank with 6 mL of 2% aluminum chloride. After homogenization and incubation in the dark for 10 min, absorbance was measured at a wavelength of 430 nm (Quettier-Deleu et al., 2000).

2.7.3 Trolox equivalent antioxidant capacity

Trolox equivalent antioxidant capacity (TEAC) was determined using the 2´2´-azinobis (3-ethylbenzothiazoli ne-6-sulfonic acid) (ABTS) radical cation and 2,2-difenil-1-picrilhidrazilo (DPPH) methods. The compound extraction was obtained with 25% methanol. We quantified them with ABTS solution. Simultaneously, a standard Trolox curve (2,000 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 (Roca et al., 2020).

For the DPPH method, the extraction was performed with methanol solution (25%). Colorimetric quantification was performed with DPPH reagents (300 mM acetate buffer at pH 3.6, 10 mM TPTZ, and 20 mM ferric chloride). A standard Trolox curve (2,000 μM) was generated with absorbance at 517 nm. The results are expressed in μg Trolox Eq/g of dry sample (Vera & Schmalko, 2019).

2.7.3 Ascorbic acid

The ascorbic acid in the samples was extracted with an oxalic acid solution of 0.4% and 20% acetone and quantified using 2.6-dichlorophenol-indophenol (Egoville et al., 1988). The absorbance was measured at 520 nm. L-ascorbic acid was used as a standard.

2.7.4 Total carotenoids

The extraction of carotenoids was conducted with cold acetone and petroleum ether according to the methodology described by Rodriguez-Amaya and Kimura (2004). The absorbance of the ether extract was measured at a wavelength of 450 nm. The extinction coefficient of carotenoids in petroleum ether (2,500) was considered in the calculation of total carotenoids.

2.7.5 Chlorophyll and carotenoid content

In a light-protected beaker, 0.8 g of the encapsulates or freeze-dried Chillangua was added along with 15 mL of cold acetone. After continuous agitation for 2 days, the mixture was filtered into translucent 25 mL values and adjusted with acetone. The resulting extract was analyzed at three wavelengths: 662, 645, and 470 nm (Pataro et al., 2015).

2.8 Data analysis

The Statgraphics Centurion XV program was used to determine treatments through a central composite design, based on the factors under study: temperatures of 130 and 160°C and maltodextrin and Arabic gum concentrations of 3% and 5%. The design included two central points and two axial points, with an axial distance of 1.682, and was conducted without randomization, resulting in a total of 16 treatments (Table 1), with three replicates for each treatment.

The mathematical model used was as follows (Equation 1):

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i x_i + \sum_{i=1}^{k} \beta_{ii} x_i^2 + \sum_{i=1}^{k} \sum_{<j=1}^{k} \beta_{ij} x_i x_j + \varepsilon \quad (1)$$

Where:

β_0, β_i, and β_{ij} the intercept and the coefficients for linear quadratic and interaction terms, respectively. The leftover or unaccounted portion is represented as ε .

The analysis of response variables was conducted through an analysis of variance (ANOVA), with a confidence level > 0.05. The second-order polynomial model was selected based on the coefficient of determination R^2. The optimization of response variables was performed considering the design and response surface graphs. This allowed us to identify the maximum desirability of the variables and the best treatment. The particle size was measured using the ImageJ program, with a total of 900 repetitions for each treatment.

3 RESULTS AND DISCUSSION

3.1 Physical, chemical, functional, and antioxidant properties of Chillagua leaves

The color characteristics of the leaves in fresh state, on both the upper and underside, showed similarities in saturation (C*: 47.12 ± 9.83 and 46.96 ± 11.25); however, the upper side (L*: 28.5 ± 7.04, a*: -21.81 ± 4.66, b*: 41.48 ± 9.96) exhibited a slightly darker hue with greenish and bluish undertones compared with the underside (L*: 39.16 ± 4.53, a*: -17.32 ± 3.18, b*: 42.71 ± 9.67). Therefore, the leaves exhibit variations, but they do not display significant differences.

The physical, chemical, functional, and antioxidant capacity properties of leaves are detailed in Table 2. The pH was 6.88, along with a moisture level of 71.76%, findings that align with Rodrigues et al. (2022), with a range from 10.3 to 87%. The leaves presented low fat content and moderate protein, fiber, and ash content, and similar results have been reported by Tashi Lepcha et al. (2018) which revealed 3% ash, 31.50% fiber, 0.73% fat, and 2.63% protein. Also, Rodrigues et al. (2022) reported 1.95% fat, 6.32% fiber, and 5.25% protein in Chillagua leaves.

In compaction with other plant species, celery has the highest moisture content at 95%, while parsley shows higher values in terms of 28.38% protein, 14.42% fiber, 18.86% ash, and 46.97% ELN (digestible carbohydrates).

Table 1. Statgraphics Centurion XV experimental treatments.

Run	Temperature (°C)	Arabic gum concentration (%)	Maltodextrin concentration (%)
1	145	6.5	6.5
2	130	3.0	3.0
3	160	3.0	3.0
4	130	3.0	10
5	160	3.0	10
6	130	10.0	3
7	160	10.0	3
8	130	10.0	10
9	160	10.0	10
10	120	6.5	6.5
11	170	6.5	6.5
12	145	6.5	0.614
13	145	6.5	12.386
14	145	0.614	6.5
15	145	12.386	6.5
16	145	6.5	6.5

Note: Temperature and concentration were established through preliminary tests.

The chemical composition is influenced by factors such as fertilizers, environmental conditions (soil and geographical location), and pre- and postharvest processes (Rodrigues et al., 2022).

Polyphenols were also affected by the type of solvent used and the antioxidant enzymes that can degrade them, which explains the differences in the results found in the study for Campos et al. (2019) of 40.4 mg gallic acid/g. Therefore, the use of more efficient solvents is advisable.

The concentration of flavonoids is notably low compared with those by Aly (2010) which was 61.66 mg/g and Campos et al. (2019) which was 1.81 mg/g. The difference may be attributed partly to the extraction methodology and limited solubility of flavonoids in aqueous solvents (Singh et al., 2013). The ascorbic acid content was higher than that reported by Aly (2010) which was 0.02 mg/g and Rodrigues et al. (2022) which was 14.17 mg/g but lower than the value reported by Tashi Lepcha et al. (2018) which was 323.3 mg/g. This may be due to the environmental conditions in which the plants were grown, agronomic management, fertilization, and postharvest handling.

Photosynthetic pigments such as chlorophyll a, chlorophyll b, and carotenoids show discrepancies compared with the results obtained by Campos et al. (2019), which could be attributed

Table 2. Physical, chemical, functional, and antioxidant properties of Chillagua leaves, Celery leaves, and Parsley leaves.

Parameter		Chillangua leaves	Celery*	Parsley	Bibliography
Moisture	%FW	71.76 ± 2.51	95% 80.30%	88.00 ± 0.81	Singh, Nara, Rani, & Jaswal (2022) Fazal & Singla (2012) García Mahecha, Cortes Rodríguez, & Rodríguez Sandoval (2010)
Fat	% DW	1.34 ± 0.04	0.17% 0.6%	11.76 ± 0.43	Singh et al. (2022) Fazal & Singla (2012) García Mahecha et al. (2010)
Protein	% DW	17.47 ± 0.86	0.0–0.8%	28.38 ± 0.60	Fazal & Singla (2012) García Mahecha et al. (2010)
Fiber	% DW	22.90 ± 0.20	1.6% 1.4%	14.42 ± 0.37	Singh et al. (2022) Fazal & Singla (2012) García Mahecha et al. (2010)
Ash	% DW	14.24 ± 0.45	2.1%	18.86 ± 2.70	Fazal & Singla (2012) García Mahecha et al. (2010)
ELN*	% DW	44.05 ± 1.63	2.97% 8.6%	46.97%	Singh et al. (2022) Fazal & Singla (2012) Tolba, Youssef, & Abd-Elwahab (2008)
pH		6.88 ± 0.14			
Chlorophyll a	µg/g DW	3,883.77 ± 0.44	63.9–68.3 mg/100g	229.07 mg/100g	Rozek (2007) Tolba et al. (2008)
Chlorophyll b	µg/g DW	1,761.72 ± 0.33	32.4–33.1 mg/100g	78.46 mg/100g	Rozek (2007) Tolba et al. (2008)
Carotenoids	µg/g DW	889.19 ± 0.07		83.01 mg/100g	Rozek (2007) Tolba et al. (2008)
Polyphenols	mg Gallic acid/g DW	61.21 ± 1.09	17.2 ± 1.1		Golubkina et al. (2020)
Flavonoids	mg Quercetin/g DW	0.02 ± 0.01	6.6 ± 0.2 mg/g DW		Golubkina et al. (2020)
Ascorbic acid	mg/g DW	1.29 ± 0.07	62.6 mg/100 g	40.43 mg/g	Fazal & Singla (2012) Sarwar, Ayyub, Rezgui, Nisar, & Jilani (2019)
Antioxidant capacity					
ABTS	µmol Trolox Eq/g DW	22.40 ± 0.68	34.52 ± 3.25 µg/mL		Emad, Rasheed, El-Kased, & El-Kersh (2022)
DPPH	µmol Trolox/g DW	91.60 ± 0.79	930.8 ± 42.50 µg/mL		Emad et al. (2022)

FW: fresh weight; DW: dry weight; ELN: digestible carbohydrates or polysaccharides; mean ± standard deviation (n = 3).

to the plant's maturity. In the early growth stages, the pigment content, including carotenoids, tends to be higher due to the loss of structural water (Campos et al., 2019).

Antioxidant activity exhibits significant differences between the ABTS and DPPH methods, owing to the chemical reactivity of free radicals. Despite these differences, the DPPH method appears to be the most suitable for determining antioxidant activity in Chillangua, and the results obtained are comparable with those of Leitão et al. (2020) which was 14.77 μmol Trolox Eq/g for ABTS in Amazonian leaves *E. foetidum*.

The Chillangua has a higher level of chlorophylls, carotenoids, and total polyphenols, compared with celery and parsley, but it has a very low content of flavonoids and ascorbic acid.

3.2 Optimal encapsulation of Chillangua extract

With a confidence level greater than 0.05 (Table 3), the effects of atomization temperature, type of encapsulant, and encapsulant concentration on functional components and antioxidant activity were determined. The variables that showed linearity greater than 70% included total polyphenols (R^2 = 82.33), ABTS (R^2 = 71.58), chlorophyll a (R^2 = 76.65), and b (R^2 = 85.83), while variables such as carotenoids, vitamin C, moisture, water activity, flavonoids, DPPH, dissolution, H° parameter, L*, and b* showed no significance.

The mathematical models for the variables that show linearity are as follows, for maltodextrin (M), temperature (T), and Arabic gum (G) (Equations 2–7).

Polyphenols

$$\mathbf{Y}_1=17.45-0.19*T-0.65*M+0.07*G-0.00*T*G+0.02*M2+0.01*M*G+0.02*G2 \quad (2)$$

ABTS

$$\mathbf{Y}_2=-21.32+0.44*T-1.28*M+1.36*G+0.01*T*M-0.01*T*G-0.02*M2+0.01*G2 \quad (3)$$

Color L*

$$\mathbf{Y}_3=10.61+0.23*T+1.47*M+3.32*G+0.01*T*M-0.05*M2-\ 0.20*M*G-0.06*G2 \quad (4)$$

Color a*

$$\mathbf{Y}_4=5.98-0.08*T-0.09*M-0.08*G+0.26*T2-0.18*T*M+0.36*T*G+0.01*M2+0.01*G2 \quad (5)$$

Chlorophyll *a*

$$\mathbf{Y}_5=18.51-0.29*T+0.91*M+0.33*G-0.01*T*M-0.01*T*G+0.04*M2+0.05*G2 \quad (6)$$

Chlorophyll *b*

$$\mathbf{Y}_6=34.91-0.50*T+0.47*M+0.21*G-0.01*T*M-0.01*T*G+0.04*M2+0.02*M*G+0.05*G2 \quad (7)$$

Response surface analysis established that the combination of factors and temperature does not significantly affect the content of functional compounds and antioxidant activity, as the extract can tolerate temperatures between 160 and 210°C (García-Cárdenas et al., 2015). The type of encapsulant (mainly maltodextrin) directly influences the encapsulation of Chillangua extract.

The highest amount of total phenols, antioxidant activity, and chlorophylls a and b is obtained when using concentrations lower than 3% of maltodextrin and Arabic gum. On the contrary, at concentrations higher than 5%, an increase in luminosity is observed, and the greenish hue increases when using concentrations lower than 7% of encapsulants. However, darkening occurs when using concentrations greater than 10% of encapsulants.

Table 3. Data obtained for the Chillangua leaves.

t	Carote-noids	Chloro-phyll *a*	Chloro-phyll *b*	Diss	Poly-phenols	ABTS	DPPH	Flavo-noids	Vita-min C	Mois-ture	aw	L*	C*	H*	a*	b*	ED
1	0.24	0.83	0.54	36	1.28	10.05	3.08	0.01	0.72	4.80	0.12	48.10	8.67	94.53	−0.69	8.64	0.00
2	0.01	1.38	1.94	24	2.81	11.65	2.89	0.02	1.16	5.97	0.17	39.34	6.91	93.71	−0.46	6.89	0.49
3	0.51	3.61	3.18	38	2.97	12.80	5.36	0.02	1.22	7.53	0.30	40.83	6.36	93.06	−0.34	6.35	0.94*
4	0.41	2.76	2.46	22	1.65	8.54	4.82	0.01	1.46	6.07	0.19	52.44	6.91	93.99	−0.46	6.89	0.37
5	0.18	1.96	2.16	21	1.91	9.38	4.39	0.01	0.38	4.56	0.14	52.30	3.92	135.14	−0.60	2.33	0.41
6	0.28	2.25	2.19	22	2.57	13.24	4.58	0.01	0.30	7.62	0.23	51.80	6.65	91.46	−0.17	6.65	0.68
7	0.29	2.24	2.04	50	1.96	10.13	2.93	0.01	0.76	0.40	0.13	48.77	8.21	91.56	−0.20	8.20	0.46
8	0.12	2.95	3.64	19	1.73	8.13	2.63	0.01	1.21	5.81	0.21	51.46	7.06	94.45	−0.55	7.03	0.40
9	0.15	1.59	1.78	20	1.63	8.55	2.75	0.01	0.56	5.13	0.20	54.26	6.64	93.51	−0.39	6.62	0.26
10	0.16	1.78	2.03	20	1.80	8.38	3.81	0.01	0.51	7.61	0.29	50.18	8.17	91.19	−0.17	8.17	0.31
11	0.09	1.42	1.76	19	1.87	10.99	3.43	0.01	0.64	6.23	0.23	54.65	6.46	94.61	−0.51	6.44	0.37
12	0.38	2.61	2.25	17	2.80	12.33	4.56	0.02	0.81	8.57	0.31	46.48	5.40	90.16	−0.03	5.40	0.72
13	0.19	1.73	1.88	20	1.37	7.38	2.98	0.01	0.85	7.03	0.22	55.73	5.83	94.34	−0.44	5.82	0.00
14	0.34	2.47	2.20	11	2.08	11.90	5.27	0.01	0.70	5.43	0.19	47.53	6.45	93.79	−0.41	6.43	0.58
15	0.01	2.15	2.87	27	1.88	10.00	4.04	0.01	1.33	7.29	0.22	53.97	7.94	91.88	−0.25	7.92	0.49
16	0.16	0.94	0.91	24	1.76	11.32	4.04	0.01	1.51	5.12	0.16	54.41	4.29	96.82	−0.47	4.26	0.17
Valor-P																	
A	0.72	0.70	0.18	0.34	0.84	0.34	0.96	0.59	0.51	0.25	0.55	0.48	0.33	0.25	0.44	0.25	
B	0.3	0.24	0.95	0.27	0.00	0.00	0.25	0.02	0.88	0.76	0.29	0.00	0.59	0.18	0.03	0.43	
C	0.15	0.40	0.34	0.43	0.06	0.17	0.07	0.16	0.83	0.81	0.91	0.03	0.21	0.22	0.18	0.16	
AA	0.73	0.04	0.00	0.64	0.07	0.35	0.9	0.30	0.23	0.71	0.19	0.86	0.58	0.91	0.27	0.66	
AB	0.11	0.01	0.01	0.21	0.36	0.28	0.64	0.66	0.09	0.59	0.61	0.65	0.29	0.18	0.86	0.2	
AC	0.56	0.03	0.01	0.61	0.11	0.14	0.17	0.76	0.48	0.24	0.29	0.86	0.27	0.18	0.73	0.18	
BB	0.4	0.00	0.00	0.58	0.16	0.46	0.96	0.23	0.56	0.44	0.16	0.55	0.47	0.96	0.10	0.46	
BC	0.36	0.77	0.07	0.65	0.13	0.95	0.23	0.18	0.3	0.38	0.33	0.07	0.75	0.22	0.52	0.52	
CC	0.94	0.00	0.00	0.61	0.03	0.68	0.27	0.73	0.92	0.91	0.59	0.48	0.64	0.91	0.25	0.73	
R^2	63.66	76.65	86.83	50.93	82.33	71.58	68.48	33.98	59.25	47.70	57.69	58.81	58.08	67.35	29.63	65.46	

t: treatments; aw: water activity; ED: expected desirability; Diss: dissolution; temperature (A), concentration of maltodextrin (B), and Arabic gum (C).

3.3 Encapsulation optimization

The optimization of encapsulation is achieved by using 165.61°C, along with 2.90% maltodextrin and 1.06% Arabic gum. The ratio between these proportions is approximately 3:1, which aligns with Ortiz-Romero et al. (2021). However, Aragüez et al. (2018), Tolun et al. (2016), and García-Cárdenas et al. (2015) suggest that temperatures higher than 138, 140, and 213°C, respectively, could lead to the disintegration of phenolic compounds and oxidation-induced encapsulation.

The response surface of the optimization involving temperature (T), maltodextrin concentration (M), and Arabic gum (G) concentration is illustrated in Figure 1.

On the contrary, this study determined that treatment 3 produces the maximum desirability in terms of the content of functional compounds and antioxidant capacity when using 3% maltodextrin and 3% Arabic gum at a temperature of 160°C, as observed in Table 3.

3.4 Physical, chemical, functional, and antioxidant properties of Chillangua encapsulates

The results obtained in moisture (5.97%), dissolution time (24 s), water activity, and color highlight the stability of the powders influenced by the feed flow rate and the hydrophobicity of the encapsulates, as shown in Table 4. The moisture content of the powders is suitable compared with those obtained by Aragüez et al. (2018) which was 4.02–4.95% in orange juice, Ramakrishnan et al. (2018) which was 3.43–4.89% in tamarillo juice, and Rojas et al. (2022) which was 3.43–4.95% in aqueous lemon verbena extract. This appropriate moisture could be attributed to the incorporation of hydrocolloid encapsulants and an increase in total solids. This is supported by a water activity of 0.21 in the encapsulates, which falls within the range of dehydrated foods and prevents powder agglomeration (Paredes Nureña & Castillo Martinez, 2015). Then particle size influenced the dissolution time of the powders (Kuck & Noreña, 2016).

The color properties of the encapsulates show moderate brightness in relation to the overall scale. Additionally, the a* (-0.38 ± 0.26), b* (6.50 ± 1.95), L* (60.14 ± 5.18), C* (6.62 ± 1.58), and H° (95.89 ± 19.63) parameters suggest a tendency toward a green color very close to neutral and a leaning toward yellow.

The analysis of the encapsulates reveals the preservation of photosynthetic pigments such as chlorophyll a (102 µg/g BS) and chlorophyll b (105 µg/g BS) and the preservation of antioxidant properties, which have potential nutritional and functional benefits for health. These properties include carotenoids, total polyphenols, flavonoids, vitamin C, and the ability to neutralize free radicals.

Despite this preservation, there is a decrease in the concentration of compounds compared with lyophilized Chillangua, which is a phenomenon that can be attributed to the temperature used during the encapsulation process (Rojas et al., 2022). It is also considered that the presence of glycoside groups could have negative effects on the chemical structure, solubility, stability, as well as the extraction process (Raunelli et al., 2019).

3.5 Characterization of encapsulates by scanning electron microscopy

The morphology of the powders reflects an irregular surface, with no visible cracks and shallow dimples, as illustrated in Figure 2. The findings of Braga et al. (2020) in pineapple mint juice, Sarabandi et al. (2019) for the encapsulation of eggplant peel extract, and Kalušević et al. (2017) in soybean extract align with the results of this research, attributing these physical characteristics to the influence of drying temperature and rapid moisture removal. Additionally, the lack of uniformity in the encapsulates could be related to the drying chamber.

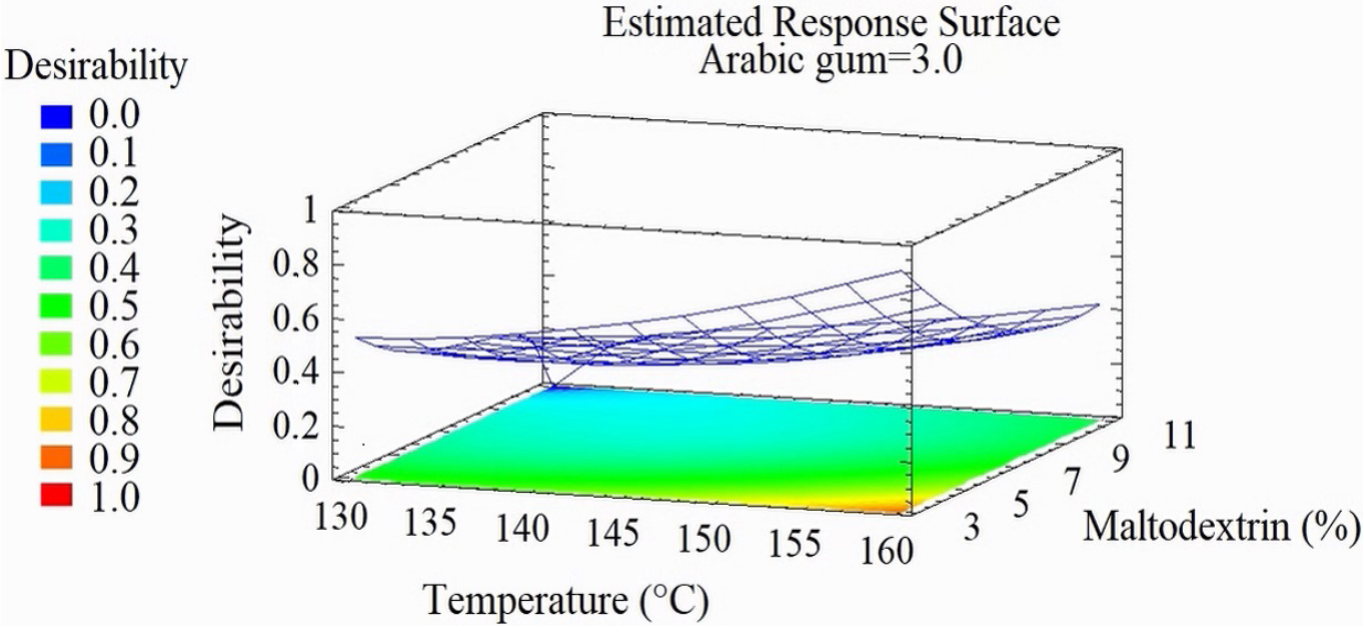

Figure 1. Response surface and contour plot of the effect between temperature and maltodextrin.

The morphology of Figure 2M obtained with 12.39% maltodextrin and 6.5% Arabic gum and at a temperature of 145°C exhibits more uniform surfaces compared with the rest of the treatments. Sarabandi et al. (2019) argued that using temperatures higher than 105°C results in more even structures. However, Figures 2A, 2M, 2N, 2O, and 2P, obtained at 145°C, and Figures 2E, 2G, and 2I, obtained at 160°C, do not show continuous and smooth surface.

At concentrations of 3% maltodextrin and 3% Arabic gum, as well as 6.5% maltodextrin and 0.5% Arabic gum (Figures 2B and 2C), the agglomeration of particles is more scattered. When a mixture of 10% maltodextrin and 3% Arabic gum is used (Figures 2D and 2E), a lower dispersion is observed compared with concentrations of 3% maltodextrin and 10% Arabic gum (Figures 2F and 2G).

The encapsulates with 10% maltodextrin and 10% Arabic gum (Figures 2H and 2J) show evidence of structure accumulation. On the contrary, the particle distribution with 0.6% maltodextrin and 6.5% Arabic gum is similar to that obtained when using 6.5% maltodextrin and 0.61% Arabic gum (Figures 2L and 3N). Finally, the dispersion of the encapsulates at concentrations of 12.39% maltodextrin and 6.5% Arabic gum (Figure 2M) exhibits a higher particle concentration compared with the use of 6.5% maltodextrin and 12.39% Arabic gum (Figure 2O).

Particle agglomerations may be attributed to the presence of moisture and an increase in maltodextrin amount. Additionally, as the amount of maltodextrin increases, the particle structures tend to become more compact and adhere to other particles (Plati et al., 2021).

As the average size distribution of Chillangua encapsulates is 4.63 ± 0.81 μm, which falls within the typical size range for microencapsulates (1–1,000 μm), it can be inferred that the process used can be classified as microencapsulation, in line with the observations of Sandoval-Peraza et al. (2017).

The elemental analysis of the encapsulates by EDS is illustrated in Table 5. According to the results obtained through EDS, the prominent elements in Chillangua encapsulates were oxygen (O), carbon (C), sodium (Na), potassium (K), zinc (Zn), calcium (Ca), chlorine (Cl), and traces of aluminum (Al). The presence or absence of these elements may be related to soil characteristics and cultivation methods, including the use of fertilizers (Rodrigues et al., 2022).

The characterization of the walls of the encapsulated materials in terms of their elemental content at a micrometric scale reveals that the percentage of mass obtained is higher for carbon and oxygen elements, possibly because they are structures of organic origin (organic matter). Additionally, maltodextrin presents C–O–C bonds in its structure, as does Arabic gum in its composition (Plati et al., 2021). It is also important to emphasize that the presence of carbon as the main element could be attributed to the carbon tape used as a grid in the equipment (Hodoroaba, 2020).

Table 4. Physical, chemical, and antioxidant properties of Chillangua encapsulates.

Parameter		Average
Moisture	%	5.97 ± 1.84
Water activity (aw)		0.21 ± 0.06
Dissolution	seg	24.40 ± 9.49
Chlorophyll a	μg/g DW	102.00 ± 0.73
Chlorophyll b	μg/g DW	105.5 ± 0.74
Carotenoids	μg/g DW	11.00 ± 0.14
Total phenols	mg gallic acid/g DW	2.00 ± 0.51
Flavonoids	Quercetin mg/g DW	0.01 ± 0.00
Vitamin C	mg/g DW	0.88 ± 0.39
Antioxidant activity ABTS	μmol Trolox Eq/g DW	10.30 ± 2.30
DPPH	μmol Trolox/g DW	3.85 ± 1.03

E: encapsulant; DW: dry weight; mean ± standard deviation (n = 3).

Table 5. Elemental composition and particle size of the encapsulated extracts of Chillangua.

	% Mass								Average particle size
	Carbon	**Oxygen**	**Sodium**	**Aluminum**	**Chlorine**	**Potassium**	**Calcium**	**Zinc**	**µm**
t1	64.14	45.84	0.63		0.22	1.96	1.02	0.57	5.79 ± 2.24
t2	87.83	52.03	0.85	0.25	0.50	4.14	1.14	0.53	3.85 ± 1.64
t3	133.28	84.77	1.13		0.51	4.06	1.50		3.42 ± 1.29
t4	98.88	61.06	0.53	0.54	0.28	2.05	0.56		5.91 ± 2.31
t5	95.11	59.47		0.90	0.31	2.29	0.70	0.66	4.75 ± 2.33
t6	130.19	75.65	1.67	0.18	0.24	2.02	1.39		3.67 ± 1.45.
t7	91.76	51.40	1.03			1.39	0.93		4.81 ± 2.29
t8	105.26	59.40	0.97		0.18	1.59	1.14	0.70	5.27 ± 2.38
t9	73.17	40.74	0.58			1.17	0.85	0.60	5.32 ± 2.59
t10	92.75	46.24	0.71			1.40	0.64		3.76 ± 1.68
t11	114.07	55.88	0.93		0.21	1.90			5.60 ± 2.08
t12	95.52	62.08	1.40	0.31	0.32	2.64	1.34	0.85	3.66 ± 1.84
t13	103.65	74.79	0.59		0.14	1.29	0.64		4.45 ± 2.03
t14	90.73	45.11			0.17	1.37			4.38 ± 1.54
t15	93.35	64.08	1.02		0.20	1.35	1.04		4.91 ± 1.91
t16	70.25	54.23				0.60	0.30	0.60	4.52 ± 2.09

t: treatments.

4 CONCLUSIONS

Chillangua exhibits a significant nutritional and antioxidant profile, characterized by low fat content but high levels of protein, fiber, ash, and components such as chlorophyll a, chlorophyll b, carotenoids, total polyphenols, flavonoids, and ascorbic acid. Furthermore, optimal encapsulation of Chillangua extract is achieved by mixing 2.90% maltodextrin and 1.06% Arabic gum at a temperature of 165°C. Encapsulation technology proved to be a successful strategy for stabilizing functional and antioxidant properties. Regarding their morphology, the encapsulates displayed a wrinkled surface with the presence of elements such as C, O, K, Zn, Cl, Al, Ca, and Na.

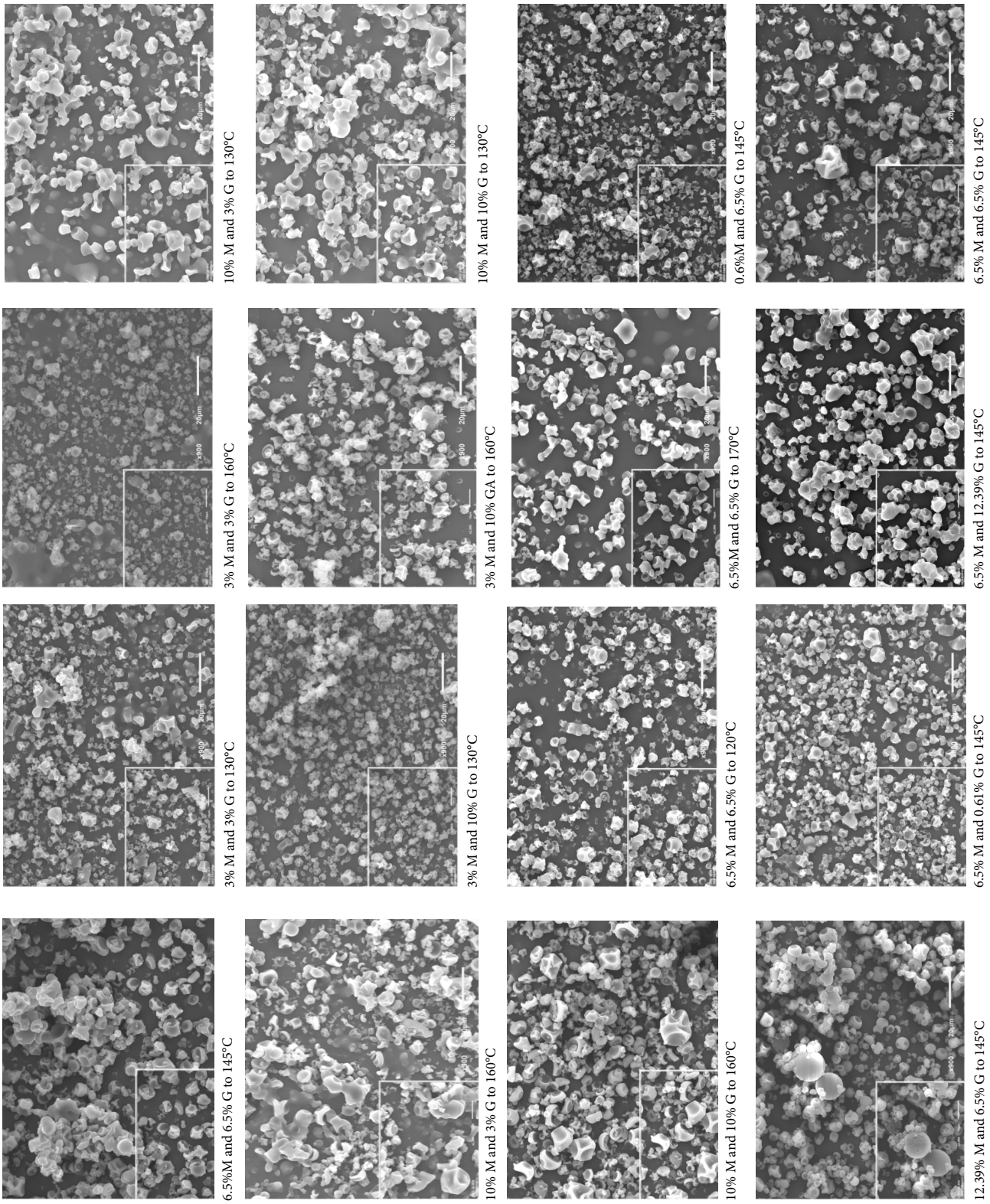

Figure 2. Morphology of the powders.

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