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Red pitaya (*Hylocereus polyrhizus*) as a source of betalains and phenolic compounds: Ultrasound extraction, microencapsulation, and evaluation of stability

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ABSTRACT

There is an increasing demand for natural pigments in food production, but challenges are related to the existence of matrices rich in these pigments, the optimization of extraction conditions and the preservation of pigments. Pitaya, the fruit of *Hylocereus polyrhizus* is considered a good source of betalains and phenolic compounds; therefore, they were extracted from its pulp by ultrasound-assisted extraction, the extract was encapsulated by spray drying, and the stability of the microencapsulated pitaya pulp extract (MPPE) was studied. MPPE showed satisfactory results of solubility, hygroscopicity, encapsulation efficiency and antioxidant activity. After 90 days of storage, MPPE showed reductions in betalains and phenolic compounds contents of only 14.28% and 11.88%, respectively, which indicates that the extract has great potential as an ingredient and/or functional pigment for application in the food and pharmaceutical industries.

1. Introduction

Caatinga is the only exclusively Brazilian biome, and its distribution is predominantly in the Northeast region. Being in an arid region, it produces fruits with high amounts of secondary bioactive compounds, which brings a high added value to the fruits and shows the importance of this biome for fruit production in the Northeast region of Brazil (Coradin et al., 2018; OECD-FAO, 2019).

Pitaya (*Hylocereus polyrhizus*) is an exotic and tropical fruit belonging to the Cactaceae family, whose high contents of betalains and polyphenols are responsible for its antioxidant capacity. These factors act as an excellent opportunity for its development as a functional food in the international fruit market, being grown in several countries, including the Northeast region of Brazil (Ibrahim et al., 2018).

Betalains have a variety of biological properties contributing to the prevention of degenerative diseases, some types of cancer, cardiovascular and gastrointestinal diseases, diabetes and obesity. When compared to anthocyanins, betalains are less susceptible to degradation, thus being pigments for potential application in food (Ibrahim et al., 2018). Phenolic compounds have high antioxidant capacity, being capable of capturing reactive oxygen species and protecting against oxidizing agents and lipid peroxidation catalysts (Kumar et al., 2019). It is estimated that the global market for natural pigments is expected to grow at a compound annual growth rate of 4.7% until 2032 (FMI, 2022), while that of phytonutrients is expected to grow at around 7.1% until 2025 (Mordor Intelligence Research & Advisory, 2023).

Sample preparation and choice of extraction techniques are crucial steps for the satisfactory isolation, identification and study of bioactive compounds from plant matrices (Kumar et al., 2021). Although conventional extraction methods are easy to reproduce, the use of toxic solvents, high temperatures and long times make them laborious, expensive and unenvironmentally unfriendly, besides affecting the final

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content of bioactive compounds (Bhagya Raj & Dash, 2020).

In this way, the development and application of alternative extraction methods become fundamental. Ultrasound-assisted extraction is a technique based on the principle of cavitation, which allows greater accessibility of the solvent to cell walls, resulting in greater process efficiency. This technology is considered promising and has been used in the extraction of bioactive compounds from fruits and vegetables to be applied in food products (Bhagya Raj & Dash, 2020; Meregalli et al., 2020; Kumar et al., 2021).

Both betalains and phenolic compounds are unstable when exposed to environmental and processing factors, such as changes in temperature, water activity, humidity, presence of light, oxygen and enzymes, thereby requiring techniques than can improve their stability. Spray drying microencapsulation has been used to improve nutrition, mask flavors and odors, prevent loss of volatiles, increase shelf life and prevent spoilage, in addition to providing benefits to consumers, including the possibility of reducing the use of fat, salt and additives (Ye et al., 2018).

Considering that synthetic pigments are associated with the emergence of food intolerance and allergies, this work aimed to extract betalains and phenolic compounds from red pitaya cultivated in the Brazilian Agreste of Pernambuco using ultrasound-assisted extraction, as a green method of extraction, and to evaluate the impact of different variables on the extraction of these compounds. We also sought to microencapsulate them and carry out a stability study during 90-day storage.

2. Material and methods

2.1. Chemicals

Ethanol, Folin-Ciocalteu phenol reagent, gallic acid ($C_7H_6O_5$), citric acid, sodium phosphate dibasic, sodium carbonate, acetic acid, methanol and metanol for HPLC, phosphoric acid, sodium hydroxide, sodium acetate trihydrate, glacial acetic acid p.a., hydrochloric acid, ferric chloride hexahydrate, ferrous sulfate heptahydrate and 2,4,6-tris(2-pyridyl)-*s*-triazine (TPTZ) were obtained from Sigma Aldrich (St. Louis, MO, USA). Standards, including catechin, chlorogenic acid were obtained from Sigma Aldrich as well. Epigallocatechin gallate, epicatechin gallate, procyanidin A2, rutin, quercetin were obtained from Extrasynthese (Genay, France), phenolphthalein and potassium biphthalate from Synth (São Paulo, SP, Brazil), and maltodextrin 10 DE from Ingredion (São Paulo, SP, Brazil). Ultrapure water was prepared using a Milli-Q system (Millipore, Bedford, MA, USA).

2.2. Material

Samples of red pitaya (*Hylocereus polyrhizus*) fruits were collected from an open field orchard located in Garanhuns, Agreste region of Pernambuco, Brazil (8° 59′ 8.582″ S 36° 24′ 31.496″ W), in an area of 1 ha. The fruits were collected at a point of maturity suitable for human consumption. The collection was carried out on January 2019. Samples were randomly collected from 100 plants, totaling 30 kg of fruits.

2.3. Preparation of lyophilized pulp samples

The fruits were washed in running water, sanitized in a 2.5% sodium hypochlorite solution for 15 min, and manually peeled and pulped (Bonina Compacta, Itabuna, BA, Brazil). The pulp was then placed in plastic Petri dishes and wrapped with plastic film (PVC) with small holes. The samples were frozen (−80 °C) and lyophilized in a lyophilizer (Alpha 1–4 LD Plus, Christ, Osterode am Harz, Germany) for 48 h under 20 mbar pressure. The lyophilized pulp was macerated, packaged in airtight amber glass jars, frozen, and stored at −22 °C for further analysis.

Table 1
Coded and decoded levels of the independent variables used in the 2^3-experimental design employed to investigate the ultrasound-assisted extraction of betalains and phenolic compounds from red pitaya pulp (*Hylocereus polyrhizus*).

Run	Ethanol Concentration (%)	Solvent/sample ratio (mL/g)	Ultrasound immersion time (min)
1	−1 (30)	−1 (40)	−1 (10)
2	+1 (70)	−1 (40)	−1 (10)
3	−1 (30)	+1 (60)	−1 (10)
4	+1 (70)	+1 (60)	−1 (10)
5	−1 (30)	−1 (40)	+1 (20)
6	+1 (70)	−1 (40)	+1 (20)
7	−1 (30)	+1 (60)	+1 (20)
8	+1 (70)	+1 (60)	+1 (20)
9	0 (50)	0 (50)	0 (15)
10	0 (50)	0 (50)	0 (15)
11	0 (50)	0 (50)	0 (15)

2.4. Ultrasound assisted extraction and conventional extraction

An ultrasonic probe (QR1000 Ultronique, Ecosonics, Indaiatuba, SP, Brazil) was used, to assist the extraction process, in an environment protected from light at 275 W and 20 kHz. Tests using ultrasounds were carried out according to a 2^3-experimental design consisting of 8 factorial points (levels ± 1) and 3 central points (level 0) (Table 1). The data obtained were fitted to Eq. (1):

$$Y = \beta_0 + \beta_1 C + \beta_2 T + \beta_3 M + \beta_4 CT + \beta_5 CM + \beta_6 TM \quad \text{Eq. (1)}$$

where Y is the response, β_0 is the constant regression coefficient, β_1, β_2 and β_3 are the linear coefficients, while ethanol concentration (C), solvent/sample ratio (T) and ultrasonic immersion time (M) are the independent variables and β_4, β_5, β_6 the interaction effect coefficients. The response variables were the contents of betalains and phenolic compounds.

After exposure to ultrasounds, the supernatant obtained was centrifuged at 2000 rpm (20 min), filtered on quantitative filter paper (C41 black band, Unifil São Paulo, Brazil), and stored in amber glass at −22 °C. The extract that provided the best results was subjected to a conventional extraction (maceration) in order to compare the results. Sample and solvent were kept under constant agitation with a magnetic stirrer, centrifuged (1187.2×*g*/20 min), filtered on filter paper, and stored in amber glass at −22 °C.

2.5. Microencapsulation by spray drying

Microencapsulation was performed in a spray dryer, model MSD 1.0 (Labmaq do Brasil, Ribeirão Preto, SP, Brazil). The commonly used maltodextrin 10 DE (Ingredion) (15%) was selected as the carrier agent because of its low cost and high efficiency as an encapsulating compound. The extract formulation and encapsulating agent were homogenized in a magnetic stirrer for 5 min. The conditions were selected according to Bakar et al. (2012) with some modifications. The inlet and outlet temperatures were 165 and 80 °C, respectively, the mixture feed flow rate was 0.60 L/h (injector nozzle of 1.2 mm in diameter), the airflow rate 30 m^3/h, and the air pressure 0.6 bar. The microencapsulated extract was packaged in laminated plastic flexible bags and stored at −22 °C for further analysis.

2.6. Betalains quantification

Betains were quantified according to Stintzing et al. (2005) in a spectrophotometer (UV–1650PC, Shimadzu, Kyoto, Japan), and the results were fitted to Eq. (2):

$$\text{Betalains } \left(\text{mg.g}^{-1}\right) = \frac{(A \times DF \times MW \times V \times 1000)}{\varepsilon \times l \times W} \quad \text{Eq. (2)}$$

where *A* is the absorbance, *DF* is the dilution factor, *MW* is the molecular weight of betacyanins (550 g/mol) and betaxanthins (339 g/mol), *V* is the volume of the pigment solution (L), ε is the molar absorptivity coefficient of betacyanins (60.000 L mol^{-1} cm^{-1}) and betaxanthins (48.000 L mol^{-1} cm^{-1}), *l* is the optical path of the cuvette (1 cm), and *W* is the dry weight of plant material (g). The total betalain content (TBC) was the sum of betaxanthins and betacyanins present in the samples.

2.7. Total phenolics quantification

Total phenolics were determined according to Wettasinghe and Shahidi (1999) using the same spectrophotometer mentioned above at 725 nm. For analysis, 0.5 mL of extract were added to test tubes, together with 8 mL of distilled water and 0.5 mL of Folin-Ciocalteau reagent. After 3 min, 1 mL of a sodium carbonate solution (35 g/100 mL) was added, and the reaction mixture was protected from light. After 60 min, absorbance readings were performed, and the results were expressed in mg/gallic acid equivalent (mg GAE.g^{-1}).

2.8. Antioxidant activity determination

To determine the antioxidant activity, a repetition was performed under the optimized extraction conditions using 5 g of sample. The ferric reducing antioxidant power (FRAP) assay was employed according to the methodology described by Benzie and Strain (1996). Three different dilutions of the extract were prepared in triplicate. A 90 μL aliquot of each dilution of the extract was transferred to test tubes in a light-protected environment. Then, 270 μL of distilled water were added, along with 2.7 mL of FRAP reagent. The samples were homogenized in a vortex and kept in a water bath at 37 °C for 30 min. Readings were performed on a UV–Vis spectrophotometer (1900i, Shimadzu) at 595 nm using the FRAP reagent as a blank. The absorbance experimental values were plotted versus dilution, and the resulting straight line equation was determined. The results were expressed in μmol Fe^{2+}/g.

2.9. Profile of phenolic compounds by HPLC-DAD

The phenolic compounds profile was determined according to Padilha et al. (2017) and Dutra et al. (2018) using an Agilent 1260 Infinity LC System liquid chromatograph (Agilent Technologies, Santa Clara, CA, USA) coupled to a diode array detector (DAD) (model G1315D). Samples were previously diluted in phase A (0.1 mol/L phosphoric acid solution, pH 2.0) and filtered through a 0.45 μm membrane (Millex Millipore, Barueri, SP, Brazil). The oven temperature was 35 °C and the injection volume of the sample 20 μL. The eluent a mixture of solvents A and B (metanol acidified with 0.5% phosphoric acid), and the eluent flow rate 0.8 mL min^{-1}. The gradient used in the separation was 0–5 min: 5% B, 5–14 min: 23% B, 14–30 min: 50% B, 30–33 min: 80% B.

2.10. Physicochemical characterization of microencapsulated pitaya pulp extract

Water activity was determined using a water activity meter (AQUA LAB - 4 TE, Decagon Devices, São Jose dos Campos, SP, Brazil) at 25 °C, while moisture content using an infrared balance (ID-50, Marte, São Paulo, SP, Brazil) at 105 °C for 30 min for dry samples and 45 min for wet samples. Titratable acidity was determined according to the methodology of AOAC (2016). The pH was measured with a pH-meter (TEC-3MP, Tecnal, Piracicaba, SP, Brazil) previously calibrated with buffer solutions at pH 7 and 4. Soluble solids were analyzed by direct reading in an automatic refractometer (r2i300 Reichert, New York, NY, USA) and expressed in °Brix. The color of samples was determined with a colorimeter (CR-400, Konica Minolta, Tokyo, Japan) using the CIELAB system. The real color of the analyzed samples was obtained according to Eq. (3):

$$C^* = \sqrt{(a^{*2}+b^{*2})} \quad \text{Eq. (3)}$$

where C* is the chroma or saturation, i.e., the distance from the lightness axis, a* is the red-to-green color intensity, and b* is the yellow-to-blue color intensity.

The hue-angle (H°) was calculated according to Eq. (4):

$$H^\circ = \tan^{-1}(b^*/a^*) \quad \text{Eq. (4)}$$

and the color difference (ΔE) according to Eq. (5):

$$\Delta E = [(L_{sample}^* - L_{standard}^*)^2 + (a_{sample}^* - a_{standard}^*)^2 + (b_{sample}^* - b_{standard}^*)^2]^{0.5} \quad \text{Eq. (5)}$$

where L* is the lightness.

2.11. Scanning electron microscopy of microencapsulated pitaya pulp extract

Small amounts of samples were placed on the surface of a double-sided tape fixed to stubs. The samples were then coated with a thin layer of gold and metalized in a metallizer (Denton Vacuum, Desk V, Moorestown, NJ, USA). A scanning electron microscope (Vega3 LMU, Tescan Brno, Czech Republic) was used for examination of samples at 995× magnification.

2.12. Physical characterization of microencapsulated pitaya pulp extract

Apparent (or bulk) density (ρ_{ap}) was determined as described by Barbosa-Cánovas and Juliano (2005) and Caparino et al. (2012). It was calculated according to Eq. (6), and the results were expressed in g/mL:

$$\rho_{ap} = m/v \quad \text{Eq. (6)}$$

where *m* is the sample mass (g) and *v* is the total volume occupied by the powder in the beaker (mL).

Absolute density (ρ_{abs}, g.mL^{-1}) was determined according to Caparino et al. (2012) at 25 °C in a pycnometer with a thermometer. Intragranular porosity (ε) was calculated according to Eq. (7):

$$\varepsilon = 1 - \rho_{ap}/\rho_{abs} \quad \text{Eq. (7)}$$

Solubility was determined according to Cano-Chauca et al. (2005), and hygroscopicity according to Cai and Corke (2000).

2.13. Encapsulation efficiency (EE)

The encapsulation efficiency (*EE*) was determined according to Saénz et al. (2009). For the total contents of phenolic compounds and betalains in the microencapsulated extract, 100 mg of microencapsules were dispersed in 1 mL of an ethanol: acetic acid: distilled water (50:8:42 v/v) solution. The mixture was vortexed for 1 min and filtered through a 0.45 μm microfilter. For the content of phenolic compounds and betalains on the surface of the microencapsulated extract, 100 mg of microencapsules were dispersed in 1 mL of an ethanol: methanol (1:1 v/v) solution, vortexed for 5 min, and filtered through a 0.45 μm microfilter. The contents of phenolics and betalains were determined according to Wettasinghe and Shahidi (1999) and Stintzing et al. (2005), respectively. The encapsulation efficiency of phenolics and betalains was determined according to Eq. (8):

$$EE\ (\%) = \frac{Total\ Compound\ Content - Compound\ Content\ on\ Capsule\ Surface}{Total\ Compound\ Content} \times 100 \quad \text{Eq. (8)}$$

Table 2
Matrix of the 2^3-experimental design and ANOVA results for ultrasound-assisted extraction of betalains and phenolic compounds from pitaya pulp (*Hylocereus polyrhizus*).

Run	TBC (mg.g^{-1})	TPC (mg GAE.g^{-1})
1	2.02 ± 0.00 ^{a}	10.94 ± 0.06 ^{h}
2	1.26 ± 0.00 ^{e}	11.99 ± 0.06 ^{g}
3	1.79 ± 0.00 ^{c}	14.14 ± 0.02 ^{e}
4	1.23 ± 0.00 ^{f}	13.46 ± 0.01 ^{f}
5	1.66 ± 0.00 ^{d}	16.14 ± 0.01 ^{a}
6	1.06 ± 0.00 ^{gh}	14.52 ± 0.05 ^{d}
7	1.84 ± 0.01 ^{b}	16.25 ± 0.04 ^{a}
8	0.91 ± 0.00 ^{j}	15.35 ± 0.05 ^{b}
9	1.04 ± 0.00 ^{i}	15.07 ± 0.08 ^{c}
10	1.06 ± 0.00 ^{h}	15.27 ± 0.08 ^{b}
11	1.07 ± 0.00 ^{g}	15.05 ± 0.02 ^{c}
ANOVA		
R^2	0.7324	0.8881
Calculated F value	1.82	5.29
Critical F value (5%)	4.53	4.53

TBC = total betalains content, TPC = total phenolic content. Different lowercase letters show significant difference between columns. Data are presented as mean ± standard deviation (n = 3).

2.14. Encapsulation yield (Y)

The encapsulation yield (*Y*) was evaluated by gravimetry considering the relationship between the total mass of microencapsulated extract and the mass of solution (extract + wall material) that was fed to the system, based on the dry matter content (Fazaeli et al., 2012), according to Eq. (9):

$$Y\ (\%) = \frac{Total\ mass\ of\ microencapsulated\ extract\ (g)}{Mass\ of\ the\ solution\ (g)} \times 100 \qquad \text{Eq. (9)}$$

2.15. Encapsulation retention (R)

The encapsulation retention (*R*) was defined according to Eq. (10), not considering the carrier agent:

$$R\ (\%) = \frac{Content\ of\ microencapsulated\ compounds}{Content\ of\ compounds\ present\ in\ the\ feed\ solution} \times 100 \qquad \text{Eq. (10)}$$

2.16. Evaluation of microencapsulated pitaya pulp extract stability during storage

The microencapsulated pitaya pulp extract samples were packed in laminated flexible packaging and stored for 90 days in a light-protected environment. The total phenolic content (TPC), total betalain content, color, moisture, water activity and pH were evaluated at 0, 3, 8, 15, 30, 45, 60 and 90 days at 25 ± 1 °C. Such a temperature was selected to mimic commercial storage conditions.

2.17. Data analysis

The analysis of variance (ANOVA), the lack of fit test (F test), the Tukey's test, and the determination of the regression coefficients were performed with the software Statistica 7.0 (StatSoft, Tulsa, OK, USA) at a confidence interval of 95%. All determinations were carried out in triplicate.

3. Results and discussion

3.1. Experimental design and response surface of ultrasound-assisted extraction (UAE)

The pitaya pulp extract obtained by UAE exhibited TBC values ranging from 0.91 mg g^{-1} to 2.02 mg g^{-1} and TPC values ranging from 10.94 mg GAE.g^{-1} to 16.25 mg GAE.g^{-1} (Table 2). The low probability value obtained by the F test for TPC showed statistical significance of the regression model, while the model was not fitted to TBC data. The quality of the model and its general predictive capacity were additionally evaluated through the coefficient of determination (R^2). It can be seen in Fig. 1 A that 88.81% of the TPC variation was related to the time of immersion in ultrasound, which means that only 11.19% of total variations were not explained by the model. On the other hand, the R^2 value for TBC (0.7324) indicated that 73.24% of variation was related to ethanol concentration (Fig. 1 B), but no less than 26.76% of total variations were not explained by the model.

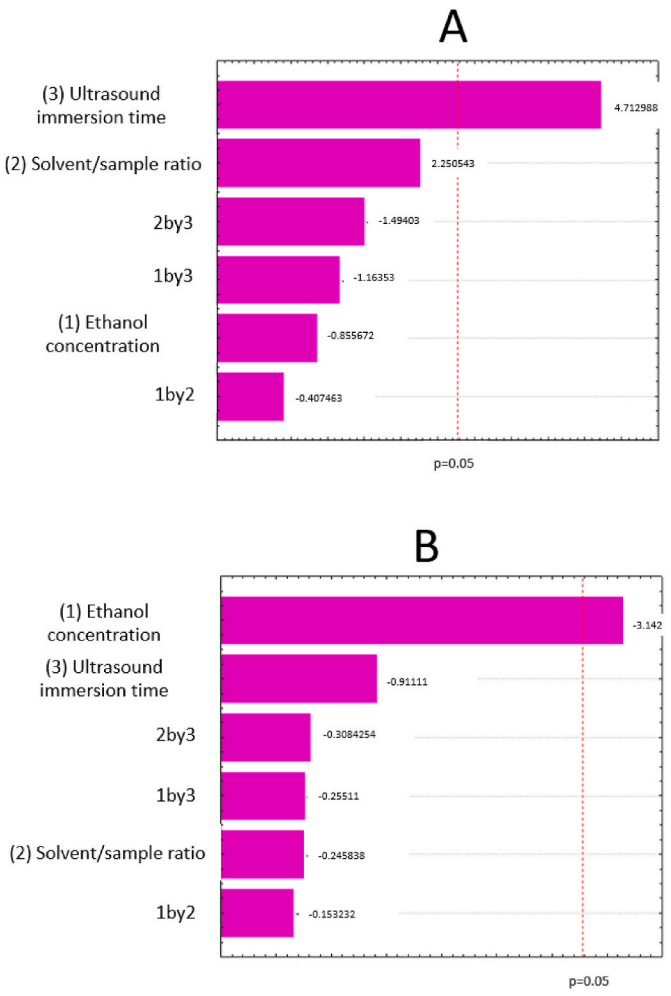

Fig. 1. Pareto diagram ($p < 0.05$) for phenolic compounds (A) and betalains (B) in pitaya (*Hylocereus polyrhizus*) pulp extracts obtained by ultrasound-assisted extraction. Variables that cross the dotted line are significant ($p < 0.05$).

Equation $Y = 14.38 + 1.46\ M$ allowed to describe the relationship between independent and dependent variables of the experimental design for TPC (Eq. (1)) using only a significant variable. The current model for TPC presented an R^2 value of 0.8881 and a F calculated from the regression of 5.29, a value higher than the critical F value (4.53) (Table 2), which suggests that the regression model was predictive and capable of representing the actual correlation between independent and dependent variables.

The positive sign of the effect of the ultrasound immersion time as independent variable (Y = 14,38 + 1,46 M) indicates that an improvement of the extraction ($p < 0.05$) of phenolic compounds is expected from an increase in the ultrasound immersion time. During extraction, the combined effect of cavitation and heat of the material matrix

Table 3
Physicochemical characteristics of pitaya pulp extract (*Hylocereus polyrhizus*) obtained by ultrasound-assisted (PPEU) and conventional extraction (PPEC).

	PPEU	PPEC
a_w	0.99 ± 0.00 [a]	0.99 ± 0.00 [a]
Moisture (g.100g^{-1})	94.86 ± 0.25 [b]	96.95 ± 0.27 [a]
pH	4.71 ± 0.00 [a]	4.33 ± 0.00 [b]
Titratable acidity (g.100g^{-1} malic acid) rowhead	0.12 ± 0.00 [a]	0.11 ± 0.03 [a]
Soluble solids (° Brix)	9.86 ± 0.05 [a]	8.50 ± 0.00 [b]
Color (CIELAB)		
L*	21.60 ± 0.11 [b]	30.75 ± 0.11 [a]
a*	16.64 ± 0.33 [b]	57.13 ± 0.21 [a]
b*	7.41 ± 0.12 [a]	−3.46 ± 0.28 [b]
C*	18.22 ± 0.35 [b]	57.24 ± 0.21 [a]
H°	24.01 ± 0.07 [a]	3.47 ± 0.28 [b]

a_w = water activity, L* = lightness, a* = red/green coordinate, b* = yellow/blue coordinate, C* = chroma, H° = hue angle. Different lowercase letters show significant difference ($p < 0.05$) between lines. Data are presented as mean ± standard deviation (n = 3).

enlarges the pores of cell wall, hence improving the contact between solute and solvent and increasing the mass transfer rate and the extraction efficiency until the equilibrium state is reached (Salar et al., 2016). In addition, an increase in the extraction time allows longer contact between the cell wall of the material and the surrounding solvent, enhancing the diffusion of phenolic compounds from the cells to the fluid medium and vice-versa (Al-Dhabi et al., 2017; Marzuki, Hamid, & Wahab, 2018; Bhagya Raj; Dash, 2020). In the present study, an ultrasound immersion time of only 20 min was sufficient to extract the highest levels of phenolic compounds, namely 16.14 mg GAE.g^{-1} (run 5) and 16.25 mg GAE.g^{-1} (run 7 of the experimental design). However, since these values were not statistically different ($p > 0.05$), the former extraction conditions using the lowest concentration of the extracting agent, i.e., 30% ethanol concentration, 40 mL/g solvent/sample ratio, and 20 min ultrasonic immersion time, were selected as the most promising from an economic point of view.

3.2. Physicochemical characterization and quantification of betalains and phenolic compounds in the selected pitaya extract

After the best extraction conditions were defined, the pitaya pulp extract obtained by ultrasound-assisted extraction (PPEU) was replicated using conventional extraction by maceration (PPEC) under the same conditions for comparison purposes. Water activity and titratable acidity did not show any significant differences ($p > 0.05$) between PPEU and PPEC (Table 3). The moisture content in PPEU was significantly lower ($p < 0.05$) and, conversely, that of soluble solids significantly higher ($p < 0.05$) than in PPEC likely due to the increase in temperature and consequent evaporation of both ethanol and water during processing (Caldas et al., 2018). Moreover, since temperature exerts a direct influence on the pH of substances, acting on the breakdown of hydrogen bonds, PPEU exhibited a significantly higher pH value ($p < 0.05$) when compared to PPEC (Table 3).

The lightness coordinate (L*), the red-green coordinate (a*) and the chroma (C*) in PPEC were significantly higher ($p < 0.05$), while the hue angle (H°) significantly lower ($p < 0.05$), than in PPEU, which means that, although both extracts were red, the latter had a darker and less saturated color than the former. These results are directly related to the increased extraction of betalains using ultrasound-assisted extraction due to the phenomenon of cavitation (Kumar; Srivastav; Sharanagat, 2021), as well as ethanol evaporation during PPEU preparation

Table 4
Contents of betalains and phenolic compounds in pitaya (*Hylocereus polyrhizus*) pulp extracts obtained by ultrasound-assisted (PPEU) and conventional extraction (PPEC).

	PPEU	PPEC
Betacyanins (mg.g^{-1})	1.10 ± 0.00 [a]	0.92 ± 0.00 [b]
Betaxanthins (mg.g^{-1})	0.56 ± 0.00 [a]	0.16 ± 0.00 [b]
Betalains (mg.g^{-1})	1.66 ± 0.00 [a]	1.08 ± 0.01 [b]
Total phenolic compounds (mg GAE.g^{-1})	16.14 ± 0.01 [a]	8.60 ± 0.02 [b]

Different lowercase letters show significant difference ($p < 0.05$) between lines. Data are presented as mean ± standard deviation (n = 3).

Table 5
Physicochemical and physical characteristics of microencapsulated pitaya (*Hylocereus polyrhizus*) pulp extract.

Physicochemical characteristics	
a_w	0.15 ± 0.00
Moisture (g.100g^{-1})	4.15 ± 0.14
pH	4.37 ± 0.01
Titratable acidity (g.100g^{-1} malic acid)	0.55 ± 0.03
Color (CIELAB)	
L*	77.17 ± 0.09
a*	29.20 ± 0.22
b*	−9.36 ± 0.02
C*	30.67 ± 0.21
H*	17.77 ± 0.12
Physical characteristics	
Apparent density (g.mL^{-1})	0.46 ± 0.01
Absolute density (g.mL^{-1})	1.06 ± 0.00
Intragranular porosity (ε)	0.50 ± 0.01
Solubility (%)	98.47 ± 0.07
Hygroscopicity (g.100g^{-1})	13.62 ± 0.14

a_w = water activity, L* = lightness, a* = red/green coordinate, b* = yellow/blue coordinate, C* = chroma, H° = hue angle. Data are presented as mean ± standard deviation (n = 3).

(Table 3).

As expected, PPEU showed significantly higher levels of betalains and phenolic compounds ($p < 0.05$) than PPEC (Table 4), in accordance with the results of phenolic compounds extraction reported by Caldas et al. (2018), Rocchetti et al. (2019), and Meregalli et al. (2020). In particular, the phenolic compounds content of the extract obtained by UAE (16.14 mg GAE.g^{-1}) was almost twice as high as that prepared through conventional extraction. These results confirm that emerging extraction techniques such as UAE show better responses than conventional techniques based on maceration stirring, and/or heating.

3.3. Physicochemical and physical characterization of microencapsulated pitaya pulp extract (MPPE)

The microencapsulated pitaya pulp extract (MPPE) showed a water activity (0.15) lower than that found by Shaaruddin et al. (2017) for pitaya powder and well lower than the threshold value (0.60) recommended to avoid the development of contaminating microorganisms (Leong et al., 2011). Although the moisture content of MPPE (4.5 g.100 g^{-1}) was higher (Table 5) than those found by other authors for microencapsulated extracts of pitaya peel and pulp (0.29 g.100 g^{-1} to 0.32 g.100 g^{-1}) (Bakar et al., 2013; Shaaruddin et al., 2017), it was lower than the maximum threshold values recommended by international legislation for whole milk powder, partly skimmed milk powder and skimmed milk powder (5 g.100 g^{-1}), as well as for cocoa powder (7.0 g.100 g^{-1}) (Codex Alimentarius, 1981; 1999). The pH (4.37) remained in the acidic range (Table 5), indicating microbiological stability. The relatively high titratable acidity of MPPE (0.55 g.100 g^{-1} malic acid) can be ascribed to the concentration of organic acids, since it

Fig. 2. Microencapsulated pitaya (*Hylocereus polyrhizus*) pulp extract.

is a dry substance.

MPPE showed significantly higher lightness (L* = 77.17) and chroma (C* = 30.67) but lower hue angle (H° = 17.77) (Table 5) compared to the non-microencapsulated extract prepared by UAE. It is likely that maltodextrin, used as the encapsulating agent, due to its white color, can have acted as a diluent for pigments such as betalains in the powdered product (Ferrari; Ribeiro & Aguirre, 2012), hence resulting in a pinkish color (Fig. 2).

Both apparent and absolute densities are important properties of dehydrated substances. The apparent density of MPPE was 0.46 g mL^{-1}, while the absolute density was 1.06 g mL^{-1} (Table 5). Li et al. (2018) and Lourenço, Moldão, & Alves (2020) found apparent density values of 0.5 g mL^{-1} for plum extract, and values between 0.18 and 0.30 g mL^{-1} for pineapple peel, respectively, both microencapsulated by spray drying. Lower apparent density values result in poorer packing due to the larger volume occupied per unit mass; therefore, there is an increase in the risk of product oxidation due to a higher amount of air within the powder, thus reducing storage stability (Lourenço et al., 2020). MPPE showed an intragranular porosity of 0.50. According to Zotarelli et al. (2017), highly porous, low density dehydrated products have interstices among particles hosting oxygen molecules, which can cause the degradation of bioactive compounds through oxidation reactions.

MPPE showed values of solubility (98.47% ± 0.07) and hygroscopicity (13.62 ± 0.14 g.100 g^{-1}) very close to those reported by Shaaruddin et al. (2017) for red pitaya pulp extract (96.12% and 14.12 g.100 g^{-1}, respectively). Such high solubility values are probably due to the use of a highly soluble carrier agent like maltodextrin 10 DE, even though the process variables were different. According to Jafari et al. (2017), the solubility of microencapsulated substances can be influenced by several factors such as raw material, carrier agents, and conditions used during microencapsulation, mainly airflow rate, temperature, and feed flow rate. On the other hand, low hygroscopicity values like those observed in both studies are considered able to ensure the physical, chemical and microbiological stability of powdered foods (Schuck & Ouest, 2011).

3.4. Scanning electron microscopy

Fig. 3 A shows that the microencapsulated pitaya pulp extract had particles with a tendency to agglomerate. This factor can be ascribed to the formation of bridges among particles due to the presence of available moisture or the absorption of moisture from the environment. The literature points out that agglomeration of smaller particles around larger particles can occur during the process of microencapsulation by spray drying, which can provide greater stability to microencapsulated compounds, as the outer particles can protect the internal ones (Alves et al., 2017). It is also possible to observe that most of the particles exhibited a spherical shape, with small wrinkles and depressions on the surface, which can be related, in general, to their shrinkage due to the drastic loss of moisture followed by cooling (Nizori et al., 2018).

3.5. Spray drying performance, quantification of bioactive compounds and antioxidant activity of microencapsulated pitaya pulp extract

The encapsulation efficiency (*EE*) of betalains was particularly high (94.94%), in agreement with the results reported by Vargas-Campos et al. (2018) and Soto-Castro et al. (2019) for betalain-rich microencapsulated cactus fruit extracts. This value is higher than those found by Fathordoobady et al. (2021) for encapsulation of betacyanins from *Hylocereus polyrhizus* by ionic gelation. The phenolic compounds also showed an *EE* value (60.75%) within the range (60–90%) considered relevant in microencapsulation by spray drying. As is known, the higher the encapsulation efficiency (*EE*), the greater the ability to keep a

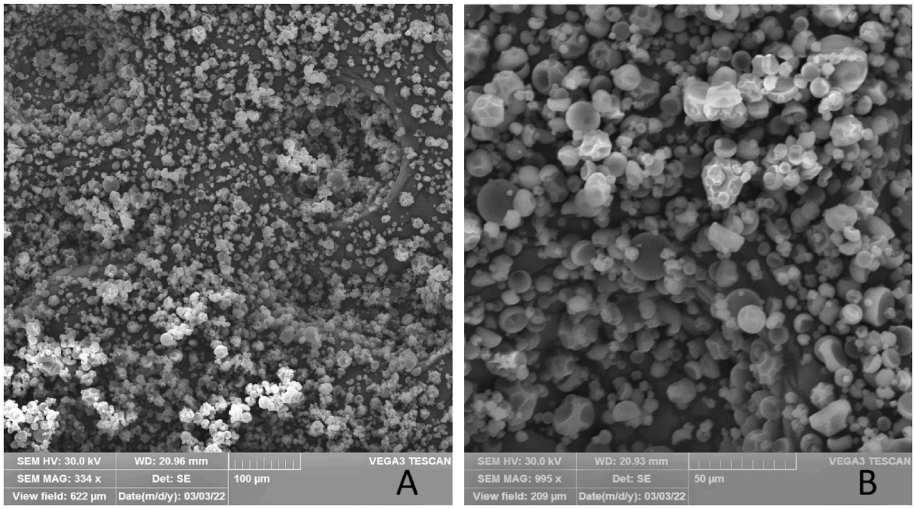

Fig. 3. Scanning electron microscopy images of microencapsulated pitaya (*Hylocereus polyrhizus*) pulp extract.

Table 6
Quantification of bioactive compounds in microencapsulated pitaya (*Hylocereus polyrhizus*) pulp extract, retention (%) and microencapsulation yield (%).

Bioactive compounds	
Betacyanins (mg.g^{-1})	0.50 ± 0.01
Betaxanthines (mg.g^{-1})	0.05 ± 0.00
Betalains (mg.g^{-1})	0.55 ± 0.01
Total phenolic compounds (mg GAE.g^{-1})	2.02 ± 0.01
Retention (%)	
Betacyanins	45.90 ± 1.07
Betaxanthines	9.74 ± 0.14
Betalains	33.63 ± 0.66
Phenolic compounds	12.54 ± 0.11
Yield (%)	17.21 ± 0.11

Data are presented as mean ± standard deviation (n = 3).

compound within the particles, and factors such as drying conditions and food matrix are crucial for obtaining a microencapsulated product with high *EE*. The wall material used is also important, as it allows bioactive compounds-polymer interaction due to electrostatic interactions and hydrogen bonds (Vergara et al., 2014). Therefore, the reductions of betalains and phenolic compounds contents after pitaya pulp extract microencapsulation can be attributed to operational aspects, such as high drying temperature that can degrade the thermosensitive compounds present in the extract as well as the type and amount of wall material used. Each wall material has in fact different characteristics that will or will not allow efficient retention of bioactive compounds (Soto-Castro, 2019). MPPE showed lower retention of betaxanthines (9.74%), when compared to betacyanins (45.90%), betalains (33.63%), and phenolic compounds (12.54%) (Table 6). Soto-Castro et al. (2019), in a study carried out on the microencapsulation by spray drying of *Escontria chiotilla* and *Stenocereus queretaroensis* extracts, ascribed the low retention of betaxanthines to the low heat stability of this class of compounds. It has been suggested that the use of higher concentrations of maltodextrin in the feed solution or combinations of different wall materials may allow greater protection of betalains and phenolic compounds against external agents and then may increase their retention (Lourenço et al., 2020; Moser et al., 2017); however, further studies are necessary so that other characteristics are not changed.

MPPE showed a lower encapsulation yield (*Y*) (Table 6) than that found in the literature (64.9%) for cactus fruit (Carmona et al., 2021). In matrices with high levels of sugar in their composition, such as pitaya, droplets collide in the drying chamber, causing deposits on the drying surface and thus reducing the yield (Cai & Corke, 2000).

3.6. Antioxidant activity of pitaya pulp extract obtained by ultrasound-assisted extraction and microencapsulated pitaya pulp extract

The pitaya pulp extract obtained by ultrasound-assisted extraction in the present study showed an antioxidant activity determined by the FRAP method (1531.56 μmol $Fe^{+2}.g^{-1}$) higher than those found in other studies carried out with pitaya. For instance, Fu et al. (2011) and Ramli et al. (2014) reported for pitaya extract obtained by conventional extraction antioxidant activities of 1.24 μmol $Fe^{+2}.g^{-1}$ and 609.17 μmol $Fe^{+2}.g^{-1}$, respectively, while Ramli et al. (2014) and Bellucci et al. (2021) reported antioxidant activities of 620.00 μmol $Fe^{+2}.g^{-1}$ and 8.25 μmol $Fe^{+2}.g^{-1}$ for extracts produced by ultrasound-assisted extraction and pulsed electric field extraction, respectively. The observed differences may be related to the pitaya varieties used, country of origin, planting, harvesting, edaphoclimatic conditions and extraction methods.

Even though the FRAP antioxidant activity of MPPE (473.11 μmol $Fe^{+2}.g^{-1}$) was less than one third of that of PPEU, it was higher than those reported for spray dried extracts of pineapple peel (Lourenço et al., 2020), grape peel (Carpes et al., 2020), and acerola pulp and residue (Rezende; Nogueira & Narain, 2018).

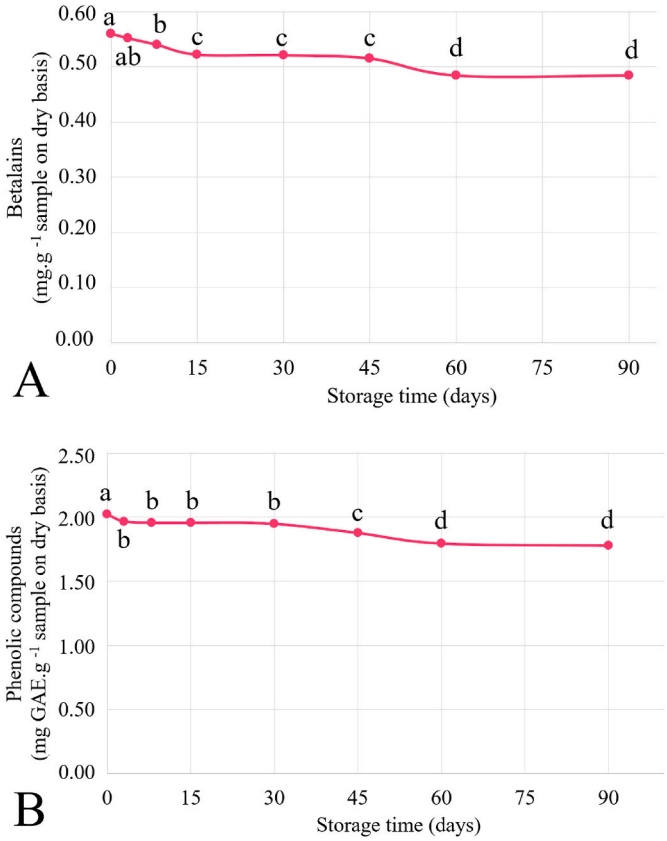

Fig. 4. Total contents of betalains (mg.g^{-1}) (A) and phenolic compounds (mg GAE.g $^{-1}$) (B) in microencapsulated pitaya (*Hylocereus polyrhizus*) pulp extract stored for 90 days at 25 °C. Different lowercase letters show significant ($p < 0.05$) differences between days. Data are presented as mean ± standard deviation (n = 3).

3.7. Microencapsulated pitaya pulp extract stability during storage

There were significant reductions ($p < 0.05$) in the contents of total betalains (14.28%) (Fig. 4 A) and total phenolic compounds (11.88%) in MPPE (Fig. 4 B) between the start and the end of the 90-day storage period, although the total content of betalains remained almost stable ($p > 0.05$) between 15 and 45 days of storage and that of phenolic compounds between 3 and 30 days and between 60 and 90 days of storage ($p > 0.05$).

Otálora et al. (2018), who observed a decrease in betaxanthins contents in a spray drying-microencapsulated extract of *Opuntia megacantha*, argued that during storage betaxanthins were more susceptible to degradation in an environment with high relative humidity. Soto-Castro et al. (2019) also observed significant losses of betalains, betacyanins and betaxanthin during storage of the spray dried extracts of *S. queretaroensis* and *E. chiotilla*, while Fathordoobady et al. (2021) observed a significant loss of betacyanins from *H. polyrhizus* after 60 days in alginate microspheres.

A similar decrease in phenolic compounds was also observed during storage of grape juice (Moser et al., 2017) and pineapple (Lourenço et al., 2020) extracts, both microencapsulated by spray drying. Such a reduction may have been the result of an increase in the moisture content of the dried extract, considering that moisture can act as a plasticizer and induce physicochemical changes in dry products, such as oxidation reactions. Moreover, other factors such as the availability of light and oxygen, the compound chemical structure, the type of wall

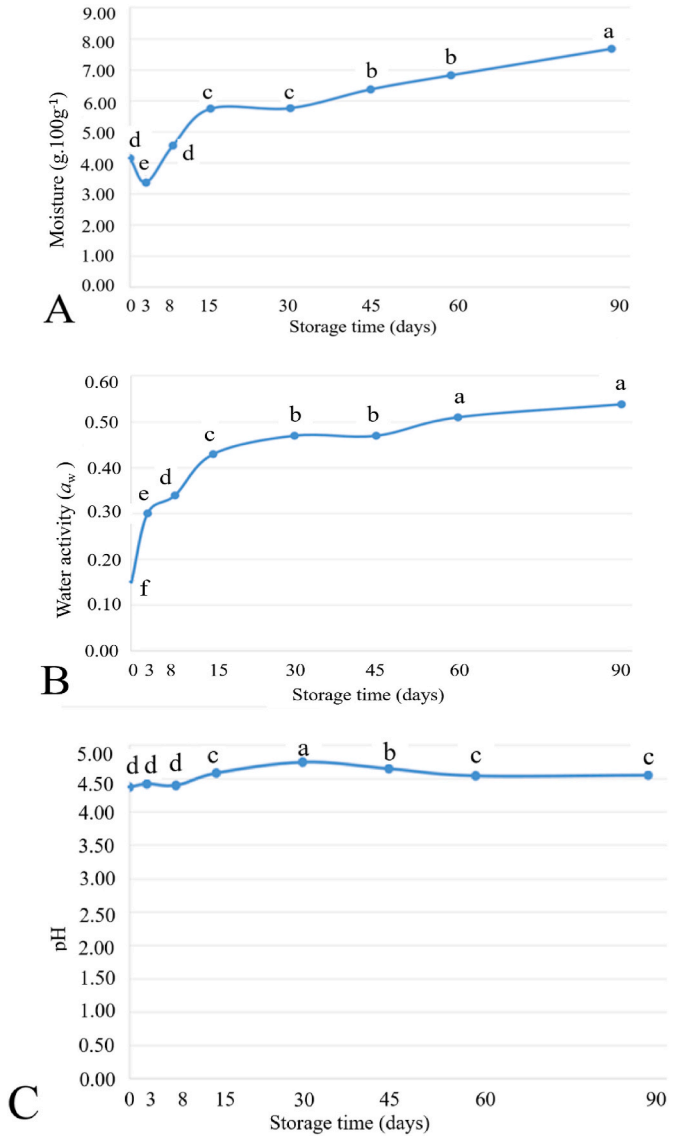

Fig. 5. Moisture (A), water activity (B), and pH (C) of microencapsulated pitaya (*Hylocereus polyrhizus*) pulp extract stored for 90 days at 25 °C. Different lowercase letters show significant ($p < 0.05$) differences between days. Data are presented as mean ± standard deviation (n = 3).

material, and even the encapsulation conditions may have played a significant role (Lourenço et al., 2020; Moser et al., 2017). Nonetheless, despite the statistical significance of reductions in the contents of betalains and phenolic compounds observed during storage, they were rather low, and MPPE still contained a considerable amount of these compounds, which indicates a great potential for its use as a functional ingredient and/or natural pigment in the food industry.

Consistently with the above hypothesis, moisture (Fig. 5 A) and water activity (a_w) (Fig. 5 B) of MPPE significantly increased ($p < 0.05$) from the start to the end of storage, but all the values kept below those established by international regulations (below 20 g.100 g^{-1}) (Codex Alimentarius, 1981; 1999). Moreover, MPPE a_w showed stable ($p > 0.05$) between 30 and 45 days of storage and between 60 and 90 days. Soto-Castro et al. (2019) observed a similar increase in the moisture content of dry extracts from *E. chiotilla* and *S. queretaroensis* fruits over 90 days of storage at 23 °C. It has been suggested that the absorption of water and the consequent increase in a_w of the microencapsulated extract can increase the barrier plasticity of the wall material and allow for a decrease in the content of phenolic compounds (Cai; Corke, 2000).

Table 7
Color results of microencapsulated pitaya (*Hylocereus polyrhizus*) pulp extract stored for 90 days at 25 °C.

Day	L*	a*	b*	C*	H°	ΔE
0	77.17 ± 0.09^{ac}	29.20 ± 0.22^{c}	−9.36 ± 0.02^{f}	30.67 ± 0.21^{c}	17.77 ± 0.12^{b}	–
3	78.25 ± 0.06^{a}	27.81 ± 0.23^{d}	−8.86 ± 0.03^{d}	29.19 ± 0.22^{e}	17.67 ± 0.10^{b}	1.83 ± 0.44
8	75.18 ± 0.19^{d}	30.12 ± 0.14^{b}	−9.24 ± 0.01^{e}	31.51 ± 0.14^{b}	17.05 ± 0.06^{c}	2.20 ± 0.15
15	77.68 ± 0.02^{a}	29.11 ± 0.08^{c}	−8.90 ± 0.02^{d}	30.44 ± 0.08^{c}	17.00 ± 0.01^{c}	0.72 ± 0.11
30	78.50 ± 0.06^{a}	27.03 ± 0.09^{e}	−9.31 ± 0.04^{ef}	28.59 ± 0.10^{f}	19.01 ± 0.04^{a}	2.54 ± 0.32
45	77.29 ± 0.32^{a}	28.8 ± 0.31^{c}	−7.95 ± 0.04^{b}	29.87 ± 0.31^{d}	15.43 ± 0.08^{d}	1.50 ± 0.14
60	78.14 ± 0.02^{ab}	28.14 ± 0.1^{d}	−7.69 ± 0.03 ^{a}	29.17 ± 0.09^{e}	15.29 ± 0.09^{de}	2.22 ± 0.18
90	75.42 ± 0.18^{bcd}	31.12 ± 0.21^{a}	−8.39 ± 0.00^{c}	32.23 ± 0.21^{a}	15.09 ± 0.09^{e}	2.78 ± 0.45

L* = lightness, a* = red/green coordinate, b* = yellow/blue coordinate, C* = chroma, H° = hue angle, ΔE = color difference. Different lowercase letters show a significant difference ($p < 0.05$) between days. Data are presented as mean ± standard deviation (n = 3).

Table 8
Phenolics profile of microencapsulated pitaya (*Hylocereus polyrhizus*) pulp extract (mg.L^{-1}).

Phenolic compounds	
Phenolic acids	mg.L^{-1}
Chlorogenic acid	1.79 ± 0.89
Total	1.79
Flavonols	
Catechin	1.35 ± 0.67
Epigallocatechin gallate	1.31 ± 0,65
Epicatechin gallate	1.17 ± 0.59
Procyanidin A2	1.82 ± 0.91
Rutin	1.96 ± 0.98
Quercetin	1.49 ± 0.74
Total	9.10

The results were expressed in mg.L^{-1}. Data are presented as mean ± standard deviation (n = 2).

However, a_w of MPPE stayed, during the whole storage period, below the maximum value considered safe to avoid the development of undesired microorganisms (0.60) (Leong et al., 2011), suggesting microbiological stability. Therefore, low-moisture microencapsules are generally more stable, while those with high levels of sugar in their composition have greater hygroscopicity, which can result in an increase in the concentration gradient of water in the product and in the surrounding air (Otálora et al., 2018).

The pH of MPPE (Fig. 5 C) slightly increased during storage (from 4.37 to 4.75) and kept almost stable mainly in the time ranges 0–8 days and 60–90 days. Although most bacteria grow optimally in almost neutral environment ($6.6 < pH < 7.5$) and some even under strongly acidic conditions ($pH < 4.5$), the pH range of MPPE appears to be unfavorable for pathogenic and spoilage microorganisms.

In relation to the L*, a* and C* coordinates, there was no important variation in these parameters during stability, probably due to the low betalain content (Table 7).

MPPE showed a significant increase ($p < 0.05$) in the hue angle at 30 days of storage, followed by a significant reduction ($p < 0.05$) in subsequent storage times. Overall, over 90 days of storage, the MPPE exhibited a light and slightly pinkish hue. However, according to the stability study results, the times of 8, 30, 60, and 90 days showed a perceptible color difference to the human eye when compared to time

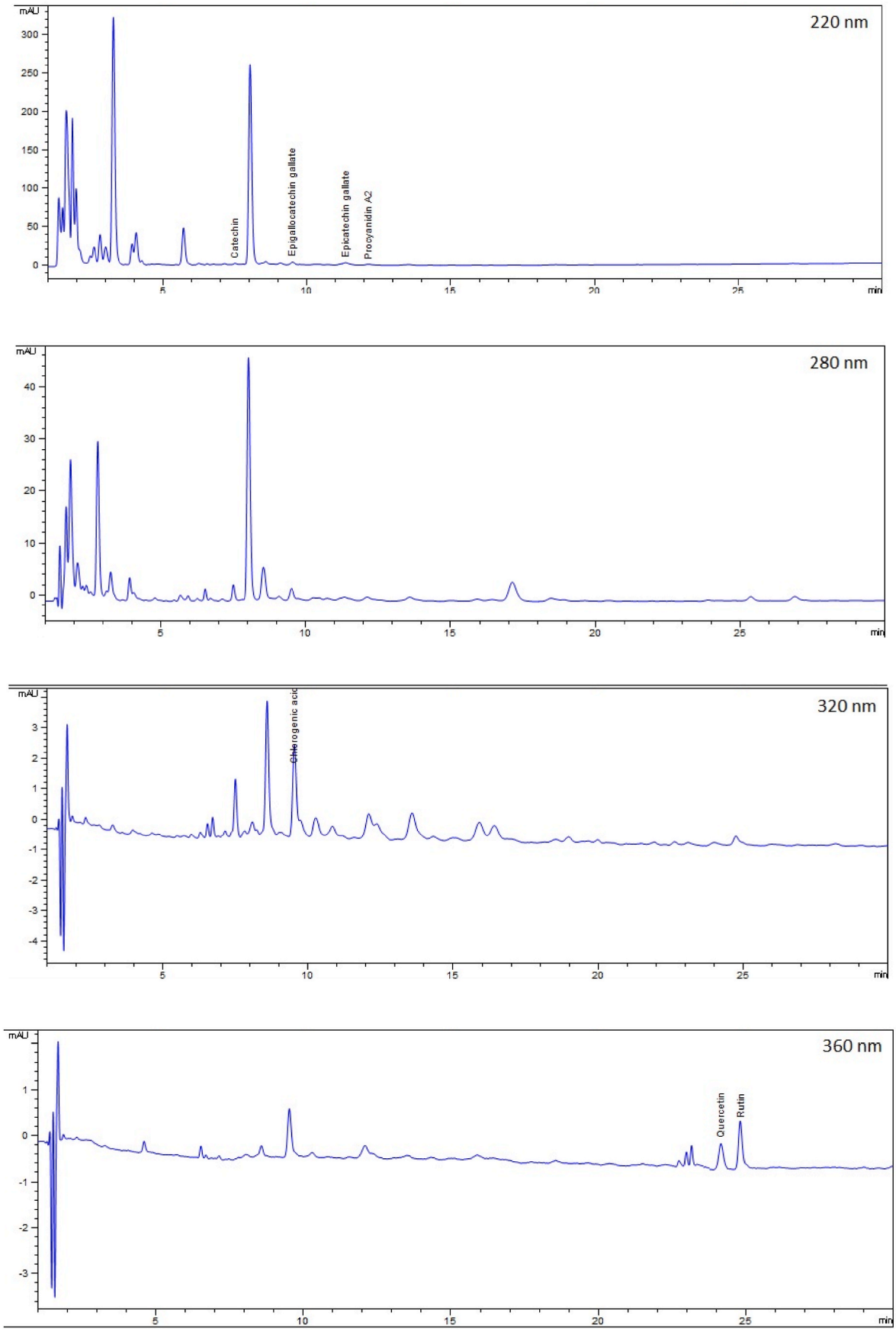

Fig. 6. HPLC-DAD chromatograms of phenolic compounds identified in microencapsulated pitaya (*Hylocereus polyrhizus*) pulp extract. The identification of the compounds is given in Table 8.

Table 9
Validation parameters of phenolic compounds determination by HPLC-DAD.

Phenolic compounds	Range (mg L^{-1})	Calibration curve	Correlation coefficient (r)	Precision (CV%)	LOD (mg.L^{-1})	LOQ (mg.L^{-1})
Phenolic acids						
Chlorogenic acid	0.55–17.5	$y = 9.608\ x + 8.203$	0.9992	0.27	0.11	0.13
Flavanols						
Catechin	0.55–17.5	$y = 24.639\ x + 0.705$	0.9999	0.26	0.06	0.07
Epicatechin gallate	0.55–17.5	$y = 9.072\ x + 0.352$	0.9997	0.40	0.06	0.07
Epigallocatechin gallate	0.55–17.5	$y = 3.615\ x + 0.405$	0.9995	0.29	0.04	0.05
Procyanidin A2	0.5–20	$y = 7.962\ x - 1.686$	0.9995	0.19	0.02	0.04
Quercetin	0.5–20	$y = 4.733\ x - 0.500$	0.9995	0.55	0.11	0.20
Rutin	0.5–20	$y = 5.132\ x - 0.030$	0.9992	0.74	0.10	0.17

y = peak height (mAU); x = amount (mg/L); CV = coefficient of variation; LOD = limit of detection; LOQ = limit of quantification.

0 ($\Delta E > 2.15$).

3.8. Phenolics profile of microencapsulated pitaya pulp extract by HPLC-DAD

Table 8 and Fig. 6 show the phenolic compounds identified in the microencapsulated pitaya pulp extract, namely chlorogenic acid, catechin, epigallocatechin gallate, epicatechin gallate, procyanidin A2, rutin and quercetin, while Table 9 lists the related instrumental results.

Tenore et al. (2012) identified, by HPLC-DAD and HPLC-MS-MS, quercetin-3-*O*-rutinoside, gallic acid, protocatechuic acid, *p*-hydroxybenzoic acid, vanillic acid, syringic acid, cinnamic acid and caffeic acid in Chinese red pitaya pulp and peel, Vijayakumar et al. (2020) identified, using UPLCQTOF/MS, four types quercetin derivatives, namely quercetin 3-*O*-(6-acetylglucoside), quercetin 7-glucoside, quercetin 3-*O*-galactoside and quercetin 3-*O*-glucuronide in the peel of red pitaya grown in Malaysia, and Suleria et al. (2020) identified, using LC-ESIQTOF-MS/MS, chlorogenic acid in the peel of red pitaya grown in Australia. As stressed by Bellucci et al. (2021), these differences in the identified compounds should be ascribed to the different identification methods, extraction procedures, solvents, fruit species and varieties, soils, and climate conditions.

4. Conclusions

The best experimental conditions for the recovery of betalains and phenolic compounds from red pitaya (*Hylocereus polyrhizus*) pulp using ultrasound-assisted extraction (UAE) was 30% ethanol concentration, 40 mL/g solvent/sample ratio and 20 min ultrasound immersion time. The pitaya pulp extract obtained using ultrasounds showed significantly higher levels of betalains and phenolic compounds than the one obtained by conventional extraction, indicating that UAE was successful in extracting these compounds. Microencapsulation by spray drying allowed satisfactory efficiency of betalains and phenolic compounds encapsulation, in addition to a high antioxidant activity and favorable physical and physicochemical characteristics. At the end of a 90-day storage stability study, the microencapsulated extract of red pitaya fruit pulp showed only a slight decrease in the levels of betalains and phenolic compounds. Even though these results indicate a great potential for use of the microencapsulated extract as a functional ingredient and/or natural pigment in the food and pharmaceutical industries, possible industrial applications would need identifying the betalain profile, increasing the stability time and applying the microencapsulated extract in a real food matrix, which will be the objects of future efforts in this field.

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CRediT authorship contribution statement

Thaís Regina Rodrigues Vieira: Conceptualization, Formal analysis, Investigation, Methodology, Visualization, Writing – original draft. **Amanda Barbosa Lima:** Investigation. **Christine Maria Carneiro Maranhão Ribeiro:** Formal analysis. **Priscilla Vanúbia Queiroz de Medeiros:** Resources. **Attilio Converti:** Visualization, Writing – review & editing. **Marcos dos Santos Lima:** Investigation, Methodology. **Maria Inês Sucupira Maciel:** Funding acquisition, Resources, Supervision, Writing – review & editing.

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.

Data availability

Data will be made available on request.

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References

Al-Dhabi, N. A., Ponmurugan, K., & Maran, P. (2017). Development and validation of ultrasound-assisted solid-liquid extraction of phenolic compounds from waste spent coffee grounds. *Ultrasonics Sonochemistry, 34*, 206–213. https://doi.org/10.1016/j.ultsonch.2016.05.005

Alves, A. I., Rodrigues, M. Z., Pinto, M. R. M. R., Vanzela, E. S. L., Stringheta, P. C., Perrone, Í. T., & Ramos, A. M. (2017). Morphological characterization of pequi extract microencapsulated through spray drying. *International Journal of Food Properties, 20*, 1298–1305. https://doi.org/10.1080/10942912.2017.1343344

AOAC. (2016). *Official methods of analysis* (20th ed.). Washington: Association of Official Analytical Chemists.

Bakar, J., Ee, S. C., Muhammad, K., Hashim, D. M., & Adzahan, N. (2012). Spray-drying optimization for red pitaya peel (*Hylocereus polyrhizus*). *Food and Bioprocess Technology, 6*, 1332–1342. https://doi.org/10.1007/s11947-012-0842-5

Barbosa-Cánovas, G. V., & Juliano, P. (2005). Physical and chemical properties of food powders. In C. Onwulata (Ed.), *Encapsulated and powdered foods* (pp. 40–67). United Kingdom: Taylor & Francis. https://doi.org/10.1201/9781420028300-6/physical-chemical-properties-food-powders-gustavo-barbosa-cánovas-pablo-juliano?context=ubx&refId=888dea72-4d1a-4be7-991b-ed6c6831bd6d.

Bellucci, E. R. B., Munekata, P. E. S., Pateiro, M., Lorenzo, J. M., & Barretto, A. C. S. (2021). Red pitaya extract as natural antioxidant in pork patties with total replacement of animal fat. *Meat Science, 171*, Article 108284. https://doi.org/10.1016/j.meatsci.2020.108284

Benzie, I. F., & Strain, J. J. (1996). The ferric reducing ability of plasma (FRAP) as a measure of "antioxidant power": The FRAP assay. *Analytical Biochemistry, 239*, 70–76. https://doi.org/10.1006/abio.1996.0292

Bhagya Raj, G. V. S., & Dash, K. K. (2020). Ultrasound-assisted extraction of phytocompounds from dragon fruit peel: Optimization, kinetics and thermodynamic

studies. *Ultrasonics Sonochemistry, 68*, Article 105180. https://doi.org/10.1016/j.ultsonch.2020.105180
Cai, Y., & Corke, H. (2000). Production and properties of spray-dried *Amaranthus* betacyanin pigments. *Journal of Food Science, 65*, 1248–1252. https://doi.org/10.1111/j.1365-2621.2000.tb10273.x
Caldas, T. W., Mazza, K. E. L., Teles, A. S. C., Mattos, G. N., Brígida, A. I. S., Conte-Junior, C. A., Borguini, R. G., Godoy, R. L. O., Cabral, L. M. C., & Tonon, R. V. (2018). Phenolic compounds recovery from grape skin using conventional and non-conventional extraction methods. *Industrial Crops and Products, 111*, 86–91. https://doi.org/10.1016/j.indcrop.2017.10.012
Cano-Chauca, M., Ramos, A. M., & Stringheta, P. C. (2005). Effect of the carriers on the microstructure of mango powder obtained by spray drying and its functional characterization. *Innovative Food Science & Emerging Technologies, 6*, 420–428. https://doi.org/10.1016/j.ifset.2005.05.003
Caparino, O. A., Tang, J., Nindo, C. L., Sablani, S. S., Powers, J. R., & Fellman, J. K. (2012). Effect of drying methods on the physical properties and microstructures of mango (*Philippine 'Carabao'* var.) power. *Journal of Food Engineering, 111*, 135–148. https://doi.org/10.1016/j.jfoodeng.2012.01.010
Carmona, J. C., Robert, P., Vergara, C., & Sáenz, C. (2021). Microparticles of yellow-orange cactos pear pulp (*Opuntia ficus-indica*) with clacode mucilage and maltodextrin as a food coloring in yogurt. *LWT - Food Science and Technology, 138*, Article 110672. https://doi.org/10.1016/j.lwt.2020.110672
Carpes, S. T., Pereira, D., Moura, C., Reis, A. S., Silva, L. D., Oldoni, T. L. C., Almeida, J. F., & Plata-Oviedo, M. V. S. (2020). Lyophilized and microencapsulated extracts of grape pomace from winemaking industry to prevent lipid oxidation in chicken paté. *Brazilian Journal of Food Technology, 23*, Article 2019112. https://doi.org/10.1590/1981-6723.11219
Codex Alimentarius, International Food Standards. (1981). *Standard for cocoa powders (cocoas) and dry mixtures of cocoa and sugars*. Retrieved from . (Accessed 22 March 2022).
Codex Alimentarius, International Food Standards. (1999). *Standard for milk powders and cream powder*. Retrieved from . (Accessed 22 March 2022).
Coradin, L., Camillo, J., & Pareyn, F. G. C. (2018). *Espécies nativas da flora brasileira de valor econômico atual ou potential – plantas para o futuro: Região Nordeste. Brasilia: Biblioteca do Ministério do Meio Ambiente*. https://ainfo.cnptia.embrapa.br/digital/bitstream/item/189688/1/Livro-Nordeste-1-2018.pdf.
Fathordoobady, F., Jarzębski, M., Pratap-Singh, A., Guo, Y., & Abd-Manap, Y. (2021). Encapsulation of betacyanins from the peel of red dragon fruit (*Hylocereus polyrhizus* L.) in alginate microbeads. *Food Hydrocolloids, 113*, Article 106535. https://doi.org/10.1016/j.foodhyd.2020.106535
Fazaeli, M., Emam-Djomeh, Z., Ashtari, A. K., & Omid, M. (2012). Effect of spray drying conditions and feel composition on the physical properties of black mulberry juice powder. *Food and Bioproducts Processing, 90*, 667–675. https://doi.org/10.1016/j.fbp.2012.04.006
Fu, L., Xu, B. T., Xu, X. R., Gan, R. Y., Zhang, Y., Xia, E. Q., & Li, H. B. (2011). Antioxidant capacities and total phenolic contents of 62 fruits. *Food Chemistry, 129*, 345–350. https://doi.org/10.1016/j.foodchem.2011.04.079
Ibrahim, S. R. M., Mohamed, G. A., Khedr, A. I. M., Zayed, M. F., & El-Kholy, A. A. E. S. (2018). Genus *Hylocereus*: Beneficial phytochemicals, nutritional importance, and biological relevance – a review. *Journal of Food Biochemistry, 42*, 1–29. https://doi.org/10.1111/jfbc.12491
Jafari, S. M., Ghalenoei, M. G., & Dehnad, D. (2017). Influence of spray drying on water solubility index, apparent density, and anthocyanin content of pomegranate juice powder. *Powder Technology, 311*, 59–65. https://doi.org/10.1016/j.powtec.2017.01.070
Kumar, N., Gupta, S., Yadav, T. C., Pruthi, V., Varadwaj, P. K., & Goel, N. (2019). Extrapolation of phenolic compounds as multi-target agents against cancer and inflammation. *Journal of Biomolecular Structure and Dynamics, 37*, 2355–2369. https://doi.org/10.1080/07391102.2018.1481457
Kumar, K., Srivastav, S., & Sharanagat, V. S. (2021). Ultrasound assisted extraction (UAE) of bioactive compounds from fruit and vegetable processing by-products: A review. *Ultrasonics Sonochemistry, 70*, Article 105325. https://doi.org/10.1016/j.ultsonch.2020.105325
Leong, S. L., Pettersson, O. V., Rice, T., Hocking, A. D., & Schnürer, J. (2011). The extreme xerophilic mould xeromyces bisporus – growth and competition at various water activities. *International Journal of Food Microbiology, 145*, 57–63. https://doi.org/10.1016/j.ijfoodmicro.2010.11.025
Li, Y., Tang, B., Chen, J., & Lai, P. (2018). Microencapsulation of plum (*Prunus salicina* Lindl.) phenolics by spray drying technology and storage stability. *Food Science and Technology, 38*, 530–536. https://doi.org/10.1590/1678-457X.09817
Lourenço, S. C., Moldão-Martins, M., & Alves, V. D. (2020). Microencapsulation of pineapple peel extract by spray drying using maltodextrin, inulin, and Arabic gum as wall matrices. *Foods, 9*, 718. https://doi.org/10.3390/foods9060718
Marzuki, N. H. C., Hamid, M. A., & Wahab, R. A. (2018). Assessment of fatty acid composition and response surface optimization of ultrasonic-assisted extraction of phenolic compounds from Pouteria campechiana pulp. *Malaysian Journal of Fundamental and Applied Sciences, 14*, 269–277. https://doi.org/10.11113/mjfas.v14n2.984
Meregalli, M. M., Puton, B. M. S., Camera, F. D., Amaral, A. U., Zeni, J., Cansian, R. L., Mignoni, M. L., & Backes, G. T. (2020). Conventional and ultrasound-assisted methods for extraction of bioactive compounds from red araçá peel (*Psidium cattleianum Sabine*). *Arabian Journal of Chemistry, 13*, 5800–5809. https://doi.org/10.1016/j.arabjc.2020.04.017
Mordor Intelligence Research & Advisory. (2023). *Mercado de fitonutrientes - crescimento, tendências, impacto do COVID-19 e previsões (2023 - 2028)*. *Mordor Intelligence*. https://www.mordorintelligence.com/pt/industry-reports/phytonutrients-market.
Moser, P., Telis, V. R. N., Andrade Neves, N., García-Romero, E., Gómez-Alonso, S., & Hermosín-Gutiérrez, I. (2017). Storage stability of phenolic compounds in powdered BRS Violeta grape juice microencapsulated with protein and maltodextrin blends. *Food Chemistry, 214*, 308–318. https://doi.org/10.1016/j.foodchem.2016.07.081
Nizori, A., Bui, L. T. T., Jie, F., & Small, D. M. (2018). Impact of varying hydrocolloid proportions on encapsulation of ascorbic acid by spray drying. *International Journal of Food Science and Technology, 53*, 1363–1370. https://doi.org/10.1111/ijfs.13699
OECD-FAO. (2019). *Agricultural outlook 2019-2028: Special focus: Latin America*. Retrieved from http://www.fao.org/3/ca4076en/ca4076en.pdf. (Accessed 22 June 2023).
Otálora, M. C., Carriazo, J. G., Osorio, C., & Nazareno, M. A. (2018). Encapsulation of cactus (*Opuntia megacantha*) betaxanthins by ionic gelation and spray drying: A comparative study. *Food Research International, 111*, 423–430. https://doi.org/10.1016/j.foodres.2018.05.058
Ramli, N. S., Ismail, P., & Rahmat, A. (2014). Influence of conventional and ultrasonicassisted extraction on phenolic contents, betacyanin contents, and antioxidant capacity of red dragon fruit (*Hylocereus polyrhizus*). *The Scientific World Journal*. , Article 964731. https://doi.org/10.1155/2014/964731
Rezende, Y. R. R. S., Nogueira, J. P., & Narain, N. (2018). Microencapsulation of extracts of bioactive compounds obtained from acerola (*Malpighia emarginata* D. C.) pulp and residue by spray and freeze drying: Chemical, morphological and chemometric characterization. *Food Chemistry, 254*, 281–291. https://doi.org/10.1016/j.foodchem.2018.02.026
Rocchetti, G., Blasi, F., Montesano, D., Ghisoni, S., Marcotullio, M. C., Sabatini, S., Cossignani, L., & Lucini, L. (2019). Impact of conventional/non-conventional extraction methods on the untargeted phenolic profile of Moringa oleifera leaves. *Food Research International, 115*, 319–327. https://doi.org/10.1016/j.foodres.2018.11.046
Saénz, C., Tapia, S., Chávez, J., & Robert, P. (2009). Microencapsulation by spray drying of bioactive compounds from cactus pear (*Opuntia ficus-indica*). *Food Chemistry, 11*, 616–622. https://doi.org/10.1016/j.foodchem.2008.09.095
Salar, R. K., Purewal, S. S., & Bhatti, M. S. (2016). Optimization of extraction conditions and enhancement of phenolic content and antioxidant activity of pearl millet fermented with *Aspergillus awamori* MTCC-548. *Resource-Efficient Technologies, 2*, 148–157. https://doi.org/10.1016/j.reffit.2016.08.002
Schuck, P., & Ouest, A. (2011). Milk powder: Physical and functional properties of milk powders (2011). In J. W. Fuquay, P. F. Fox, & P. L. H. McSweeney (Eds.), *Encyclopedia of dairy sciences* (pp. 117–124). Cambridge: Academic Press. https://www.sciencedirect.com/referencework/9780123744074/encyclopedia-of-dairy-sciences.
Shaaruddin, S., Mohd Ghazali, H., Mirhosseini, S. H., & Muhammad, K. (2017). Stability of betanin in pitaya powder and confection as affected by resistant maltodextrin. *LWT - Food Science and Technology, 84*, 129–134. https://doi.org/10.1016/j.lwt.2017.05.031
Soto-Castro, D., Chavez, G. M., León-Martínez, F., Santiago-García, A., Aragón-Lucero, I., & Antonio-Antonio, F. (2019). Spray drying microencapsulation of betalain rich extracts from *Escontria chiotilla* and *Stenocereus queretaroensis* fruits using cactus mucilage. *Food Chemistry, 272*, 715–722. https://doi.org/10.1016/j.foodchem.2018.08.069
Stintzing, F. C., Herbach, K. M., Mosshammer, M. R., Carle, R., Yi, W., Sellappan, S., Akoh, C. C., Bunch, R., & Felker, P. (2005). Color, betalain pattern, and antioxidant properties of cactus pear (*Opuntia* spp.) clones. *Journal of Agricultural and Food Chemistry, 53*, 442–451. https://doi.org/10.1021/jf048751y
Suleria, H. A., Barrow, C. J., & Dunshea, F. R. (2020). Screening and characterization of phenolic compounds and their antioxidant capacity in different fruit peels. *Foods, 9*, 1206. https://doi.org/10.3390/foods9091206
Tenore, G. C., Novellino, E., & Basile, A. (2012). Nutraceutical potential and antioxidant benefits of red pitaya (*Hylocereus polyrhizus*) extracts. *Journal of Functional Foods, 4*, 129–136. https://doi.org/10.1016/j.jff.2011.09.003
Vargas-Campos, L., Valle-Guadarrama, S., Martinez-Bustos, F., Salinas-Moreno, Y., Lobato-Calleros, C., & Calvo-Lopez, A. D. (2018). Encapsulation and pigmenting potential of betalains of pitaya (*Stenocereus pruinosus*) fruit. *Journal of Food Science and Technology-Mysore, 55*, 2436–2445. https://doi.org/10.1007/s13197-018-3161-7
Vergara, C., Saavedra, J., Sáenz, C., García, P., & Robert, P. (2014). Microencapsulation of pulp and ultrafiltered cactus pear (*Opuntia ficus-indica*) extracts and betanin stability during storage. *Food Chemistry, 157*, 246–251. https://doi.org/10.1016/j.foodchem.2014.02.037
Vijayakumar, R., Abd Gani, S. S., Zaidan, U. H., Halmi, M. I. E., Karunakaran, T., & Hamdan, M. R. (2020). Exploring the potential use of Hylocereus polyrhizus peels as a source of cosmeceutical sunscreen agent for its antioxidant and photoprotective properties. *Evidence-based Complementary and Alternative Medicine, 2020*, Article 7520736. https://doi.org/10.1155/2020/7520736
Wettasinghe, M., & Shahidi, F. (1999). Evening primrose meal: A source of natural antioxidants and scavenger of hydrogen peroxide and oxygen-derived free radicals. *Journal of Agricultural and Food Chemistry, 47*, 1801–1812. https://doi.org/10.1021/jf9810416
Ye, Q., Georges, N., & Selomulya, C. (2018). Microencapsulation of active ingredients in functional foods: From research stage to commercial food products. *Trends in Food Science & Technology, 78*, 167–179. https://doi.org/10.1016/j.tifs.2018.05.025
Zotarelli, M. F., Silva, V. M., Durigon, A., Hubinger, M. D., & Laurindo, J. B. (2017). Production of mango powder by spray drying and cast-tape drying. *Powder Technology, 305*, 447–454. https://doi.org/10.1016/j.powtec.2016.10.027