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REVIEW PAPER

Physicochemical characteristics and bioactive compounds of the Xique-xique (*Pilosocereus gounellei*) cactus from Caatinga Brazilian: are they nutritive and functional?

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Abstract

Xique-xique (*Pilosocereus gounellei*) is a cactus found in the Caatinga biome which is used in folk medicine for treating inflammation, and also used in animal food. This study evaluated its proximate composition and bioactive compounds, as well as its bioaccessibility and antioxidant activity. Experiments were performed on different parts of the plant. The central stem of the xique-xique can be considered a source of insoluble dietary fiber (5.18 g/100 g) and soluble fiber (1.37 g/100 g). The vascular cylinder contained the highest mineral content: K (308.4 mg/100 g), Ca (145.57 mg/100 g), Mg (182.40 mg/100 g), and Mn (7.71 mg/100 g). The vascular cylinder group presented the highest activity for capturing DPPH radicals (572.96 µM of TEAC/100 g) and ferric reducing ability (1912.95 µM of TEAC/100 g). Catechin and epigallocatechin-gallate were the major phenolic compounds, while catechin, epigallocatechin gallate and procyanidin B and gallic acid were the most bioaccessible compounds after in vitro gastrointestinal digestion. Xique-xique can be considered as a potential food alternative with nutritional value and bioactive compounds, indicating its potential use and enrichment in various food products.

Keywords Antioxidants · Bioaccessibility · Cactaceae · Proximate composition · Unconventional food plants

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Introduction

Brazil has a vast territorial extension made up of different biomes with different climatic characteristics, thus contributing to the biodiversity of plant species exploited for consumption for their nutritional properties and health benefits [1]. The Cactaceae family is the sixth family with the most species found in the Brazilian Caatinga, and in this same biome it presents itself as the third in diversity [2, 3]. Caatinga native cacti, such as *Pilosocereus gounellei* and *Cereus jamacarau*, are species that stand out in this biome and are natural resources, used in periods of prolonged drought, either as food or as animal fodder [4]. These plants are found in regions with high temperatures and are resistant to drought. Because they have physiological mechanisms specialized in water storage, cactaceae become reserves of water and food, providing essential resources in times of drought [5]. Due to their morphological and physiological adaptations to low rainfall, cacti are prominent in the Caatinga [6].

Cacti are considered an unconventional food plant and constitute a source of food for the Brazilian semiarid population destined for their own consumption, in addition to animal forage [7]. However, some of these plants are not used or are underutilized [8] due to a lack of scientific information on their nutritional and biological properties. The genus *Pilosocereus* has thirty-five species distributed from Mexico to Paraguay, with greater diversity of species occurring in Brazil [9].

The specie *Pilosocereus gounellei* popularly known as xique-xique, is a cactus that is found exclusively in the Brazilian Caatinga. It grows in the semiarid region, in shallow soils and sandy and rocky outcrops [10, 11]. Its structure presents an erect stem, hazy green color, and its branches are separated from each other with curved, broad and soft sides in the direction of the ground armed with sharp spines [12]. Because it is a cactacea extracted from nature, there is no tradition of cultivation by man and for this reason, during the dry period, it is destined to feed ruminants [13].

Ethnobotanical studies reports that for human consumption, the cladodes (which are modified stems) are usually eaten peeled, fresh or cooked for use in cakes, sweets, biscuits and flours, and their fruits are collected and appreciated due to their delicate, sweet taste, being consumed *in natura* by the local population [6, 14, 15]. In Brazilian folk medicine, the cladodes are used to treat prostate gland inflammation, jaundice, hyperglycemia and injuries [16]. Scientific studies have verified the presence of phytochemicals such as flavonoids, pinostrobin, β-sitosterol, β-sitosterol, stigmasterol, kaempferol and quercetin [17, 18].

A recent study by our group demonstrated that the intake of xique-xique juice in rats with colitis had a protective effect on intestinal inflammation and decreased both pro-inflammatory markers and oxidative stress. These effects were attributed to the phenolic compounds and fibers present in the xique-xique juice [19]. [20] indicated the potential prebiotic effect of xique-xique cladode juice. Another in vivo study demonstrated significant gastroprotection by the ethanolic extracts of the xique-xique cladodes [18].

This study aims to evaluate following in the xique-xique cactus: proximate composition, sugar profile, amino acid, and minerals as well as the profiles of the phenolic compounds. Further, the antioxidant potential and bioaccessibility of the phenolic compounds after simulated gastrointestinal digestion of xique-xique cactus were assessed.

Material and methods

Chemical products and reagents

Pepsin, alpha-amylase (from human saliva Type IX-A, 1000–3000 U mg/protein), pancreatin (from porcine pancreas 8 × USP specifications), and bile salts were obtained from Sigma-Aldrich (St. Louis, Missouri, USA). Hydrochloric acid (HCl) (37% w/w) and methanol were purchased from Neon (São Paulo, Brazil). The 2,2′-diphenyl-2-picrylhydrazyl (DPPH) and hydroxy-2,5,7,8-tetramethylchroman-2-carboxylic acid (Trolox) chemicals were obtained from Sigma-Aldrich Chemical, SA (Hamburg, Germany) and 2,4,6-tris (2-pyridyl)-s-triazine (TPTZ) was purchased from Sigma-Aldrich Chemical, SA (Milan, Italy).

External standards of the phenolics: chlorogenic acid, syringic acid, gallic acid, p-coumaric acid, trans-caftaric acid, caffeic acid, hesperidin, naringenin, procyanidin B1, procyanidin B2, catechin and epicatechin Sigma-Aldrich (St. Louis, MO, USA). Procyanidin A2, epicatechin gallate, epigallocatechin gallate, kaempferol 3-glucoside, quercetin 3-glucoside, quercetin 3-rutinoside (rutin), myricetin came from Extrasynthese (Genay, France). Cis-Resveratrol and trans-resveratrol were obtained from Cayman Chemical Company (Ann Arbor, MI, USA).

Standards of phenolic compounds for high-performance liquid chromatography (HPLC): catechin, epicatechin, epigallocatechin gallate, procyanidin B1 and B2, quercetin 3-glucoside, rutin, kaempferol 3-glucoside, gallic acid, and syringic acid were purchased from Sigma-Aldrich (St. Louis, MO, USA). Sugar standards: glucose, fructose, galactose, sucrose, arabinose and xylose were obtained from Sigma-Aldrich (St. Louis, MO, USA). The mineral standards: magnesium, calcium, sodium, manganese, phosphorus, iron, zinc and selenium were obtained from Specsol (Quimlab, Jacareí, Brazil) and Merck (Darmstadt, Germany), and the amino acid standards were from Sigma-Aldrich.

Plant material

Xique-xique (*Pilosocereus gounellei*) cladodes were collected in a private growing area located in the municipality of Boa Vista, Paraíba, Brazil (07°15′32″ S 36°14′24″ W) in three distinct lots (approximately 30 kg) in April 2016. The cladodes were selected considering their physical integrity, and then transported in boxes made of polystyrene under a temperature of 5 ± 1.0 °C.

The material was botanically identified by Prof. Dr. Leonardo Person Felix of the Centre of Agrarian Sciences of the Federal University of Paraíba—(*CCA/UFPB*) and the certified species (17.562) was deposited in the Prof. Jaime Coelho Morais Herbarium (*CCA/UFPB*). Plant material collection was recorded in the Information and Biodiversity System of Brazil (*SISBIO*) number (62681) and the National System for Management of Genetic Heritage and Associated Traditional Knowledge (*SISGEN*) number (AA17429).

Preparation of samples

The sample preparation was followed by washing the cladodes in running water and disinfecting by immersion in chlorinated water (100 ppm) for 15 min, and then rinsed. A sample was collected from each batch, homogenized and triplicates were made. They were subsequently cut into pieces of 5 cm × 5 cm and divided into two structural groups, classified as vascular cylinder and central stem, as illustrated in Fig. 1. The experimental groups were crushed with a processor (Brand Oster® model 2619-057) to form a paste and stored under freezing conditions (− 20 ± 1.0 °C) for later analysis.

Physical characterization and proximate composition

The proximate composition was determined by analyses of moisture content, ash, protein, and total fiber (soluble an insoluble), as recommended by the methodology according to the *Association of Official Analytical Chemist (AOAC)* [21]: moisture, ash, protein (method 920.87); dietary fibers (insoluble and soluble) contents (method 991.43). The physical and physical–chemical parameters of water activity (wa) (AquaLab® model CX-2) pH was measured using a potentiometer (Quimis® model Q400as). Total acidity was determined by titrations with 0.1 N NaOH, using phenolphthalein as an indicator and the results was expressed in % acid malic. Total soluble solids (TSS) were measured by a digital refractometer (Hanna®, HI 96,801). The lipids were

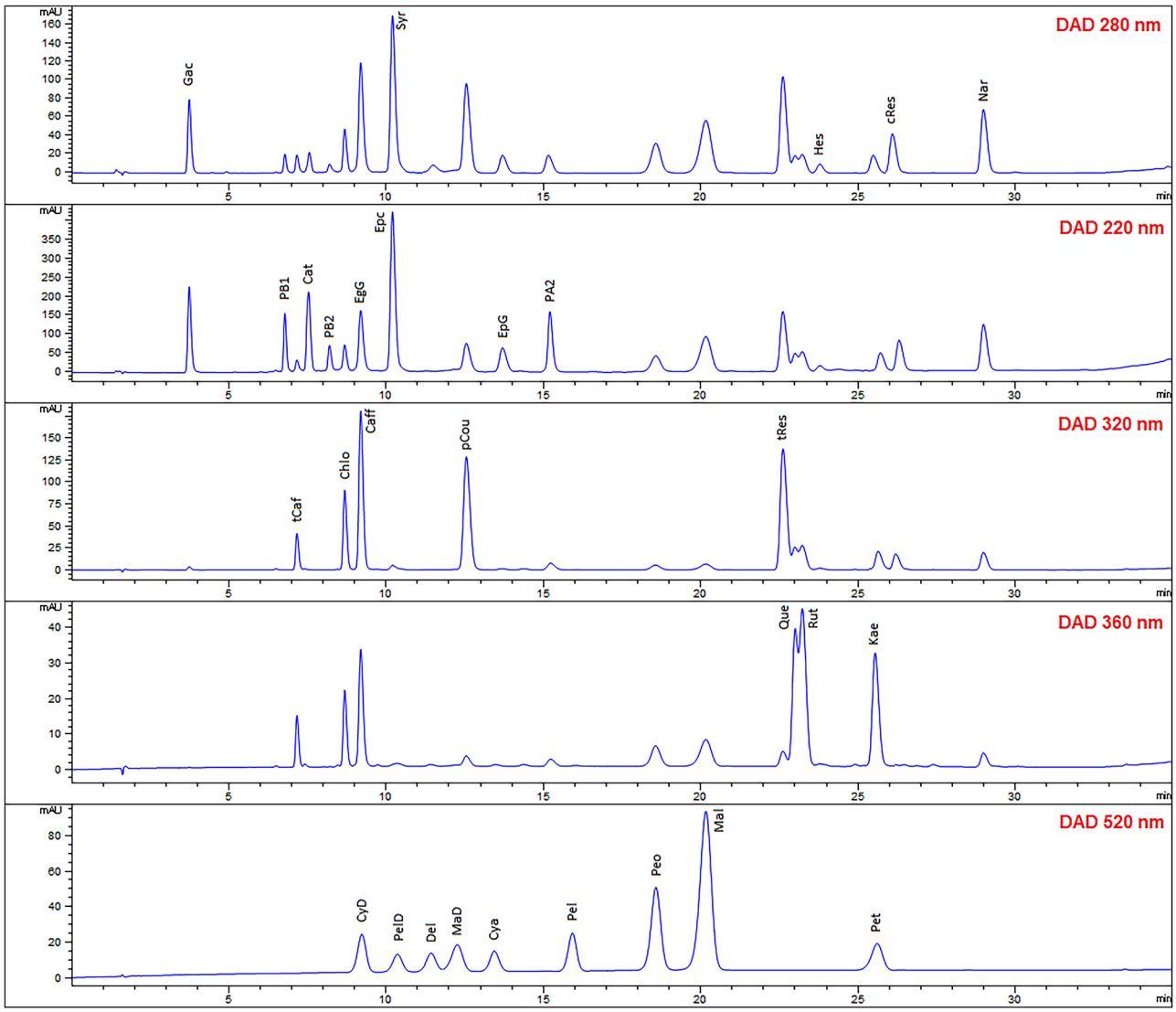

Fig. 1 Chromatogram of the phenolic compounds external standards in the sample of the xique-xique

determined according to [22], with extraction in a chloroform–methanol solution (2:1).

Sugar profile

The hydrolysis process of the vascular cylinder was performed in triplicate according to [23], in which 300 mg of sample was hydrolyzed with 3 mL of 72% sulphuric acid (m/v) in a 30 ± 1 °C bath for 60 min, with samples being stirred every five min. Next, 84 mL of distilled water was added after 60 min, and the hydrolysis process ended in the autoclave for an additional 60 min. Sugar were determined following the methodology of [24]. The samples were filtered using a vacuum pump, and the liquid was analyzed by ion exchange chromatography with pulsed amperometric detection (HPLC-PAD) using Dionex® chromatograph (Sunnyvale, CA, USA) coupled with CarboPac PA1 (4 × 250 mm) column, PA1 (4 × 50 mm) guard column, GP50 gradient pump, ED40 electrochemical detector, and PEAKNET software. The mobile phases used were A NaOH (50 mmol) and B (NaOAc 500 mmol + NaOH 50 mmol). The run was performed for 40 min using the following elution gradients: from 0 to 25 min: 50% mobile phase A (50 mmol NaOH) and 50% D (water); column cleaning was then performed from 25 to 35 min using 62% B (500 mmol Sodium Acetate + 50 mmol NaOH) and 38% A (50 mmol NAOH); and the initial condition was then repeated (50% A and 50% D) from 35 to 40 min (Stabilization Period for the next run). Calibration curves were constructed using standard compounds ranging from 1 to 100 mg/L. Identification of the compound peaks was performed by comparing the retention times obtained in the injected standards. The standards used were glucose, fructose, galactose, sucrose, arabinose and xylose (Sigma-Aldrich, St. Louis, MO, USA). The triplicate mean results were expressed as g/100 g of fresh weight.

Mineral profile

The method described by the [21] (Methods 999.10) was used for determining the mineral profile. Thus, we used acid digestion assisted by microwaves as the sample preparation method, with 0.5 g of sample being weighed in Teflon tubes for the digestion, along with 7 mL of nitric acid which was purified by *sub-boiling* distillation (Distillacid®, Berghof, Eningen, Germany) and 1 mL of 30% hydrogen peroxide (Merck®, Darmstadt, Germany) added. The digestion proceeded at a maximum temperature of 170 °C for 37 min, and then the tube contents were transferred with purified water by reverse osmosis (Gehaka®, São Paulo, Brazil) to a 25 mL balloon. The analyses were performed in triplicate, and the analytical blank was prepared following the same procedure, omitting the sample.

Mineral determination was carried out on an atomic emission spectrometer with plasma and inductive coupling (ICP OES 5100 VDV, Agilent Technologies, Tokyo, Japan). The analysis conditions were: radio frequency power (1.2 kW); plasma flow rate (12 L/min); auxiliary flow rate (1.0 L/min); nebulization flow (0.7 L/min); plasma view (axial for Fe, Mn, and Zn; radial for Ca, K, Mg, P, Na); wave lengths: Ca (317.933 nm), Fe (259.940 nm), K (766.491 nm), Mg (279.553 nm), Mn (257.610 nm), P (213.618 nm), Na (589.592 nm), Se (196.026 nm), and Zn (206.200 nm). Quantification of the elements in samples consists of the correlation between measuring the signal intensity emitted/absorbed by the inorganic elements in relation to the analytical curve. The standard multi-element solution was acquired on Specsol with NIST certified analysis and traceability (MICPG6V-125). Results were expressed as mg/100 g of fresh weight.

Amino acid profile

The amino acid composition was determined according to the methodology described by [25]. The sample was subjected to acid hydrolysis (6 mol/L HCl) under heating (115 °C/22 h). After protein hydrolysis and the release of amino acids, phenylisothiocyanate (PITC) pre-column derivatization and separation of the phenylthiocarbamyl amino acid derivatives (PTC-aa) were performed in high-performance liquid chromatography (Shimadzu Corporation, Tokyo, Japan) on a C18-Luna-Phenomenex reverse phase column (250 mm × 4.6 mm, particle 5 µm; Phenomenex Inc., Torrence, CA, USA). The amino acids were identified and quantified using a standard external method (Pierce/PN 20088) and DAD detector at 254 nm, as described by [26]. The results were expressed as g/100 g of protein in dry weight.

Determination of antinutritional factors

Considering that these are some of the antinutritional factors usually presented as secondary products of plant metabolism, the concentrations of tannins, phytic acid and trypsin inhibitor were determined. Quantification of the total tannins was performed according to the method described by [21] (method 952.03), the phytic acid content according to [27] (detection limit of the 0.05 mg/g methods (considering the smallest point on the curve)), and trypsin inhibitor using the methodology described by [28]. The results were expressed in fresh weight.

Extraction phenolic compounds of the xique-xique vascular cylinder

The xique-xique vascular cylinder was weighed (2.5 g) in a polyethylene tube, then 10 mL of methanol and 10 mL of

acetone were added and subjected to ultrasound for a period of 30 min. It was then centrifuged and the supernatant was collected. Extraction was repeated 2 more times under the same conditions using the residue. The supernatants were combined and submitted to the concentration in a rotaevaporator (Fisatom 802, São Paulo, Brazil). Finally, the extract was resuspended in 2 mL of methanol and filtered through a PVDF filter (0.22 µm). This extract was used to determine the phenolic compound profile and antioxidant activity.

Determination of the phenolic compound profile

Identification and quantification of phenolic compounds was performed according to the methodology by [29], using an Agilent 1260 Infinity LC System liquid chromatograph (Agilent Technologies, Santa Clara, USA) attached to a diode arrangement detector (DAD) (model G1315D). The data were processed using the OpenLAB CDS ChemStation Edition software (Agilent Technologies, Santa Clara, USA). A Zorbax Eclipse Plus RP-C18 column (100×4.6 mm, 3.5 µm) and a Zorbax C18 pre-column (12.6×4.6 mm, 5 µm) (Zorbax®, USA) were used. The temperature was 35 °C and the injection volume was 20 µL of the sample, previously diluted in stage A, and filtered through a 0.45-µm membrane (Millex Millipore®, Barueri, SP, Brazil). The solvent flow was 0.8 mL/min. The solvent A was a phosphoric acid solution (0.1 mmol, pH 2.0) and solvent B was a solution of methanol with H_3PO_4. The gradient used in the separation was from 0 to 5 min: 5% B; 5–14 min: 23% B; 14–30 min: 50% B; 30–33 min 80% B. Detection of the compounds was carried out at 220, 280, 320, 360 and 520 nm, and identification and quantification by comparison with external standards. The chromatogram obtained from the mixture of the phenolic compounds external standards is shown in Fig. 1 in the supplementary material.

The spectral purity of the peaks was assessed by the threshold test (purity factor > 990) in order to ensure that there was no coelution in the quantified peaks, following the methodology of [30]. Chromatograms typical of the analysis of individual phenolic compounds in the sample of the xique-xique are shown in Figs. 2, 3 and 4.

In vitro gastrointestinal digestion of phenolic compounds

The xique-xique vascular cylinder was submitted to gastrointestinal physiology conditions and was evaluated in three sequential phases: oral, gastric and intestinal, as described by [31] with modifications. The crushed vascular cylinder (10 g) was then mixed with 5 mL of saline solution (2.38 g of Na_2HPO_4, 0.19 g of KH_2PO_4, 8 g of NaCl and 200 U/L of α-amylase) in amber flasks, and the mixture was agitated for 10 min in a water bath at 37 ± 2 °C to 95 rpm for the oral phase simulation. Then, 1 mg of pepsin was added and the system was acidified to 2.0 pH with HCl 0.1 mol/L, incubated at 37 °C with agitation at 95 rpm for 2 h for the gastric phase simulation. Then, 20 mL gastric chyme was immediately cooled and followed by the intestinal phase conditions. Then, 2.5 mL of pancreatin (80 mg) dissolved in 0.5 M $NaHCO_3$ and 2.5 mL were added bile salts. The intestinal phase were incubated on a shaker (95 rpm) at 37 °C for 2 h. The cellulose membranes (molecular weight cutoff of 12.000 Da) were completely immersed in a tube until it reached a pH of 5.0. In the final step, the dialysis membrane was removed and rinsed with distilled water. The dialyzed and non-dialyzed fractions were analyzed.

Bioaccessibility (BC) was determined following Eq. (1):

$$\text{Bioaccessibility (\%)} = (\text{BC dialyzed fraction} / \text{BC non-dialyzed fraction}) \times 100 \quad (1)$$

Antioxidant capacity

DPPH· radical scavenging (2,2-difenil-1picrilhidrazila)

Antioxidant capacity was determined by the ability to turn off the DPPH· radical according to the method of [32], with adaptations. Next, a DPPH solution of 0.06 mmol/L in methanol p.a. was prepared and stored in amber glass at 20 ± 1 °C until its use. An aliquot of 2.9 mL of this solution was mixed with 100 µL of the xique-xique extract and incubated at room temperature for 30 min in the dark. Afterwards, an assessment regarding the solution absorbance at 515 nm was conducted using a Cary 60 spectrophotometer (Agilent Technologies, Malaysia). The results were expressed in µM Trolox/100 g of the vascular cylinder, as determined from a standard Trolox curve (0–620.5 µM).

Ferric reducing ability plasm (Fe^{+2}) (FRAP)

The antioxidant potential by the FRAP method was determined using the methodology described by [33]. The xique-xique extract (90 µL) and 270 µL of distilled water were mixed with 2.7 mL of FRAP reagent. The FRAP reagent was prepared in 0.3 mol/L acetate buffer (pH 3.6), TPTZ (2,4,6-tris (2-pyridyl) -s-triazine) 10 mmol/L in a solution of HCl 40 mmol/L and 20 mmol $FeCl_3$). They were subsequently mixed in a tube and incubated for 30 min in a water bath at 37 °C. Absorbance (595 nm) was measured using the Cary 60 spectrophotometer (Agilent Technologies, Malaysia). The standard curve was performed with Trolox and the results are expressed in µM Trolox/100 g of vascular cylinder.

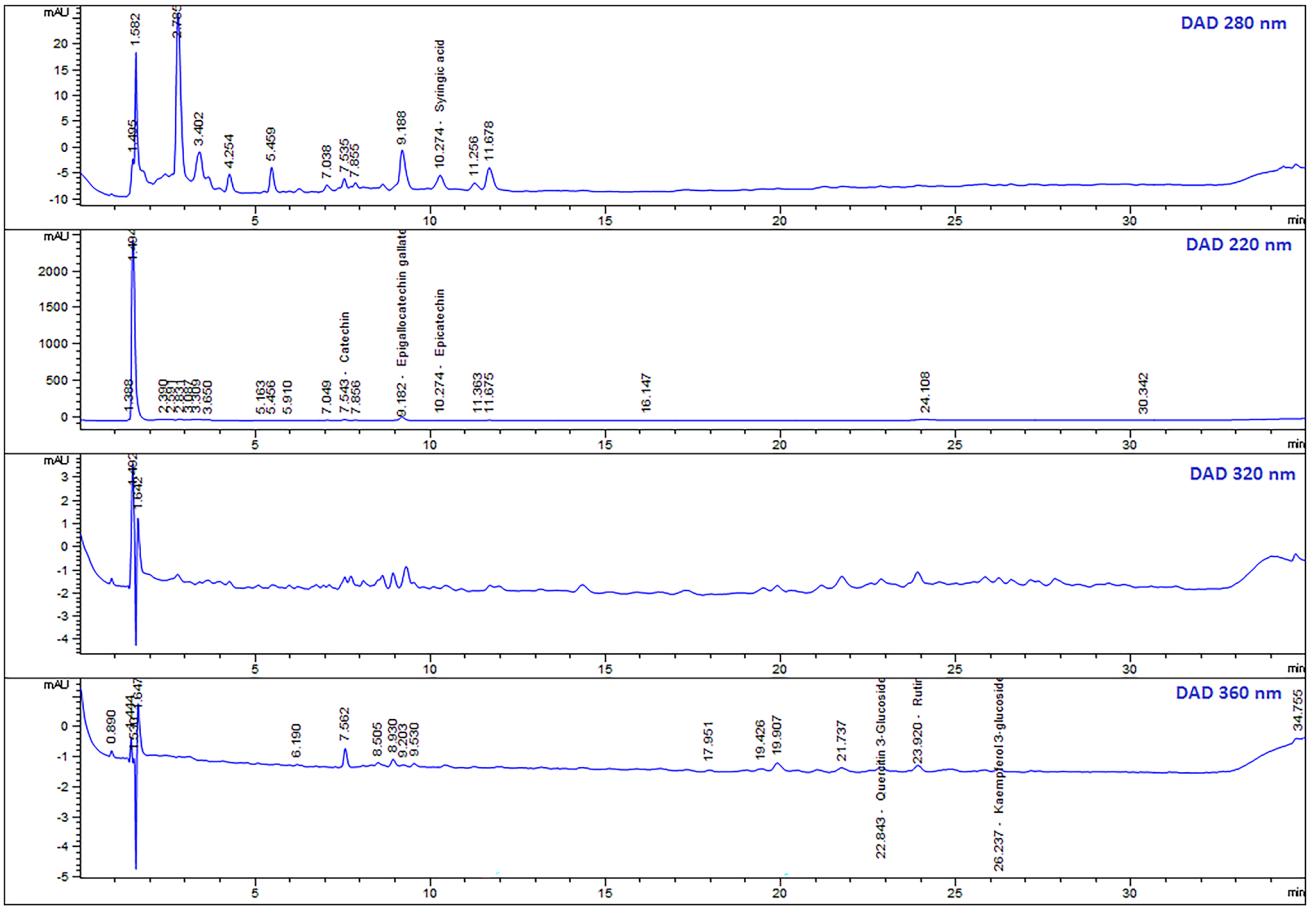

Fig. 2 Chromatograms typical of the analysis of total phenolic compounds in the xique-xique sample

Statistical analysis

All analyses were performed in triplicate. The values were expressed as mean ± standard deviation. The difference between means was evaluated using the Student's t test, after verifying the normal distribution. Statistical analyses were performed using GraphPad Prism 5.0 software (GraphPad Software Inc., San Diego, CA, USA).

Results and discussion

Physical characterization and proximate composition

The water activity and pH values did not differ between the experimental groups ($p > 0.05$). The pH of the xique-xique cladodes ranged from 4.77 to 4.87 (Table 1). These results are similar to those obtained by [34] in rackets from *Opuntia ficus indica*; both they and [35] found values of 4.7 and 4.64 for *Opuntia dillenii* and *Opuntia ficus indica* cladodes, respectively. Despite the similar pH in different species of cacti, our study demonstrated that cladodes have a slightly high pH compared to the pH 4.3 found in xique-xique fruits in the studies by [36]. In our study the central stem had a higher acidity content (0.09%) and total soluble solids (TSS) (2.71) than the vascular cylinder (0.07%) ($p < 0.05$) (Table 1). [12] presented variations of 1.50 to 1.75 and 2.75 (TSS) in the vascular cylinder and central stem of xique-xique. Still, according to the author, total soluble solids were mainly due to high sugar content.

The xique-xique presented a higher moisture content in the vascular cylinder than in the central stem ($p < 0.05$), as can be seen in Table 2. This indicates that most of the water is stored in this region. This corroborates the study by [12], which determined that the central stem of xique-xique is the most suitable to flour production because of the high solids content in this part. It may also indicate a more significant yield; the vascular cylinder would be better used in producing jams and sweets because of their high moisture content.

The high moisture content in cactus is a result of their morphological and physiological adaptations. This includes the water storage in its the stems, which

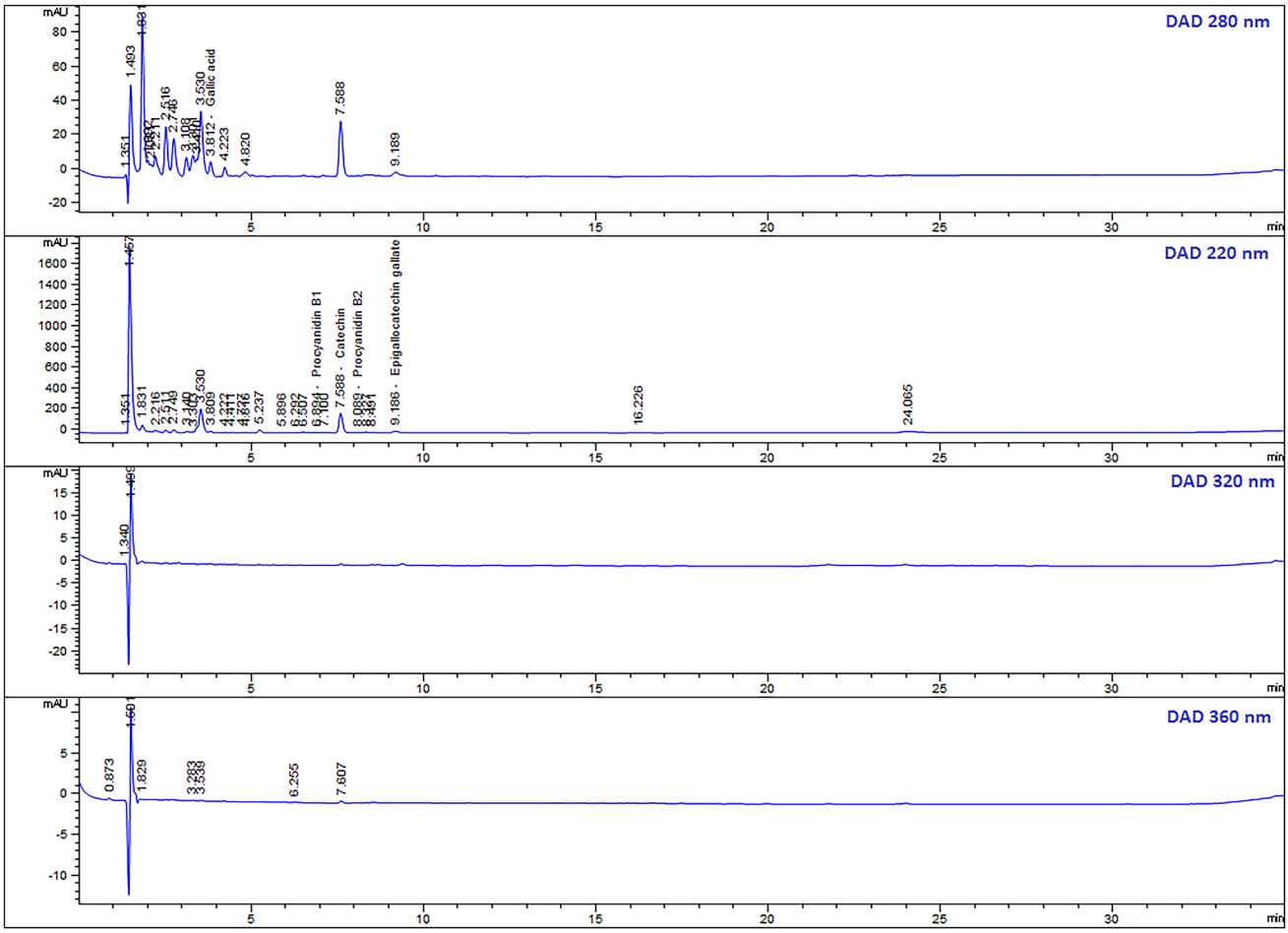

Fig. 3 Chromatograms typical from the analysis of phenolic compounds in the xique-xique sample in the dialyzed fraction

prevents loss by evaporation; furthermore, the roots absorb and accumulate dew during the night, achieving greater efficiency in water usage, enabling their survival during the dry season [6]. Values close to those found in this study have been reported for other Cactaceae, such as in the study by [35] considering *Opuntia dillenii* and *Opuntia ficus indica,* obtaining moisture values between 92.0 g/100 g to 94.0 g/100 g, respectively, which are close to the values found in the xique-xique vascular cylinder. These parameters are nutritional quality indicators; however they can appear to be reduced in vegetable arrays, varying according to soil, and the cultivation location [35].

Fructose was the main sugar in xique-xique, with the highest content in the central stem (1.07 g/100 g). Glucose, xylose, arabinose, galactose and sucrose were also identified, which are present in greater quantities in the central stem than the vascular cylinder (Table 2). [37] researched the mucilage composition extracted from *Opuntia dillenii* and identified arabinose, galactose and rhamnose as major sugars and, to a lesser extent, xylose and glucose, attributing these results to the extraction and maturity procedures of the cladodes. These sugars were also identified by [38], as they are characteristic of the Cactaceae, and are present in the mucilage. According to [39], the reason for the accumulation of these sugars is related to metabolic processes such as respiration and photosynthesis, with the growth and development of plants being influential features.

In relation the total dietary fiber, the central stem presented higher content (6.54 g/100 g), with more insoluble fiber (5.18 g/100 g) and soluble fiber (1.37 g/100 g) than the vascular cylinder (Table 2). According to [40] the dietary fiber content found in another cactus species, *Opuntia ficus-indica*, was 3.1 g/100 g, and had soluble fiber (1.00 g/100 g), being close to that found in this study [20]. When evaluating the potential prebiotic effects in vitro, of the freeze-dried juice of xique-xique cladodes, they observed that the present and available nutrients, especially the soluble fiber, could act as a substrate for the Lactobacillus. These fibers are characterized as a source of

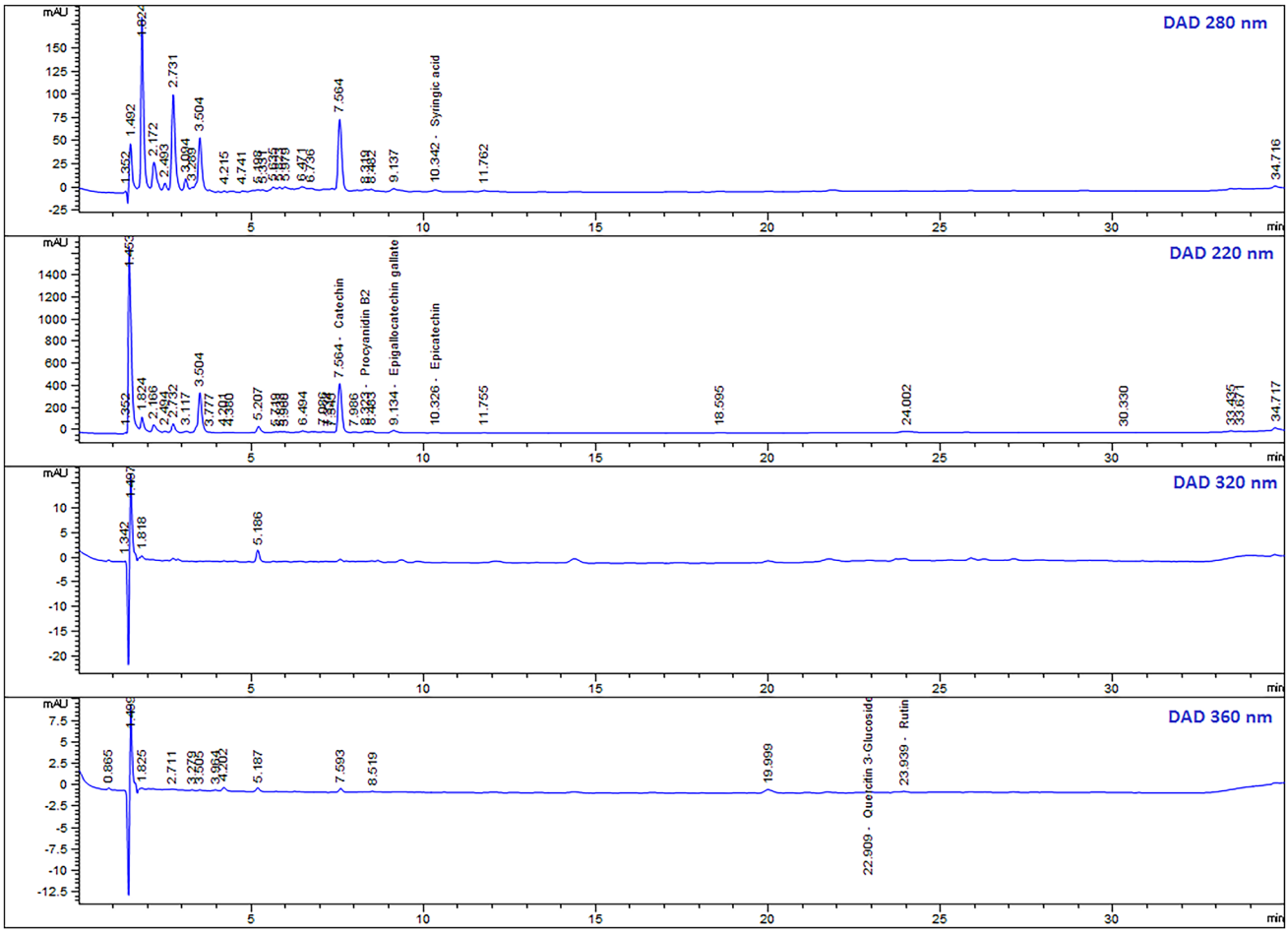

Fig. 4 Chromatograms typical of the analysis of phenolic compounds in the xique-xique sample in the non-dialyzed fraction

Table 1 Mean values of water activity, pH, acidity and total soluble solids (TSS) in xique-xique cladodes

Variables	Experimental groups	
	Vascular cylinder	Central stem
Water activity	0.97 ± 0.01[a]	0.97 ± 0.01[a]
pH	4.77 ± 0.21[a]	4.87 ± 0.21[a]
Acidity (%)	0.07 ± 0.01[b]	0.09 ± 0.02[a]
TSS	2.00 ± 0.42[b]	2.71 ± 0.23[a]

Values expressed in g/100 g fresh weight

Acidity results were expressed as% malic acid

The difference between means was evaluated using the Student's t test

TSS total soluble solids

[a,b]Mean ± standard deviation with different lowercase letters on the same line differed by Student's t test ($p < 0.05$) between samples

energy that benefit the health of the intestinal microbiota in addition to the composition of the sugars present in this cactus. In this way, they obtained results with effects similar to those found by fructooligosaccharides (FOS).

Table 2 Mean values of physico-chemical variables in xique-xique cladodes

Variables (g/100 g)	Experimental groups	
	Vascular cylinder	Central stem
Moisture	94.16 ± 0.99[a]	87.13 ± 1.64[b]
Proteins	0.49 ± 0.00[b]	0.76 ± 0.01[a]
Lipids	0.28 ± 0.00[b]	0.77 ± 0.04[a]
Sugars		
Fructose	0.31 ± 0.02[b]	1.07 ± 0.02[a]
Glucose	0.14 ± 0.02[b]	0.54 ± 0.01[a]
Xylose	0.06 ± 0.01[b]	0.37 ± 0.02[a]
Arabinose	0.06 ± 0.01[b]	0.20 ± 0.01[a]
Galactose	0.01 ± 0.01[b]	0.06 ± 0.01[a]
Sucrose	0.01 ± 0.00[b]	0.07 ± 0.01[a]
Total fiber	2.70 ± 0.01[b]	6.54 ± 0.01[a]
Insoluble fiber	1.83 ± 0.00[b]	5.18 ± 0.05[a]
Soluble fiber	0.87 ± 0.01[b]	1.37 ± 0.04[a]
Ashes	1.99 ± 0.04[a]	1.59 ± 0.00[b]

Values expressed in g/100 g fresh weight

The difference between means was evaluated using the Student's t test

[a,b]Mean ± standard deviation with different lowercase letters on the same line differed by Student's t test ($p < 0.05$) between samples

The xique-xique soluble fiber has aroused interest because of its prebiotic effect and its technological application, and can also be used in the production of several products with antioxidant action [41].

The ash levels were higher in the vascular cylinder (1.99 g/100 g) than in the central stem (1.59 g/100 g) ($p < 0.05$). When studying the chemical composition of different species of Prickly Pears [42], were able to observe that the ash content for *Opuntia dillenni* corresponded to 0.437% and for *Opuntia ficus* it was 0.392% [35], when studying the chemical composition of different species of cactus pads, obtained results referring to 1.23 g/100 g for *Opuntia dillenni* and 1.08 g/100 g for *Opuntia ficus indica*. These studies demonstrate the potential of micronutrients (minerals) present in cacti, and in particular in xique-xique as will be shown in the results of mineral profile of this study.

Mineral profile

The mineral profile of xique-xique is shown in Table 3. Potassium (308.40 mg/100 g), magnesium (182.40 mg/100 g) and calcium (145.57 mg/100 g) were the predominant minerals in the vascular cylinder and were denser than in the central stem ($p < 0.05$). The content of sodium was greater in the central stem (122.31 mg/100 g) than in the vascular cylinder (24.05 mg/100 g). According to the mineral profile presented, xique-xique is an important vegetal source of some essential minerals. The consumption of 100 g of the raw vascular cylinder *in natura* contributes to 43.42% of the recommended daily intake (RDI) for magnesium, 14.50% for calcium, and 9.1% for potassium. On the other hand, 100 g of the central stem represents 16% of the RDI for sodium, according to the recommendations of the Dietary Reference Intakes (Table 3) [43, 44]. Xique-xique has a higher concentration of magnesium than the cladodes of *Opuntia dillenni* (84.5 mg/100 g) and *Opuntia ficus indica* (94.1 mg/100 g) and the amount of calcium in xique-xique was close to that of *Opuntia dillenni* (157 mg/100 g) [35].

Table 3 Mineral profile in xique-xique cladodes

Mineral	Experimental groups		
	Vascular cylinder	Central stem	Recommendation (mg)*
Potassium	308.4 ± 3.65^{a}	101.6 ± 1.65^{b}	$3.400^{(1)}$
Magnesium	182.40 ± 2.10^{a}	167.10 ± 4.11^{b}	$420^{(2)}$
Calcium	145.57 ± 5.54^{a}	99.72 ± 4.80^{b}	$1000^{(1)}$
Sodium	24.05 ± 0.43^{b}	122.31 ± 0.72^{a}	$1500^{(1)}$
Manganese	7.71 ± 0.03^{a}	4.56 ± 0.33^{b}	$2.3^{(1)}$
Phosphor	2.94 ± 0.01^{b}	6.79 ± 0.07^{a}	$700^{(2)}$
Zinc	0.45 ± 0.01^{a}	0.22 ± 0.01^{b}	$11^{(2)}$
Iron	0.12 ± 0.01^{b}	0.26 ± 0.01^{a}	$8^{(2)}$
**Selenium	2.73 ± 0.03^{a}	2.01 ± 0.39^{b}	$0.055^{(2)}$

Values expressed in mg/100 g fresh weight

The difference between means was evaluated using the Student's t test

[a,b]Mean ± standard deviation with different lowercase letters on the same line differed by Student's t test ($p < 0.05$) between samples

*Based on National Academies of Sciences, Engineering, and Medicine 2019. Dietary Reference Intakes, 2019. Washington, DC: The National Academies Press. Based on an adult man aged 31–50 years

**Values expressed in µg/100 g fresh weight

[(1)]Adequate Intake

[(2)]Recommended Dietary Allowances

Aiming to take advantage of the xique-xique cladodes [45, 46], elaborated and researched the mineral composition of juice, jam and yogurt using the part of the vascular cylinder of this cactacea as raw material, reporting significant mineral content.

According to [45], from the recommendations of the [43], consumption of 100 ml of xique-xique juice would provide 20.65% magnesium, 10.72% potassium and 2.69% calcium. The consumption of 100 ml of xique-xique juice could provide 265.22% manganese [46] observed in xique-xique jam the presence of higher concentrations of potassium, magnesium, calcium and manganese; in this case, the processing of the xique-xique cladodes resulted in higher concentrations of the minerals, which is interesting from the nutritional point of view. According to the authors of the research, and following the recommendations of [43], the portion of jam (20 g) would provide 6.91%, 7.15%, 25.48% and 434.78% of selenium, iron, magnesium and manganese, respectively. Thus, both juice and jam, products obtained from the xique-xique cladodes, represent this cactus as a source of significant contribution to the intake of minerals, providing important nutrients for the health and maintenance of the human organism.

Amino acid profile

The present study quantified 18 amino acids present in xique-xique (Table 4). The concentration of amino acid did not differ significantly in the analyzed parts ($p > 0.05$). The amino acid arginine had a higher content followed by leucine, phenylalanine, valine, threonine and aspartic acid [47] when addressing in their review studies the fruits and cladodes of *Opuntia ficus indica* observed higher levels of proline, taurine and serine, followed by glutamine, leucine, lysine, valine, arginine, phenylalanine and isoleucine, respectively [48], found in the fruits the highest levels of proline, glutamine and serine and glutamine, valine and serine in the cladodes of *Opuntia ficus indica*. The amino acid

Table 4 Amino acid content in xique-xique cladodes

Variables (g/100 g)	Experimental groups	
	Vascular cylinder	Central stem
Aspartic acid	0.58 ± 0.12	0.51 ± 0.10
Glutamic acid	0.48 ± 0.12	0.51 ± 0.10
Serina	0.53 ± 0.12	0.34 ± 0.10
Glycine	0.53 ± 0.12	0.33 ± 0.10
Histidine	0.19 ± 0.01	0.14 ± 0.01
Arginine	0.96 ± 0.12	0.88 ± 0.10
Threonine	0.61 ± 0.12	0.39 ± 0.10
Alanine	0.49 ± 0.12	0.34 ± 0.10
Proline	0.55 ± 0.12	0.36 ± 0.10
Tyrosine	0.39 ± 0.12	0.27 ± 0.10
Valine	0.65 ± 0.12	0.39 ± 0.10
Methionine	0.10 ± 0.02	0.05 ± 0.01
Cystine	0.02 ± 0.00	0.01 ± 0.00
Isoleucine	0.46 ± 0.12	0.29 ± 0.10
Leucine	0.68 ± 0.00	0.46 ± 0.10
Phenylalanine	0.66 ± 0.47	0.32 ± 0.10
Lysine	0.55 ± 0.49	0.33 ± 0.10
Tryptophan	ND	ND

Values expressed in g/100 g of protein in dry weight

Student's t test has been used to evaluate the differences between samples

There no was difference between amino acids content of the groups

ND not detected

values presented in our study with the *Pilosocereus gounellei* cladodes expressed different values than those found in the *Opuntia* cladodes, because, although these cacti belong to the same family, they are of different genera and species.

Information about amino acid composition in Cactaceae is scarce in the literature, so the present study provides unprecedented information about the content of amino acids in the xique-xique cladodes of the northeastern semi-arid region, contributing to scientific knowledge and, consequently, the potential for its use in nutrition.

Antinutritional factors, phenolic compounds, bioaccessibility and antioxidant capacity

According to the results of the proximate composition and the physical–chemical characterization, the group of the xique-xique vascular cylinder was selected as the raw material in the elaboration of a jam, resulting in a patent and an article [46] on its use as a sub-ingredient in the production of goat yogurt. Further analyses of the vascular cylinder of the xique-xique were carried out, with the objective of analyzing its compounds for possible beneficial nutritional activity, including the determination of antinutritional factors, phenolic profile, bioaccessibility and antioxidant capacity.

Antinutritional factors

The concentration of tannin in this work was 13.45 mg/100 g, trypsin inhibitor was 0.18 mg/ g, and the phytic acid values were undetectable when less than 0.05 mg/g. The trypsin inhibitor may be considered an anti-nutrient, for being an inhibitor of the enzyme secreted by the pancreatic juice that performs the digestion of proteins. Tannins, on the other hand, may present characteristics, favorable or not to food, depending on their structure and association with phenolic compounds. Their astringency makes them undesirable from the sensory point of view and when they bind to proteins, carbohydrates and minerals they interfere with their bioavailability [49]. The composition of four varieties of *Opuntia ficus-indica* seeds were investigated by [50] and their total tannin values ranged from 4.1 mg EC/100 g to 6.6 mg EC/100 g [51] evaluated the bioactive components of the fruit of two cultivars of pigmented forage palm (*Opuntia* spp.) and obtained total tannin results between 42 to 103 mg/Kg. These results allow us to verify the presence of tannins in these cacti, whose concentration varies according to the location of the plant. The particular structural composition of the tannin present and which predominant characteristic it presents to these cactaceas was not identified. Heating can be a way to inactivate these compounds, as the trypsin inhibitor is denatured with heat treatment, just as phytic acid can undergo dephosphorylation with heating [49].

The effects of tannins on human health are still questionable because although they bind to some components and reduce their bioavailability, they also have a strong antioxidant action. In this way, the inactivation of the trypsin inhibitor is desirable, while the reduction of tannins in xique-xique requires further study.

Profile and bioaccessibility of phenolic compounds

The phenolic compound profile for the xique-xique vascular cylinder is shown in (Table 5). Among the group of identified flavonoids, catechin, epicatechin, epigallocatechin gallate, procyanidin B1, procyanidin B2, quercetin-3-glucoside, rutin and kaempferol glucoside are listed, with catechin (77.13 mg/100 g) and epigallocatechin gallate (328.23 mg/100 g) having the highest concentrations. One of the main catechin-rich foods found in the literature is green tea, and in the study by [52] maximum values of 3799 mg/100 g are reported for epigallocatechin gallate, and 375 mg/100 g for catechin. Other foods known to be rich in catechins are grapes and their derived beverages, such as wines. In the work of [53], wines from different countries

Table 5 Profile of phenolic compounds in xique-xique vascular cylinder

Phenolic compounds	Xique-xique vascular cylinder			
	Profile	*Dialyzed fraction*	*Non-dialyzed fraction*	Bioaccessibility %
Flavanols				
Catechin	77.13 ± 1.52	2.64 ± 0.05	17.19 ± 0.35	13.31
Epicatechin	1.89 ± 0.03	ND	0.03 ± 0.01	ND
Epigallocatechin gallate	328.23 ± 7.64	0.34 ± 0.01	1.15 ± 0.02	22.82
Proanthocyanidins				
Procyanidin B	ND	0.04 ± 0.01	ND	ND
Procyanidin B	ND	0.04 ± 0.01	0.51 ± 0.01	7.27
Flavonols				
Quercetin 3-glucoside	14.97 ± 0.30	ND	0.11	ND
Rutin	4.80 ± 0.10	ND	0.03 ± 0.01	ND
Kaempferol glucoside	2.54 ± 0.05	ND	ND	ND
Phenolic acids				
Gallic	ND	0.35 ± 0.01	ND	ND
Syringic	14.74 ± 0.29	ND	0.07 ± 0.01	ND

Values expressed in mg/100 g dry weight

Bioaccessibility (%) = (BC dialyzed fraction/BC non-dialyzed fraction) × 100

ND not detected

and different grape varieties were analyzed, with catechin values ranging from 26 to 160 mg/L.

The antioxidant benefits present in some foods (such as green tea) are attributed to the presence of catechins and epigallocatechin gallate in their composition. A number of studies have attributed antioxidant, anticarcinogenic, antimicrobial, antiviral, anti-inflammatory and antidiabetic properties to catechins [54]. Epigallocatechin gallate is considered the most abundant, active and commonly used catechin, and presents the following quality indicators; it is a catechin and also has antioxidant, anti-inflammatory, hypolipidemic and anti-hypoglycemic properties [55].

Among the identified flavonoids, quercetin-3-glucoside showed the highest result (14.97 mg/100 g), followed by rutin (4.80 mg/100 g) and kaempferol 3-glucoside (2.54 mg/100 g). Gallic acid and syringic acid were also among the phenolic acids identified; however only syringic acid had a significant value in the profile (14.74 mg/100 g), while gallic acid was only expressed in the *IN* (dialysed) fraction. According to the in vivo studies by [56], gallic acid has antioxidant, antihyperglycemic and antilipidemic effects, in addition to hepatoprotection effect [57].

The results for the bioaccessibility of the phenolic compounds identified in the xique-xique vascular cylinder are shown in Table 5. Catechin (13.31%), epigallocatechin gallate (22.82%), and procyanidin B (7.87%) were still bioaccessible after going through the gastrointestinal tract simulation conditions. Some compounds in this study were not bioaccessible, such as epicatechin, quercetin-3-glucoside, rutin, kaempferol 3-glucoside, and syringic acid. According to [58], non-bioaccessible compounds are also important because they present positive effects on correct bowel functioning, and they also may display inhibitory effects on intestinal microbiota.

In general, bioaccessibility is the feed content which is released from the matrix to the intestinal tract and which is available for absorption [59]. The changes which occur during this phase are evaluated through an in vitro gastrointestinal simulation because a number of changes arise during the gastric and intestinal stages, and various factors are involved in these changes such as the array of food, pH, temperature, enzymes, inhibitors or enhancers of absorption, among others [60, 61].

The number of the compounds released in the dialyzed and non-dialyzed phases was less in this study than that presented in the original composition. The changes between the phases showed that the amount of phenolic acid from the dialyzed stage was inferior to the non-dialyzed stage, which, according to [60], indicates the incomplete release or degradation of these compounds. Also, the additional extraction time and the effect of intestinal digestion (enzyme action) facilitated the release of phenolics bound to the array. Furthermore, the change in the *in-phase* to the *out-phase* values occurred because of the transition from gastric acid to the intestinal alkaline system [62].

Antioxidant capacity

Antioxidants were evaluated in the present work regarding their inhibition activity of the DPPH radical, being found to be 572.96 μM of TEAC/100 g for the xique-xique vascular cylinder. In the FRAP method, the antioxidant activity of vascular cylinder was found as 1912.95 μM of TEAC/100 g, being higher compared to DPPH method.

The content of flavonoids found in foods may exert a significant antioxidant capacity, due to the flavonoid structure. As well as the correlation between the content of phenolic compounds and the antioxidant activity measured by the FRAP and DPPH methods [63]. The values of the antioxidant capacity of the xique-xique can thus be justified by the presence of the phenolic compounds present in this matrix, for example, the catechin and the epigallocatechin gallate expressed in significant quantities in the profile of the phenolic compounds extracted in the vascular cylinder of the xique-xique.

Conclusion

Xique-xique cladodes present important nutrients such as a significant content of soluble and insoluble total fiber and of minerals, especially calcium, magnesium, selenium, and zinc in the vascular cylinder. The soluble fiber content shown in the vascular cylinder composition of xique-xique points to its potential in technological application and in the development of new products, suggesting further investigations into its functional potential. Thus, the profile of phenolic compounds and the study of bioaccessibility, in addition to mineral content, enable elucidation of the antioxidant capacity of this cactacea.

Based on our study, it is possible to perceive the xique-xique as an alternative food of great nutritional importance. The study contributes with information for the development of derived products, using the xique-xique as raw material. There remains a need for further studies on its use as a raw material to ensure food quality and safety, especially for human consumption.

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Declarations

Conflict of interest The authors declare that there is no conflict of interest.

References

1. M.C. Pereira, R.S. Steffens, A. Jablonski, P.F. Hertz, A.O. Rios, M. Vizzotto, S.H. Flôres, Characterization, bioactive compounds and antioxidant potential of three Brazilian fruits. J. Food Compost. Anal. **29**, 19–24 (2013)
2. P. Ortega-Baes, S. Sühring, J. Sajama, E. Sotola, M. Alonso-Pedano, S. Bravo, H. Godínez-Alvarez, H. Diversity and conservation in the cactus family. in *Desert Plants* (2010), pp. 157–173
3. E.M.S. Ribeiro, M.V. Meiado, I.R. Leal, The role of clonal and sexual spread in cacti species dominance at the Brazilian Caatinga. Gaia Sci. **9**(2), 27–33 (2015)
4. J.G.M. Silva, G.F.C. Lima, E.M. Aguiar, M.M.T. Rêgo, P. gounellei e Mandacaru na alimentação animal. (2013) Natal: EMPARN.
5. R.M.F. Magalhães, M.S.S. Carneiro, A.B. Castro, R.L. Edvam, E.S. Pereira, S.F. Silva, J.N.C. Torreão, Nutritional quality of Pilosocereus gounellei using different spine removal and plant storage methods. Arq. Bras. Med. Vet. Zootec **70**(6), 1988–1996 (2018)
6. C.M. Lucena, R.F.P. Lucena, G.M. Costa, T.K.N. Carvalho, G.G.S. Costa, R.R.N. Alves, D.D. Pereira, J.E.S. Ribeiro, C.A.B. Alves, Z.G.M. Quirino, E.N. Nunes, Use and knowledge of Cactaceae in Northeastern Brazil. J. Ethnobiol. Ethnomedicine **9**, 1–11 (2013)
7. V.F. Kinupp, H. Lorenzi, in *Plantas Alimentícias Não-Convencionais (PANC) no Brasil: Guia de identificação, aspectos nutricionais e receitas ilustradas*. 1st edn. (Instituto Platarum de Estudos da Flora, 2014)
8. E.M.F. Chaves, R.F. Morais, R.F.M. Barros, Popular food practices using wild plants: a potential to minimize nutritional insecurity in the semiarid region of Northeast Brazil. Gaia Sci. **11**(2), 287–313 (2017)
9. E.R. Monteiro, C.A. Mangolin, A.F. Neves, G.R. Orasmo, J.G.M. Silva, M.F.P.S. Machado, Genetic structure of *Pilosocereus gounellei* (Cactaceae) as revealed by AFLP marker tp guide proposals for improvement and restoration of degraded areas in Caatinga biome. Genet. Mol. Res. **14**, 16966–16974 (2015)
10. C.M. Lucena, T.K.N. Carvalho, J.E.S. Ribeiro, Z.G.M. Quirino, A. Casas, R.F.P. Lucena, Conhecimento botânico tradicional sobre cactáceas no Semiárido do Brasil. Gaia Sci. **9**(2), 77–90 (2015)
11. A.M. Oliveira, M.O.L. Freire, W.A.V.S. Silva, M.R.A. Ferreira, P.M.G. Paiva, L.A.L. Soares, P.L. Medeiros, B.M. Carvalho, T.H. Napoleão, Saline extract of *Pilosocereus gounellei* stem has antinociceptive effect in mice without showing acute toxicity and altering motor coordination. Regul. Toxicol. Pharmacol. **95**, 289–297 (2018)
12. C.A. Almeida, R.M.S. Figueiredo, A.J.M. Queiroz, F.M.N. Oliveira, Physical and chemical characteristics of xiquexique pulps. Rev. Ciênc. Agron. **38**, 440–443 (2007)
13. R.N. Furtado, E.C.M. Filho, M.S.S. Carneiro, E.S. Pereira, M.C.P. Rogério, A.P. Pinto, *Pilosocereus gounellei* in the water supply for finishing sheep in regions of climatic vulnerability. Small Rum. Res. **173**, 88–93 (2019)
14. E.M.F. Chaves, R.F.M. Barros, Cactáceas: Recurso alimentar emergencial no semiárido. Nordeste do Brasil. Gaia Sci. **9**, 129–135 (2015)
15. V.T. Nascimento, M.A.S. Vasconcelos, M.I.S. Maciel, U.P. Albuquerque, U.P. Famine, foods of Brazil's seasonal dry forests:

ethnobotanical and nutritional aspects. Econ. Bot. **66**(1), 22–34 (2012)
16. G.E.N. Dias, I.F. Gorgonio, J.A. Ramalho, K.M. Oliveira, E.J.R.C.E. Guedes, F.C. Leite, M.F. Alves, J.K.S. Maciel, M.F.V. Souza, C.M.B.L. Lima, M.F.F.M. Diniz, Acute oral toxicity and anti-inflammatory activity of ethanolic extract from *Pilosocereus gounellei* (Fac Weber) in rats. Int. J. Pharmacogn. Phytochem. Res. **7**, 1007–1012 (2015)
17. J.K.S. Maciel, O.S. Chaves, S.G. Brito Filho, Y.C.F. Teles, M.G. Fernandes, T.S. Assis, P.D. Fernandes, A.P. Andrade, L.P. Felix, T.M.S. Silva, N.S.M. Ramos, G.R. Silva, M.F.V. Souza, New Alcamide ant antioxidant activity of *Pilosocereus gounellei* A. Weber ex K. Schum. Bly. Ex Rowl (Cactaceas). Molecules **21**(1), 11 (2016)
18. G.A. Sousa, I.S. Oliveira, F.V. Silva-Freitas, A.F.S.C. Viana, B.P.S. Neto, F.V.M. Cunha, R.L.G. Gonçalves, A.C.M.L. Filho, M.P.M. Amaral, R.C.M. Oliveira, P.D. Fernandes, J.K.S. Maciel, T.M.S.S. Silva, M.F.V. Sousa, F.A. Oliveira, Gastroprotective effect of ethanol extracts of cladodes and roots of *Pilosocereus gounellei* (A. Weber ex K. Schum.) Bly. Ex Rowl (Cactaceae) on experimental ulcer models. J. Ethnopharmacol. **28**, 100–108 (2018)
19. P.O.A. Assis, G.C.B. Guerra, D.F.S. Araújo, L.F.L.I. Andrade, A.A. Araújo, R.F.J. Araújo, T.G. Carvalho, M.F.V. Souza, G.S.C. Borges, M.S. Lima, F.R.L. Rolim, R.A.V. Rodrigues, R.C.R.E. Queiroga, Intestinal anti-inflammatory activity of xique–xique (*Pilosocereus gounellei* A. Weber ex K. Schum. Bly. Ex Rowl) juice on acetic acid-induced colitis in rats. Food Funct. **10**, 7275–7290 (2019)
20. T.S. Ribeiro, K.B. Sampaio, F.N.D.D. Menezes, P.O.A. Assis, M.S. Lima, M.E.G. Oliveira, R.Q.R.E. Queiroga, In vitro evaluation of potential prebiotic effects of a freeze-dried juice from *Pilosocereus gounellei* (A. Weber ex K. Schum. Bly. Ex Rowl) cladodes, an unconventional edible plant from Caatinga biome. 3 Biotech **10**(10), 1–9 (2020). https://doi.org/10.1007/s13205-020-02442-8.
21. AOAC. Association of Official Analytical Chemists, *Official Methods of Analysis*, 19th edn. (AOAC, Washington, 2016).
22. J. Folch, M. Lees, G.H. Sloane-Stanley, A simple method for the isolation and purification of total lipids from animal tissues. J. Biol. Chem. **226**, 497–509 (1957)
23. A. Sluiter, B. Hames, R. Ruiz, C. Scarlata, J. Sluiter, D. Templeton, D. Crocker, Determination of structural carbohydrates and lignin in biomass. Anal. Methods **1617**, 1–16 (2008)
24. P.F. Ávila, M.B.S. Forte, R. Goldbeck, Evaluation of the chemical composition of a mixture of sugarcane bagasse and straw after different pretreatments and their effects on comercial enzyme combinations for the production of fermentable sugars. Biomass Bioenergy. **116**, 180–188 (2018)
25. J.A. White, R.J. Hart, J.C. Fry, An evaluation of the Waters Pico-Tag system for the amino-acid analysis of food materials. J. Automat. Chem. **8**(4), 170–177 (1986)
26. S.R. Hagen, B. Frost, J. Augustin, Precolumn phenylisothiocyanate derivatization and liquid chromatography of aminoacids in food. J. Assoc. Off. Anal. Chem. **72**(6), 912–916 (1989)
27. M. Latta, M. Eskin, A simple and rapid colorimetric method for phytate determination. J. Agric. Food Chem. **28**(6), 1313–1315 (1980)
28. J.J. Rackis, J.E. McGhee, M. Kakade, G. Puski, Problems encountered in measured trypsin inhibitor activity of soy flour. Report of a collaborative analysis. Cereal Sci. Today. **19**(11), 513–515 (1974)
29. M.C.P. Dutra, L.L. Rodrigues, D. Oliveira, G.E. Pereira, M.S. Lima, Integrated analyses of phenolic compounds and minerals of Brazilian organic and conventional grape juices and wines: validation of a method for determination of Cu Fe and Mn. Food Chem. **269**, 157–165 (2018)
30. C.V.S. Padilha, G.A. Miskinis, M.E.A.O. Souza, G.E. Pereira, D. Oliveira, M.T. Bordignon-Luiz, M.S. Lima, Rapid determination of flavonoids and phenolic acids in grape juices and wines by RP-HPLC/DAD: method validation and characterization of commercial products of the new Brazilian varieties of grape. Food Chem. **228**, 106–115 (2017)
31. P.M.L. Oliveira, A.M. Dantas, A.R.S. Morais, L. Gibbert, C.C.H. Kruger, M.S. Lima, M. Magnani, G.S.C. Borges, Juá fruit (Ziziphus joazeiro) from Caatinga: a source of dietary fiber and bioaccessible flavanols. Food Res. Int. **129**, 108745 (2020). https://doi.org/10.1016/j.foodres.2019.108745
32. W. Brand-Williams, M.E. Cuvelier, C. Berset, Use of a free radical method to evaluate antioxidant activity. LWT-Food Sci. Technol. **28**, 25–30 (1995)
33. M.S.M. Rufino, R.E. Alves, E.S. Brito, S.M. Morais, C.G. Sampaio, J. Pérez-Jiménez, F.D. Saura-Calixto, Comunicado técnico online. Metodologia Científica: Determinação da Atividade Antioxidante Total em Frutas pelo Método de Redução do Ferro (FRAP) (2006) ISSN 1679–6535.
34. M.C. Rouhou, S. Abdelmoumen, S. Thomas, H. Attia, D. Ghorbel, Use of green chemistry methods in the extraction of dietary fibers from cactus rackets (*Opuntia ficus* indica): structural and microstructural studies. Int. J. Biol. Macromol. **116**, 901–910 (2018)
35. L.P. Méndez, F.T. Flores, J.D. Martín, E.M.R. Rodríguez, C.D. Romero, Physicochemical characterization of cactus pads from *Opuntia dillenii* and *Opuntia ficus* indica. Food Chem. **188**, 393–398 (2015)
36. V.T. Nascimento, N.P. Moura, M.A. Vasconcelos, M.I.S. Maciel, U.O. Albuquerque, Chemical characterization of native wild plants of dry seasonal forests of the semi-arid region of northeastern Brazil. Food Res. Int. **44**, 2112–2119 (2011)
37. P. Kalegowda, A.S. Chauhan, S.M.N. Urs, *Opuntia dillenii* (Ker-Gawl) Haw cladode mucilage: physico-chemical, rheological and functional behavior. Carbohydr. Polym. **157**, 1057–1064 (2017)
38. L. Medina-Torres, E. Brito-De La Fuente, B. Torrestiana-Sanchez, R. Katthain, Rheological properties of the mucilage gum (*Opuntia ficus* indica). Food Hydrocoll. **14**, 417–424 (2000)
39. W.J. Fulkerson, D.J. Donaghy, Plant-soluble carbohydrate reserves and senescence—key criteria for developing an effective grazing management system for ryegrass-based pastures: a review. Aust. J. Exp. Agric. **41**, 261–275 (2001)
40. E. Santiago, M. Dominguez-Fernández, C. Cid, M.-P. Peña, Impact of cooking process on nutritional composition and antioxidants of cactus cladodes (*Opuntia ficus*-indica). Food Chem. **240**, 1055–1062 (2018)
41. A. Mrabet, R. Rodríguez-Gutiérrez, F. Rubio-Senent, H. Hamza, R. Rodríguez-Arcos, R. Guillén-Bejarano, M. Sindic, A. Jiménez-Araujo, Enzymatic conversion of date fruit fiber concentrates into a new product enriched in antioxidant soluble fiber. LWT **75**, 727–734 (2017)
42. E.M.D. Medina, E.M.R. Rodríguez, C.D. Romero, Chemical characterization of *Opuntia dillenii* and *Opuntia ficus* indica fruits. Food Chem. **103**, 38–45 (2007)
43. DRI. *Dietary Reference Intakes for Sodium and Potassium*. (National Academies of Sciences, Engineering, and Medicine. The National Academies Press, Washington, 2019) https://doi.org/10.17226/25353
44. S.M.T. Gharibzahedi, S.M. Jafari, The importance of minerals in human nutrition: bioavailability, food fortification, processing effects and anoencapsulation. Trends Food Sci. Technol. **62**, 119–132 (2017)
45. P.O.A.A. Carvalho, G.C.B. Guerra, G.S.C. Borges, F.F. Bezerril, K.B. Sampaio, T.S. Ribeiro, M.T.B. Pacheco, R.F. Milani, R.

Goldbeck, P.F. Ávila, M.S. Lima, M.F.V. Souza, R.C.R.E. Queiroga, Nutritional potential and bioactive compounds of xique-xique juice: an unconventional food plant from Semiarid Brazilian. J. Food Process. Preserv. (2021). https://doi.org/10.1111/jfpp.15265

46. F.F. Bezerril, M. Magnani, M.T.B. Pacheco, M.F.V. Souza, R.M.F. Figueiredo, M.S. Lima, G.S.C. Borges, M.E.G. Oliveira, T.C. Pimentel, R.C.R.E Queiroga, *Pilosocereus gounellei* (xique-xique) jam is source of fibers and mineral and improves the nutritional value and the technological properties of goat milk yogurt. LWT-Food Sci. Technol. (2021) https://doi.org/10.1016/j.lwt.2020.110512
47. S.M. Kamble, P.P. Debaje, R.C. Ranveer, S.K. Sahoo, Nutritional importance of cactus: a review. Trends Biosc. **10**(37), 7668–7677 (2017)
48. M. Aragona, E.R. Lauriano, S. Pergolizzi, C. Faggio, *Opuntia ficus*-indica (L.) Miller as a source of bioactivity compounds for health and nutrition. Nat. Prod. Res. **32**(17), 2037–2049 (2018)
49. C.M.J. Benevides, M.V. Souza, R.D.B. Souza, M.V. Lopes, Fatores antinutricionais em alimentos: revisão. Segurança Alimentar e Nutricional, Campinas **18**(2), 67–79 (2011)
50. N. Chougui, A. Tamendjari, W. Hamidj, S. Hallal, A. Barras, T. Richard, R. Larbat, Oil composition and characterisation of phenolic compounds of Opuntia ficus-indica seeds. Food Chem. **139**, 796–803 (2013)
51. R.K. Cruz-Bravo, S.H. Guzmán-Maldonado, H.A. Araiza-Herrera, J.A. Zegbe, Storage alters physicochemical characteristics, bioactive compounds and antioxidant capacity of cactus pear fruit. Postharvest Biol. Technol. **150**, 105–111 (2019)
52. C.-M. Liu, C.-Y. Chen, Y.-W. Lin, Estimation of tea catechin levels using micellar electrokineticchromatography: a quantitative approach. Food Chem. **150**, 145–150 (2014). https://doi.org/10.1016/j.foodchem.2013.10.140
53. R. Van Leeuw, C. Kevers, J. Pincemail, J.O. Defraigne, J. Dommes, Antioxidant capacity and phenolic composition of red wines from various grape varieties: specificity of Pinot Noir. J. Food Compost. Anal. **36**(1–2), 40–50 (2014)
54. V.K. Ananingsih, A. Sharma, W. Zhou, Green tea catechins during food processing and storage: a review on stability and detection. Food Res. Int. **50**, 469–479 (2013)
55. S. Legeay, M. Rodier, L. Fillon, S. Faure, N. Clere, Epigallocatechin Gallate: a review of its beneficial properties to prevent metabolic syndrome. Nutrients **7**, 5443–5468 (2015)
56. V.R. Punithavathi, P.S.M. Prince, R. Kumar, J. Selvakumari, J. Antihyperglycaemic, antilipid peroxidative and antioxidant effects of gallic acid on streptozotocin induced diabetic Wistar rats. Eur. J. Pharmacol. **650**, 465–471 (2011)
57. M.K. Rassol, E.P. Sabina, R.P. Preety, S. Patel, N. Mandal, P.P. Mishra, J. Samuel, Hepatoprotective and antioxidant effects of gallic acid in paracetamol-induced liver damage in mice. J. Pharm. Pharmacol. **62**, 638–643 (2010)
58. A. Ribas-Agustí, O. Martín-Belloso, R. Soliva-Fortuny, P. Elez-Martínez, Food processing strategies to enhance phenolic compounds bioaccessibility and bioavailability in plant-based foods. Crit. Rev. Food Sci. Nutr. **58**(15), 2531–2548 (2017)
59. F.J. Barba, L.R.B. Mariutti, N. Bragagnolo, A.Z. Mercadante, G.V. Barbosa-Cánovas, V. Orlien, Bioaccessibility of bioactive compounds from fruits and vegetables after thermal and nonthermal processing. Trends Food Sci. Technol. (2017). https://doi.org/10.1016/j.tifs.2017.07.006
60. J. Bouayed, L. Hoffman, T. Bohn, Total phenolics, flavonoids, anthocyanins and antioxidant activity following simulated gastrointestinal digestion and dialysis of apple varieties: bioaccessibility and potential uptake. Food Chem. **128**, 14–21 (2011)
61. F. Saura-Calixto, J. Serrano, I. Goñi, Intake and bioaccessibility of total polyphenols in a whole diet. Food Chem. **101**, 492–501 (2007)
62. D. Tagliazucchi, E. Verzelloni, D. Bertolini, A. Conte, In vitro bio-accessibility and antioxidant activity of grape polyphenols. Food Chem. **120**, 599–606 (2010)
63. N. Zeghada, E. Ahmed, A. Belkhirid, Y.V. Heyden, K. Demeyer, Antioxidant activity of *Vitis vinifera*, *Punica granatum*, *Citrus aurantium* and *Opuntia ficus* indica fruits cultivated in Algeria. Heliyon. (2019). https://doi.org/10.1016/j.heliyon.2019.e01575