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Nutritional potential and bioactive compounds of xique-xique juice: An unconventional food plant from Semiarid Brazilian

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Abstract

Xique-xique (*Pilosocereus gounellei*) considered an unconventional species, is a cactus popularly consumed by local populations of Semiarid Brazilian and the cladodes are used in food preparations. The objective of this study was to elaborate juice with cladodes xique-xique and to characterize its nutritional composition, determine the bioaccessibility of phenolic compounds, and antioxidant activity. The juice presented relevant soluble fibers (0.55 g/100 ml) and minerals contents. Nineteen phenolics compounds were identified in xique-xique juice. Flavanols were the major class of phenolic compounds with epigallocatechin gallate (14.52 mg/ml) and catechin (4.71 mg/ml). After the gastrointestinal digestion catechin (16.49%), epigallocatechin gallate (23%), procyanidin B1 (27.81%) and B2 (15.79%), gallic (50.74%), and caffeic (17%) acids were bioaccessible, demonstrating a strong correlation with high antioxidant activity by ORAC and FRAP assay. This research demonstrates xique-xique juice is a new source of intake of minerals, fibers and phenolic compounds, and that it also aggregates value to an underexploited specie.

Practical applications

Xique-xique (*Pilosocereus gounellei*) considered an unconventional species is a cactus popularly consumed by local populations of Semiarid Brazilian, and the cladodes are used in food preparations. Despite the widespread use of xique-xique by the communities of the Brazilian semi-arid region, still, there is no scientific data on the nutritional composition of xique-xique cladode juice. The juice represents an alternative source for the consumption of fiber, minerals, and bioaccessible phenolic compounds, since it presents a variety of phenolic compounds that contribute to the antioxidant capacity. Furthermore, it can be used for application in the food industry in the manufacture of different drinks. The elaboration of products using unconventional food plants, rich in nutrients and bioactive compounds, could be a health benefit and add value to local resources.

1 | INTRODUCTION

Brazil is one of the countries in the world with the majority biodiversity of edible plant species distributed in the different biomes (Frankelin et al., 2018). Unconventional and underutilized species are being studied, defined as food species that have one or more parts with food potential and no common use, and can include native, cultivated or spontaneous plants (Kinupp & Lorenzi, 2014). These species that are consumed by a limited part of the population, mostly by communities, are restricted to specific area (Frankelin et al., 2018).

The literature reports various studies with unconventional species demonstrating their nutritional potential and bioactive compounds; for example, species belonging to Cactaceae is already recognized by the presence of bioactive compounds, like phenolic acids and flavonoids (Agostini-Costa, 2020; Gonçalves et al., 2015), such as *Opuntia* (Aruwa et al., 2019; Bakari et al., 2017; Boutakiout et al., 2018; Santiago et al., 2018) and xique-xique (*Pilosocereus gounellei*) (Maciel et al., 2016; Silva et al., 2018).

P. gounellei (A. Weber ex K. Schum. Bly. ex Rowl) (Figure 1a), known as "xique-xique," is columnar cactus endemic to the Brazilian semi-arid region with a wide distribution in the Caatinga, being found in sandy-stony soils (Almeida et al., 2007).

In addition to natura fruit (Figure 1b) consumption (Lucena et al., 2013), xique-xique cladodes have been used by local communities to develop various products such as biscuits, cakes, sweets, bakery products, flour for preparing couscous, and are also in consumed cooked or roasted (Almeida et al., 2007; Nascimento et al., 2012). Its use has been registered in folk medicine to treat prostate inflammation, jaundice, hyperglycemia, and injuries (Dias et al., 2015). Studies have reported the presence of phytosterols, triterpenes. and phenolic compounds in xique-xique (Maciel et al., 2016; Nascimento et al., 2012).

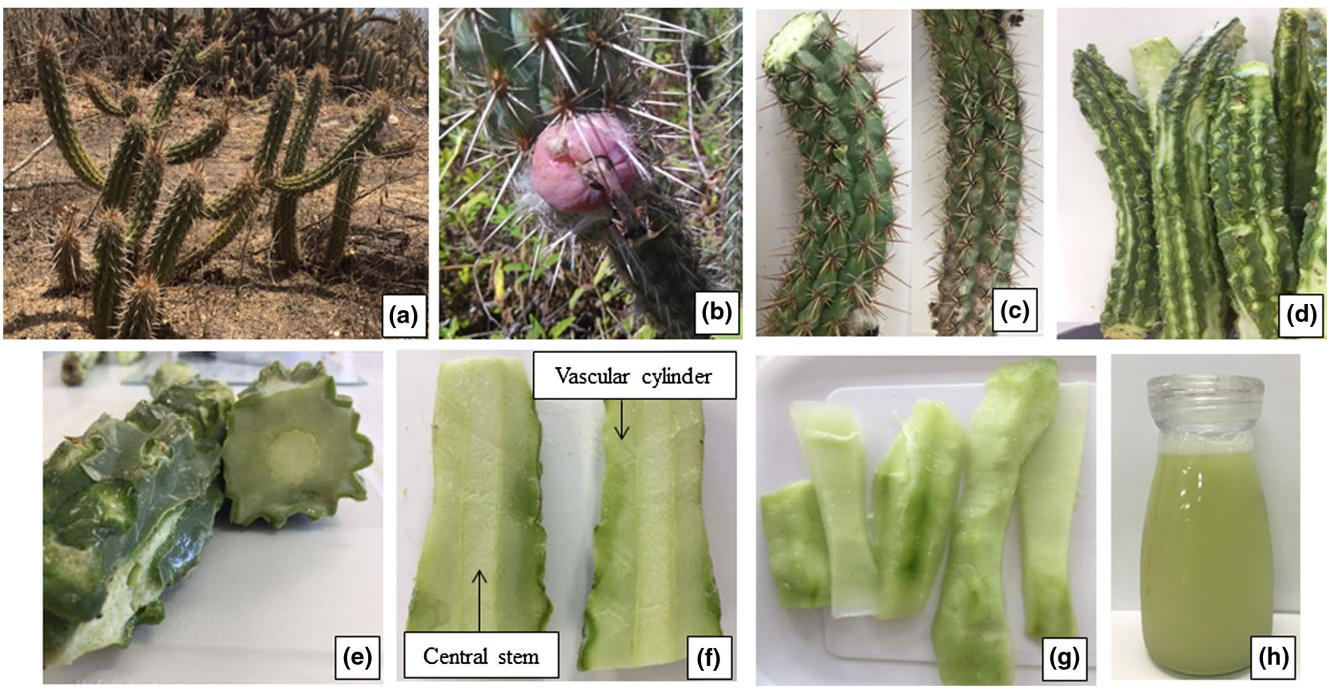

FIGURE 1 Xique-xique structures and cladode juice (a) *Pilosocereus gounellei* (xique-xique); (b) Xique-xique fruit; (c) Cladodes with thorns; (d) Cladodes without thorns; (e) Cladode cross section; (f) Horizontal section showing vascular cylinder and the central stem; (g) Vascular cylinder without central stem; (h) Xique-xique cladode juice (personal archive)

Recent study from our group showed that the ingestion of xique-xique juice (Figure 1h) had a protective effect on intestinal inflammation, decrease in pro-inflammatory markers, and oxidative stress in an animal model of inflammatory bowel disease. These effects were attributed to the phenolic compounds and fibers present in juice (Assis et al., 2019). Other studies demonstrated the significant gastroprotection of the ethanolic extracts from cladodes and roots of xique-xique on experimental ulcer models (Sousa et al., 2018).

Despite the widespread use of xique-xique by the communities of the Brazilian semi-arid region, still, there is no scientific data on the nutritional composition of xique-xique cladode juice. Therefore, this study aimed to develop xique-xique juice and characterize its nutritional properties, phenolic compounds, antioxidant potential, and evaluate the bioaccessibility of phenolic compounds and their antioxidant potential.

2 | MATERIALS AND METHODS

2.1 | Chemical products and reagents

Pepsin, α-amylase, pancreatin, glycodeoxycholate, taurodeoxycholate, and taurocholate were purchased from Sigma-Aldrich (St. Louis, Missouri, USA). Hydrochloric acid (HCl) (37% w/w) and methanol were purchased from Neon (São Paulo, Brazil).

Hydroxy-2,5,7,8-tetramethylchroman-2-carboxylic acid (Trolox) was purchased 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).

Standards of phenolic compounds for high-performance liquid chromatography (HPLC) were purchased from Sigma-Aldrich (St. Louis, MO, USA). Sugar standards were purchased from Sigma-Aldrich (St. Louis, MO, USA), and the mineral standards were purchased from Specsol (Quimlab, Jacareí, Brazil) and Merck (Darmstadt, Germany).

2.2 | Plant material

Xique-xique (*P. gounellei*) cladodes were collected in a private growing area located in Boa Vista, Paraíba, Brazil (latitudinal −7.16762352 and longitudinal −36.1432815) in three lots in November 2016. The cladodes were selected considering their physical integrity and the spines were removed upon collection. Botanical identification was performed by Prof. Dr. Leonardo Person Felix (Center for Agricultural Sciences Herbarium, Federal University of Paraíba (CCA/UFPB), and the certified species was deposited in the Prof. Jaime Coelho Morais Herbarium (CCA/UFPB) under voucher no. 17.562. 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).

2.3 | Juice extraction

Xique-xique cladodes were washed in water and sanitized with chlorine water under immersion of 100 ppm for 15 min for the treatment and subsequent removal of their skins.

To obtain the juice (Figure 1h), the central stem was removed and the vascular cylinder of the xique-xique (Figure 1g) was utilized and taken to a MecVal horizontal pulper, filtered in a sieve (20 mesh) and then stored in sterile glass bottles at −20 ± 2°C.

One produced lot of juice was lyophilized (LIOBRAS L101), which remained in the drying process for 24 hr at the average condenser temperature of −40 ± 1°C under vacuum until the samples were free of apparent moisture. The materials were then packed and stored at −20°C.

2.4 | Nutritional composition

The following analytical determinations were performed using xique-xique juice, according to the Association of Official Analytical Chemistry Methods – AOAC (2016): moisture; protein by the Micro Kjeldahl method; ash; total, soluble and insoluble fibers, and lipids according to Folch, Less-Stoane, and Stanley (1957).

2.5 | Determination of sugars

Sugars were determined to follow the methodology of Ávila et al. (2018). Thus, the xique-xique juice was filtered through a nylon syringe filter (0.22 μm), where 50 μl of the sample was injected and the profile was analyzed by ion exchange chromatography pulsed with amperometric detection (HPLC-PAD), using a Dionex chromatograph (Sunnyvale, USA) equipped with the CarboPac PA-1 column (4 × 250 mm), a CarboPac PA-1 guard column (4 × 50 mm), GP50 pump, ED40 electrochemical detector, and PEAKNET software. The mobile phases used were: A (50 mMol sodium hydroxide (NaOH)) and B (500 mMol sodium acetate (NaOAc) + 50 mMol NaOH) eluted at a rate of 1 ml/min of mobile phase with a run time of 40 min. The linear gradient was performed with 50% mobile phase A and 50% D (water) as an initial condition for 25 min. A column cleaning was then performed from 25 to 35 min using 62% B and 38% A, and the initial condition was repeated (50% A and 50% D) from 35 to 40 min (Stabilization Period for the next race). Identification of the compound peaks was performed by comparison with 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). Results were expressed as mg/100 ml of juice.

2.6 | Determination of minerals

Dry digestion (ashes) was used as the sample preparation method (AOAC, 2016). For this purpose, a lyophilized xique-xique juice (0.50 g) sample was heated on a heating plate (IKA, Staufen, Germany) and incinerated in a muffle (Fornitec, São Paulo, Brazil) for 10 hr at 450°C. An inductive coupling plasma emission spectrometer (ICP OES 5,100 VDV, Agilent Technologies, Tokyo, Japan) was used for the quantification of the inorganic elements. The optimized operating conditions of ICP OES were: plasma power, 1.20 kW; argon flow, 12.0 L/min; auxiliary argon flow, 1.0 L/min; mist flow, 0.7 L/min; number of replicates, 3. Analytical curves for the minerals were prepared from 10 mg/100 ml (Specsol - Quimlab, Jacareí, Brazil) and 1,000 mg/100 ml (Titrisol - Merck, Darmstadt, Germany) analytical standard dilutions with correlation coefficients (r) greater than 0.9999. Results were expressed as mg/100 ml of juice.

2.7 | Physical and chemical parameters

Color parameters (*L*, *a**, *b**) were established in the CIE system, using a Minolta CR-400 Chroma-meter (Konica Minolta, Osaka, Japan); density by pycnometry; acidity by titration expressed in citric acid equivalent; pH using a potentiometer (Q400AS; Quimis, Diadema, São Paulo, Brazil); water activity determined using the AQUALAB apparatus (model CX-2 Water Activity System, Washington, USA) according to the manufacturer's instructions; and soluble solids (°Brix) using a digital refractometer (Hanna, HI 96801).

2.8 | Antinutritional factors

The phytic acid content was analyzed according to Latta and Eskin (1980), while trypsin inhibitor was used according to Rackis et al. (1974). The method described by AOAC (2016) was followed for determining total tannins. Results were expressed as mg/100 ml of juice.

2.9 | Extraction and determination of the phenolic compounds profile

The lyophilized xique-xique juice (2.5 g) was weighed, added with 10 ml of methanol and 10 ml of acetone, and then subjected to ultrasound for 30 min. Next, it was centrifuged and the supernatant was collected. Extraction was repeated two 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).

The phenolic compounds were identified according to the methodology validated by Dutra et al. (2018), 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). The column used was a ZORBAX Eclipse Plus RP-C18 (100 × 4.6 mm, 3.5 μm) and a ZORBAX C18 pre-column (12.6 × 4.6 mm, 5 μm) (ZORBAX, USA). The temperature was 35°C and the injection volume was 20 μl of the sample, previously diluted in stage A, and filtered through a membrane of 0.45 μm (Millex Millipore, Barueri, SP, Brazil). The solvent flow was 0.8 ml/min. 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, where solvent A was phosphoric acid solution (0.1 M, pH 2.0) and solvent B was acidified with methanol with 0.5% H_3PO_4. Phenolic compound detection was carried out at 220, 280, 320, 360, and 520 nm and identification and quantification were performed by comparison with external standards.

2.10 | In vitro gastrointestinal digestion

The digestion procedure was performed in three sequential phases: oral, gastric, and intestinal, as described by Dutra et al. (2017). Aliquots of the xique-xique juice (50 ml) were mixed with 5 ml of saline solutions (2.38 g Na_2HPO_4, 0.19 g KH_2PO_4, 8 g NaCl, and 200 U L^{-1} α-amylase) in amber vials and the mixture was stirred for 10 min in a bath at 37 ± 2°C at 95 rpm for the simulation of the oral phase. Then 1 mg of pepsin was added and the system was acidified to pH 2.0 with 0.1 M HCl, incubated at 37°C with shaking at 95 rpm for 1 hr for gastric phase simulation. The mixture was then immediately cooled. The gastric phase fraction was submitted under intestinal phase conditions. Cellulose dialysis membrane segments (12.000 Da molecular weight cut) were filled with $NaHCO_3$ (0.5 M). The membrane was completely immersed in a tube until a pH of 5.0 was reached. After this, 5 ml of pancreatin (0.12 g) and bile salts (40 mg glycodeoxycholate in 1 ml saline), taurodeoxycholate (25 mg in 1 ml saline) and taurocholate (40 mg in 1 ml solution saline) were added to the tube. Samples were incubated on a shaker (95 rpm) at 37°C for 2 hr to complete the intestinal phase. In the final step, the dialysis membrane was removed and rinsed with distilled water. The dialyzed and non-dialyzed fractions were analyzed for the phenolic compound profile. Index bioaccessibility was expressed as the percentage and determined according to (Equation 1):

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

where BC dialyzed corresponds to the concentration of the phenolic compounds in the dialyzed fractions (bioaccessible fraction) and BC non-dialyzed is the concentration of the phenolic compounds in xique-xique juice.

2.11 | Antioxidant activity

The xique-xique juice (50 ml) was centrifuged at 5,500× *g*/15 min to remove possible interferents, and the supernatant was used for determining the antioxidant activity by FRAP (Ferric Reducing Ability Plasma) using the methodology described by Rufino et al. (2006) and ORAC (Oxygen Radical Absorbance Capacity) methodology adapted from Zulueta et al. (2009). The standard curve was created with Trolox and the results were expressed in μM TEAC/100 ml juice.

2.12 | Statistical analysis

All analyses were performed in triplicate. The data are expressed as mean ± standard deviation (*SD*). Correlations were calculated using Pearson's correlation (*r*). The difference between antioxidant activities were evaluated using the Student's *t*-test. Statistical analyses were performed using GraphPad Prism 5.0 software (GraphPad Software Inc., San Diego, CA, USA).

3 | RESULTS AND DISCUSSION

3.1 | Nutritional composition

Table 1 presents the nutritional composition of the xique-xique juice. The high ash content in xique-xique juice corresponds to approximately 44.57% of the constituents on the dry basis. The ash content expresses an important result in the inorganic content, indicating the presence of minerals (Bakari et al., 2017).

The xique-xique juice has presented high fibers content, which represent of 24% of the total solids of juice, and 88% of total fibers were soluble. Some soluble fermentable fibers are prebiotic candidates, as well as other types of fibers, as long as they are selectively used by the host microbiota and promote beneficial effects (Gibson et al., 2017). Considering the nutritional importance the ingestion these components, xique-xique juice is a new source for consumption of soluble fiber. Additionally, a recent study from our group evaluated in vitro potential prebiotic effects of this freeze-dried xique-xique cladode juice. The study has shown that the juice induces stimulatory effects on the growth and metabolism of different isolated *Lactobacillus*. These effects are indicative of potential prebiotic properties. These stimulatory effects could be linked particularly by the contents of soluble fibers present in the juice (Ribeiro et al., 2020).

Glucose and fructose were the major sugars present in the juice, while galactose, sucrose, xylose, and arabinose appear in smaller amounts (Table 1). These sugar contents may be related to the growth of the plant, which depends on the storage of substances, mainly carbohydrates, which are mobilized as soluble sugars (glucose, fructose, and sucrose) and stored in various organs for growth and maintenance of the osmotic cell homeostasis (Rosa et al., 2009).

3.2 | Mineral content

Table 2 shows that the juice has high potassium and magnesium contents, as well as notable calcium content. For microminerals, manganese was majority. In general, the accumulation of some minerals is higher in the cladode than in the fruit, because fruits have a lower transpiration rate than the cladodes, and many minerals follow the greatest transpiration pull (Bañuelos et al., 2012), thus justifying high mineral content in xique-xique juice.

TABLE 1 Nutritional composition of xique-xique juice

	g/100 ml
Moisture[a]	97.42 ± 0.37
Protein[a]	0.29 ± 0.06
Lipids[a]	0.09 ± 0.01
Ash[a]	1.15 ± 0.03
Total fibers[a]	0.62 ± 0.03
Soluble fibers[a]	0.55 ± 0.02
Insoluble fibers[a]	0.07 ± 0.02
Sugars composition[b]	mg/100 ml
Glucose	10.75 ± 0.05
Fructose	7.89 ± 0.01
Galactose	2.86 ± 0.01
Sucrose	2.58 ± 0.02
Arabinose	0.56 ± 0.01
Xylose	0.25 ± 0.02

[a]Values expressed in g/100 ml fresh weight.
[b]Values expressed in mg/100 ml fresh weight.

TABLE 2 Mineral composition of xique-xique juice

Minerals	mg/100 ml	Recommendation (mg)[a]
Potassium (K)	364.49 ± 4.00	3,400[b]
Magnesium (Mg)	86.73 ± 0.81	420[c]
Calcium (Ca)	26.90 ± 0.82	1,000[b]
Sodium (Na)	19.46 ± 0.38	1,500[b]
Manganese (Mn)	6.10 ± 0.36	2.3[b]
Phosphorus (P)	2.50 ± 0.02	700[c]
Iron (Fe)	0.15 ± 0.01	8[c]
Zinc (Zn)	0.10 ± 0.01	11[c]

Note: Values expressed in mg/100 ml fresh weight.
[a]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.
[b]Adequate intake.
[c]Recommended dietary allowances.

According to the recommendations of the Dietary Reference Intakes (DRI, 2019) (Table 2), the consumption of 100 ml of xique-xique juice would supply 20.65%, 10.72%, and 2.69% of magnesium, potassium, and calcium of these dietary recommendations, respectively. For manganese, the consumption of 100 ml of xique-xique juice would provide 265.22% of the recommendation for this mineral.

Manganese is a micro-element that plays a vital role as a structural part in many enzymes, in addition to being important for normal brain functioning and proper nervous system activity throughout the body, as well as proper and normal human bone structure growth (Gharibzahedi & Jafari, 2017). High calcium, magnesium, and potassium levels are used for energy and sports drinks to maintain the mineral pool during periods of physical exhaustion (Dehbi et al., 2014), while also performing important functions in the body such as immune system health, muscle contraction, nerve transmission, and blood pressure regulation (Gharibzahedi & Jafari, 2017).

Thus, the xique-xique juice presents a relevant source vegetal for the intake of minerals, which are important for the organism, so that cladodes and juice can be used for application in the food industry in the manufacture of different drinks to provide a source of minerals.

3.3 | Physical and chemical parameters

Table 3 shows the physical and chemical parameters of the xique-xique juice. The evaluated juice presented reduced luminosity, with *L** value corresponding to 21.70, and coordinates *a** (−1.77) and *b** (4.55), indicating yellowish-green tint in the final product. The juice density was 1.01 g/ml, being in agreement with Stintzing et al. (2003), who found density values close to those detected in the present study for the cactus juices of the *Opuntia* (1.04–1.05 g/cm^3) and *Hylocereus* genera (1.03–1.04 g/cm^3) of different cultivars. The xique-xique juice was characterized by pH value (5.23) and acidity (0.07% citric acid).

The water activity of the xique-xique juice was high in this study (0.93). It is common for cactus to exhibit high moisture content and water activity, and according to Williams et al. (2014), the succulent stems of columnar cactus are the main photosynthetic organs and the pith and cortex in many columnar cactus and other succulent plants without stems are able to store large amounts of water and other important resources for use during long periods of scarcity. This may be a possible explanation for the result found in this study, since no water was added for the elaboration of xique-xique juice, so high moisture content and water activity are inherent to the cactus.

The xique-xique juice has presented content of 1.85 °Brix, similar to that found by Almeida et al. (2007), who detected variations of 1.50–1.75 °Brix in the vascular cylinder (branches and stems) of the xique-xique. However, higher values were found by Boutakiout et al. (2015) for cladode juice of two *Opuntia* species, *Opuntia ficus-indica*, and *Opuntia megacantha* (5.1–11 °Brix), varying according to species and season. It can be seen that the content of total soluble solids may vary according to the cacti genus.

TABLE 3 Physical and chemical parameters of xique-xique juice

Variables	
Color	
*L**	21.70 ± 0.12
*a**	−1.77 ± 0.01
*b**	4.55 ± 0.10
Density (g/ml)	1.01 ± 0.01
Acidity (%)	0.07 ± 0.01
pH	5.23 ± 0.01
Water activity	0.93 ± 0.01
Total soluble solids (°Brix)	1.85 ± 0.07

3.4 | Antinutritional factors

The phytic acid and trypsin inhibitor values for the xique-xique juice were not detectable when lower than 0.05 and 0.1 mg/ml, respectively.

The total tannin content found in the juice was 8.44 mg/100 ml. Tannins are secondary plant metabolites that may form a less digestible complex with dietary proteins and may bind and inhibit endogenous protein, such as digestive enzymes (Emire et al., 2013) and, on the other hand, are shown to have beneficial and therapeutic effects for health. The biological activity of the tannins is dependent on the degree of polymerization and concentration, since the plants have a diversity of this compound (Ghosh, 2015).

3.5 | Profile and bioaccessibility of phenolic compounds

Nineteen (19) compounds were identified in the xique-xique juice (Table 4). Among them, the major compounds are flavanols, which highlights epigallocatechin gallate and catechin contents. Phenolic acids are the second group of phenolic compounds, with gallic acid being the largest phenolic found. Studies have shown that gallic acid has antioxidant, anti-hyperglycemic, and anti-lipidic effects (Punithavathi et al., 2011). In turn, epigallocatechin gallate has been shown to have neuroprotective, antioxidant, and anti-inflammatory effects (Khalatbary & Khademi, 2018).

Studies with xique-xique cladodes presented phytochemical isolation by HPLC, in which kaempferol and quercetin were found (Maciel et al., 2016; Sousa et al., 2018). These compounds demonstrated antioxidant activity (Maciel et al., 2016). These compounds are also reported in other species of the Cactaceae family, such as in *Opuntia ficus-indica* and *Opuntia megacantha* cladodes (Boutakiout et al., 2018) and *Pilosocereus arrabidae* fruits (Gonçalves et al., 2015).

When exposed to gastrointestinal digestion conditions, only gallic acid (50.74%) procyanidin B1 (27.81%), epigallocatechin

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TABLE 4 Phenolic compounds and bioaccessibility of xique-xique juice

Xique-xique juice				
Phenolic compounds[a]		**Dialyzed[b]**	**Non-dialyzed[b]**	**Bioaccessibility (%)**
Flavanols				
Catechin	4.71 ± 0.09	8.74 ± 0.17	44.23 ± 0.88	16.49
Epicatechin	0.32 ± 0.01	ND	0.28 ± 0.01	ND
Epicatechin gallate	0.28 ± 0.01	ND	ND	ND
Epigallocatechin gallate	14.56 ± 0.29	5.64 ± 0.11	18.88 ± 0.38	23.00
Proanthocyanidins				
Procyanidin B1	0.04 ± 0.01	0.12 ± 0.01	0.32 ± 0.01	27.81
Procyanidin B2	0.09 ± 0.01	0.23 ± 0.01	1.22 ± 0.02	15.79
Procyanidin A2	0.10 ± 0.01	ND	ND	ND
Flavonoids				
Quercitin 3-glucoside	0.24 ± 0.01	ND	0.47 ± 0.01	ND
Rutin	0.72 ± 0.01	ND	0.22 ± 0.01	ND
Kaempferol 3-glucoside	0.18 ± 0.01	ND	0.15 ± 0.01	ND
Phenolic acids				
Gallic acid	1.74 ± 0.03	1.46 ± 0.03	1.42 ± 0.03	50.74
Caffeic acid	0.26 ± 0.01	0.07 ± 0.01	0.33 ± 0.01	17.00
Syringic acid	0.81 ± 0.02	0.05 ± 0.01	0.68 ± 0.01	7.33
Caftaric acid	0.05 ± 0.01	ND	ND	ND
Chlorogenic acid	0.26 ± 0.01	ND	0.37 ± 0.01	ND
Stilbenes				
Trans-Resveratrol	0.07 ± 0.01	ND	ND	ND
Cis-Resveratrol	0.16 ± 0.01	ND	0.34 ± 0.01	ND
Flavonones				
Naringenin	0.09 ± 0.01	ND	ND	ND
Hesperidin	1.92 ± 0.04	ND	1.88 ± 0.04	ND

Note: Values expressed in mg/100 g dry weight.

Abbreviation: ND, not detected.

[a]Methanol:acetone (1:1) extract.

[b]Xique-xique juice

gallate (23%), caffeic acid (17%), catechin (16.49%), procyanidin B2 (15.79%) and syringic acid (7.33%) were bioacessible. These results demonstrate that some compounds are bioaccessible and capable of traversing the cellulose membrane, which simulates the gastrointestinal wall in these assays.

The compounds non detected in dialyzed fraction could be due to alkaline pH values, chemical reactions, mainly oxidation and polymerization, interaction with other dietary compounds, and/or bile salts (Lucas-González et al., 2018).

Moreover, catechin, epigallocatechin gallate, procyanidin B1, procyanidin B2, gallic acid, caffeic acid, and cis-resveratrol presented major content after gastrointestinal digestion (dialyzed + non-dialyzed fraction), and this fact can be explained due to chemical changes that occur due to the rupture bonds under acidic or alkaline conditions gastrointestinal digestion as presented in other studies with cajá, seriguela, and umbu-cajá (Dutra et al., 2017) and juá (Oliveira et al., 2020).

The non-bioaccessible (non-dialyzed) compounds are nutritionally relevant given their positive effects on the proper intestinal microbiota functioning, inhibitory effects on intestinal pathogens (Ribas-Agustí et al., 2018), maintaining redox balance, and thus preventing the development of gastrointestinal diseases related to the generation of reactive oxygen species during the digestion process (Barba et al., 2017). Recent study our research group demonstrates that the intestinal anti-inflammatory activity of xique-xique juice could be fibers and phenolic compounds (Assis et al., 2019).

When exposed to gastrointestinal digestion conditions, the xique-xique juice reduced or increased the phenolic compound contents after digestion. Gallic acid presented highest bioaccessibility, followed by procyanidin B1, epigallocatechin gallate, caffeic acid, catechin, procyanidin B2, and syringic acid. These results demonstrate that some compounds are soluble and capable of traversing the cellulose membrane, which simulates the gastrointestinal wall. This fact can be explained due to cladodes having high content fiber,

TABLE 5 Antioxidant activity of xique-xique juice

	Xique-xique juice	Dialyzed fraction
FRAP	136.68 ± 8.21[b]	179.53 ± 12.30[a]
ORAC	1,070.25 ± 56.90[b]	1,358.2 ± 109.53[a]

Note: Different letters in the same line indicate significant differences in the antioxidant activity between xique-xique juice and dialyzed fraction ($p < .05$). Values expressed in μM TEAC/100 ml.

as demonstrated in this work, thus, the high amount of phenolic compounds was bound to cell wall fiber. However, trituration processes applied during juice processing can make conjugated phenolic acids more accessible to the extraction process because of the breakdown of cellular constituents (Dutra et al., 2017).

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3.6 | Antioxidant activity

According to the antioxidant capacity shown in Table 5, the xique-xique juice presented the highest antioxidant potential against the ORAC assay which is a method based on the deactivation of oxygenated radicals in comparison with the iron reduction capacity by the FRAP method. These results can be explained by the high correlation of procyanidin B1 ($r = 0.94$), procyanidin B2 ($r = 0.94$), gallic acid ($r = 0.94$), and syringic acid ($r = 0.94$), while catechin and epigallocatechin gallate ($r = 0.86$) had a moderate correlation with the ORAC method. Moreover, quercetin 3-glucoside ($r = 0.97$) had a high correlation with the FRAP method.

The bioaccessible fraction presents a higher absorption capacity of oxygenated radicals and reduction capacity of iron ions compared than to juice; this can be justified due to the higher concentrations of catechin, procyanidin B1, and procyanidin B2 present in the bioaccessible fraction when compared to juice due to the hydrolysis gastrointestinal digestion process contributing to the hydrolysis of phenolic compounds conjugated to fibers.

4 | CONCLUSION

The xique-xique juice presented a relevant content of total fibers, mainly soluble fibers, and high mineral content, such as manganese, and expressive magnesium, potassium, and calcium contents. In addition, the juice represents an alternative source for the consumption of fiber, minerals, and bioaccessible phenolic compounds, since it presents a variety of phenolic compounds that contribute to the antioxidant capacity fronts to iron ions and oxygenated radicals.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interest.

DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available on request from the corresponding author. The data are not publicly available due to privacy or ethical restrictions.

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REFERENCES

Agostini-Costa, T. S. (2020). Bioactive compounds and health benefits of Pereskioideae and Cactoideae: A review. *Food Chemistry*, *327*, 126961. https://doi.org/10.1016/j.foodchem.2020.126961

Almeida, C. A., Figueirêdo, R. M. F., Queiroz, A. J. M., & Oliveira, F. M. N. (2007). Physical and chemical characteristics of xiquexique pulps. *Revista Ciência Agronômica*, *38*, 440–443.

AOAC. (2016). *Official methods of analysis of AOAC*. International 20th edition. Author.

Aruwa, C. E., Amoo, S., & Kudanga, T. (2019). Phenolic compound profile and biological activities of Southern African Opuntia ficus-indica fruit pulp and peels. *LWT*, *111*, 337–344. https://doi.org/10.1016/j.lwt.2019.05.028

Assis, P. O. A., Guerra, G. C. B., Araújo, D. F. S., Andrade, L. F. L. I., Araújo, A. A., Araújo, R. F. Jr, & Queiroga, R. C. R. E. (2019). 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 & Function*, *2019*, 7275–7290. https://doi.org/10.1039/c9fo00920e

Ávila, P. F., Forte, M. B. S., & Goldbeck, R. (2018). 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 and Bioenergy*, *116*, 180–188. https://doi.org/10.1016/j.biombioe.2018.06.015

Bakari, S., Daoud, A., Felhi, S., Smaoui, S., Gharsallah, N., & Kadri, A. (2017). Proximate analysis, mineral composition, phytochemical contents, antioxidant and antimicrobial activities and GC-MS investigation of various solvent extracts of cactus cladode. *Food Science Technology*, *37*, 286–293. https://doi.org/10.1590/1678-457X.20116

Bañuelos, G. S., Stushnoff, C., Walse, S. S., Zuber, T., Yang, S. I., Pickering, I. J., & Freeman, J. L. (2012). Biofortified, selenium enriched, fruit and cladode from three Opuntia Cactus pear cultivars grown on agricultural drainage sediment for use in nutraceutical foods. *Food Chemistry*, *135*, 9–16. https://doi.org/10.1016/j.foodchem.2012.04.021

Barba, F. J., Mariutti, L. R. B., Bragagnolo, N., Mercadante, A. Z., Barbosa-Cánovas, G. V., & Orlien, V. (2017). Bioaccessibility of bioactive compounds from fruits and vegetables after thermal and nonthermal processing. *Trends in Food Science & Technology*, *67*, 195–206. https://doi.org/10.1016/j.tifs.2017.07.006

Boutakiout, A., Elothmani, D., Hanine, H., Mahrouz, M., Meurlay, D. L., Hmid, I., & Ennahli, S. (2018). Effects of different harvesting seasons on antioxidant activity and phenolic content of prickly pear cladode juice. *Journal of the Saudi Society of Agricultural Sciences*, *17*, 471–480. https://doi.org/10.1016/j.jssas.2016.11.005

Boutakiout, A., Elothmani, D., Mahrouz, M., & Hanine, H. (2015). Effect of season on proximate composition of cladode juice of two species of cactaceae. *International Journal of Technology Enhancements and Emerging Engineering Research*, *3*, 1–8. https://doi.org/10.1016/j.jssas.2016.11.005

Dehbi, F., Hasib, A., Ouatmane, A., Elbatal, H., & Jaouad, A. (2014). Physicochemical characteristics of Moroccan prickly pear juice (*Opuntia ficus* indica L.). *International Journal of Emerging Technology and Advanced Engineering*, *4*, 300–306.

Dias, G. E. N., Gorgonio, I. F., Ramalho, J. A., Oliveira, K. M., Guedes, E. J. R. C. E., Leite, F. C., & Diniz, M. F. F. M. (2015). Acute oral toxicity and anti-inflammatory activity of ethanolic extract from *Pilosocereus gounellei* (Fac Weber) in rats. *International Journal of Pharmacognosy and Phytochemical Research*, *7*, 1–6.

DRI. Dietary Reference Intakes for Sodium and Potassium. *National Academies of Sciences, Engineering, and Medicine 2019*. The National Academies Press. https://doi.org/10.17226/25353

Dutra, M. C. P., Rodrigues, L. L., Oliveira, D., Pereira, G. E., & Lima, M. S. (2018). 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 Chemistry*, *269*, 157–165. https://doi.org/10.1016/j.foodchem.2018.07.014

Dutra, R. L. T., Dantas, A. M., Marques, D. D. A., Batista, J. D. F., Meireles, B. R. L. D. A., de Magalhães Cordeiro, Â. M. T., Magnani, M., & Borges, G. D. S. C. (2017). Bioaccessibility and antioxidant activity of phenolic compounds in frozen pulps of Brazilian exotic fruits exposed to simulated gastrointestinal conditions. *Food Research International*, *100*, 650–657. https://doi.org/10.1016/j.foodres.2017.07.047

Emire, S. A., Jha, Y. K., & Mekam, F. (2013). Role of anti-nutritional factors in food industry. *Beverage & Food World*, 23–28.

Folch, J., Lees, M., & Sloane-Stanley, G. H. (1957). A simple method for the isolation and purification of total lipids from animal tissues. *Journal of Biological Chemistry*, *226*, 497–509.

Frankelin, M. F., Vieira, T. F., Garcia, J. A. A., Correa, R. C. G., Monteiro, A. R. G., Bracht, A., & Peralta, R. A. (2018). Phytochemical, nutritional and pharmacological properties of unconventional native fruits and vegetables from Brazil. In S. A. Petropoulos, I. C. F. R. Ferreira, & L. Barros (Eds.), *Phytochemicals in vegetables: A valuable source of bioactive compounds* (pp. 444–464). Bentham Science Publishers.

Gharibzahedi, S. M. T., & Jafari, S. M. (2017). The importance of minerals in human nutrition: Bioavailability, food fortification, processing effects and nanoencapsulation. *Trends in Food Science & Technology*, *62*, 119–132. https://doi.org/10.1016/j.tifs.2017.02.017

Ghosh, D. (2015). Tannins from foods to combat diseases. *International Journal of Pharma Research and Review*, *4*, 40–44.

Gibson, G. R., Hutkins, R., Sanders, M. E., Prescott, S. L., Reimer, R. A., Salminen, S. J., & Reid, G. (2017). Expert consensus document—The international scientific association for probiotics and prebiotics (ISAPP) consensus statement on the definition and scope of prebiotics. *Nature Reviews-Gastroenterology & Hepatology*, *14*, 491–512. https://doi.org/10.1038/nrgastro.2017.75

Gonçalves, A. S. M., Peixe, R. G., Sato, A., Muzitano, M. F., Souza, R. O. M., Machado, T. B., & Leal, I. C. R. (2015). *Pilosocereus arrabidae* (Byles & Rowley) of the Grumari sandbank, RJ, Brazil: Physical, chemical characterizations and antioxidant activities correlated to detection of flavonoids. *Food Research International*, *70*, 110–117. https://doi.org/10.1016/j.foodres.2014.10.009

Khalatbary, A. R., & Khademi, E. (2018). The green tea polyphenolic catechin pigallocatechin gallate and neuroprotection. *Nutritional Neuroscience*, *23*, 281–294. https://doi.org/10.1080/1028415X.2018.1500124

Kinupp, V. F., & Lorenzi, H. (2014). *Plantas Alimentícias Não Convencionais (PANC) no Brasil: Guia de identificação, aspectos nutricionais e receitas ilustradas*. Instituto Plantarum de Estudos da Flora.

Latta, M., & Eskin, M. (1980). A simple and rapid colorimetric method for phytate determination. *Journal of Agricultural and Food Chemistry*, *28*, 1313–1315.

Lucas-González, R., Viuda-Martos, M., Pérez Álvarez, J. A., & Fernández-López, J. (2018). Changes in bioaccessibility, polyphenol profile and antioxidant potential of flours obtained from persimmon fruit (Diospyros kaki) co-products during in vitro gastrointestinal digestion. *Food Chemistry*, *256*, 252–258. https://doi.org/10.1016/j.foodchem.2018.02.128

Lucena, C. M., Lucena, R. F. P., Costa, G. M., Carvalho, T. K. N., Costa, G. G. S., Alves, R. R. N., & Nunes, E. N. (2013). Use and knowledge of cactaceae in northeastern Brazil. *Journal of Ethnobiology and Ethnomedicine*, *9*, 1–11. https://doi.org/10.1186/1746-4269-9-62

Maciel, J. K. S., Chaves, O. S., Filho, S. G. B., Teles, Y. C. F., Fernandes, M. G., Assis, T. S., & Souza, M. F. V. (2016). New Alcamide and antioxidant activity of *Pilosocereus gounellei* A. Weber ex K. Schum. Bly. ex Rowl. (Cactaceae). *Molecules*, *21*, 1–13. https://doi.org/10.3390/molecules21010011

Nascimento, V. T., Vasconcelos, M. A. S., Maciel, M. I. S., & Albuquerque, U. P. (2012). Famine foods of Brazil's seasonal dry forests: Ethnobotanical and nutritional aspects. *Economic Botany*, *66*, 22–34.

Oliveira, P. M. L., Dantas, A. M., Morais, A. R. S., Gibbert, L., Kruger, C. C. H., Lima, M. S., Magnani, M., & Borges, G. S. C. (2020). Juá fruit (*Ziziphus joazeiro*) from Caatinga: A source of dietary fiber and bioaccessible flavanols. *Food Research International*, *129*, 108745. https://doi.org/10.1016/j.foodres.2019.108745

Punithavathi, V. R., Prince, P. S. M., Kumar, R., & Selvakumari, J. (2011). Antihyperglycaemic, antilipid peroxidative and antioxidant effects of gallic acid on streptozotocin induced diabetic Wistar rats. *European Journal of Pharmacology*, *650*, 465–471. https://doi.org/10.1016/j.ejphar.2010.08.059

Rackis, J. J., Mc Ghee, J. E., Kakade, M., & Puski, G. (1974). Problems encountered in measured trypsin inhibitor activity of soy flour. Report of a collaborative analysis. *Cereal Science Today*, *19*, 513–515.

Ribas-Agustí, A., Martín-Belloso, O., Soliva-Fortuny, R., & Elez-Martínez, P. (2018). Food processing strategies to enhance phenolic compounds bioaccessibility and bioavailability in plant-based foods. *Critical Reviews in Food Science and Nutrition*, *58*, 2531–2548. https://doi.org/10.1080/10408398.2017.1331200

Ribeiro, T. S., Sampaio, K. B., Menezes, F. N. D. D., Assis, P. O. A., Lima, M. S., Oliveira, M. E. G., Souza, E. L., & Queiroga, R. C. R. E. (2020). 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*, 1–9.

Rosa, M., Prado, C., Podazza, G., Interdonato, R., González, J. A., Hilal, M., & Prado, F. E. (2009). Soluble sugars—Metabolism, sensing and abiotic stress. *Plant Signaling & Behavior*, *4*, 388–393. https://doi.org/10.4161/psb.4.5.8294

Rufino, M. S. M., Alves, R. E., de Brito, E. S., de Morais, S. M., Sampaio, C. G., Pérez-Jiménez, J., & Saura-Calixto, F. D. (2006). Metodologia científica: Determinação da atividade antioxidante total em frutas pelo método de redução do ferro (FRAP). *Comunicado Técnico Embrapa*. *125*, 1–4.

Santiago, E. D., Domínguez-Fernández, M., Cid, C., & De Peña, M.-P. (2018). Impact of cooking process on nutritional composition and antioxidants of cactus cladodes (*Opuntia ficus-indica*). *Food Chemistry*, *240*, 1055–1062. https://doi.org/10.1016/j.foodchem.2017.08.039

Silva, A. A., Dantas, R. L., Véras, C. S., Alvez, R. E., & Silva, S. M. (2018). Physical and physicochemical characteristics, bioactive compounds and antioxidant activity of xiquexique (*Pilosocereus gounellei* (A. Weber Ex K. Schum.)) Fruit. *Semina: Ciências Agrárias*, *39*, 1969–1980. https://doi.org/10.5433/1679-0359.2018v39n5p1969

Sousa, G. A., Oliveira, I. S., Silva-Freitas, F. V., Viana, A. F. S. C., Neto, B. P. S., Cunha, F. V. M., Gonçalves, R. L. G., Lima Filho, A. C. M., Amaral, M. P. M., Oliveira, R. D. C. M., Fernandes, P. D., Maciel, J. K. S., da Silva, T. M. S., Souza, M. D. F. V., & Oliveira, F. A. (2018). 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. *Journal of Ethnopharmacology*, *218*, 100–108. https://doi.org/10.1016/j.jep.2018.02.009

Stintzing, F. C., Schieber, A., & Carle, R. (2003). Evaluation of colour properties and chemical quality parameters of cactus juices. *European Food Research and Technology*, *216*, 303–311. https://doi.org/10.1007/s00217-002-0657-0

Williams, D. G., Hultine, K. R., & Dettman, D. L. (2014). Functional tradeoffs in succulent stems predict responses to climate change in columnar cacti. *Journal of Experimental Botany*, *65*, 3005–3013. https://doi.org/10.1093/jxb/eru174

Zulueta, A., Esteve, M. J., & Frigola, A. (2009). ORAC and TEAC assays comparison to measure the antioxidant capacity of food products. *Food Chemistry*, *114*, 310–316. https://doi.org/10.1016/j.foodchem.2008.09.033

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