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Quality pattern evaluation of frozen soursop pulps: an assessment based on chemical composition and chemometric analysis

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Abstract

In the present study, the physicochemical properties and mineral composition of commercially-frozen soursop pulps from the southern region of the state of Bahia, Brazil, were evaluated. The physicochemical parameters such as soluble solids (°Brix), acidity and pH were determined according to reference methodologies. For mineral composition evaluation, samples were digested in a closed microwave system and the elements K, P, S, Ca, Mg, Fe, Cu, Zn, and Mn were quantified through inductively coupled plasma optical emission spectrometry (ICP OES). Soluble solids content ranged from 4.77 to 15.37 °Brix among the different brands of analyzed frozen pulps. Titratable acidity (equivalent to citric acid) and pH ranged from 0.49 to 1.00 and 3.61 to 4.57, respectively. Some samples presented irregularities in regard to quality pattern established in the Brazilian legislation. The following range of concentration, in μg g^{-1}, were found for essential elements: K (544-1761), P (44.3-139), S (71.8-174), Ca (24.3-77.0), Mg (47.2-132), Fe (1.40-4.05), Mn (0.32-1.22), Cu (0.30-0.88) and Zn (0.36-0.84). The use of principal component analysis (PCA) enabled a more in-depth data analysis, performing correlations between different variables and frozen pulp brands.

Keywords: *Annona muricata* L.; frozen soursop pulp; physicochemical properties; essential elements; multivariate statistics; principal component analysis.

Practical Application: The majority of recently published studies on soursop pulps only address evaluation of physicochemical properties and/or antioxidant activity. Thus, the innovative feature of this work is the assessment of quality pattern of frozen soursop pulps, correlating physicochemical properties with their mineral composition. For this, chemometric tools and a large number of samples have been used.

1 Introduction

Soursop (*Annona muricata* L.) is a native species from tropical areas of North and South America, but can also currently be found in Asia, Africa and American countries (Coria-Téllez et al., 2018; Moghadamtousi et al., 2015). In the Brazilian Northeast, the soursop is cultivated in several areas, including the southern region of the state of Bahia. Due to its sensorial qualities, the soursop fruit can be consumed in natural form or used as a commodity by the agro-industry for the manufacture of products such as juices, ice creams and frozen pulps (Badrie & Schauss, 2009; Empresa Brasileira de Pesquisa Agropecuária, 1998). Several studies have been carried out with soursop fruits (Costa et al., 2014; Espinosa et al., 2013; Jiménez et al., 2014; Passos et al., 2015; Sacramento et al., 2003; Santana et al., 2017), but the characterization of processed products such as commercial frozen pulps has yet to be widely explored.

Soursop pulp is defined as the unfermented and undiluted product with minimum total solids content, obtained from the edible part of the fruit using appropriate technological processes. Brazilian legislation establishes that the fruits used in frozen pulp production must always be healthy, clean and free of parasites and animal or vegetable waste (Brasil, 2000). The pleasant taste of the fruit makes frozen soursop pulp a product of high acceptance by consumers. Brazil's Ministry of Agriculture, Livestock, and Supply (Brasil, 2000) regulates the identity and quality pattern for soursop pulp production, aiming to ensure final product quality. According to the Brasil (2000), the minimum value for the titratable acidity of frozen soursop pulp (in citric acid, g $100g^{-1}$) must be 0.6. Regarding pH and soluble solids content, the established minimum values are 3.50 and 9.0 °Brix, respectively.

Mineral composition of the fruit pulp is another relevant factor. Some chemical elements provide essential functions to human organisms and need to be ingested from food sources since they are not synthesized by the body (McDowell, 2003). It is known that major elements Ca and P assist in bone constitution, while the macroelement Mg is involved in ATP production. The microelements Fe and Mn have a fundamental role in the respiratory process and protein metabolism, respectively (Food

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and Agriculture Organization of the United Nations, 2004; McDowell, 2003). Other elements such as K, S, Zn, and Cu also develop an important role in the maintenance of good organism function. Multi-element determination through spectrometric techniques after acid digestion procedure has often been employed for mineral composition evaluation of several fruit and fruit based-product samples (Caidan et al., 2014; Pacheco et al., 2017; Potortí et al., 2018; Rodríguez-Solana et al., 2014; Santos et al., 2016). Inductively coupled plasma optical emission spectrometry (ICP OES) is considered an efficient technique for multi-element determination due to the possibility of simultaneous analysis and low detection limits (LOD).

When aiming for data treatment obtained from a large number of samples and variables, multivariate analysis methods such as principal component analysis (PCA) are extremely useful. PCA is a chemometric tool that consists of transforming the original data matrix into new sets of loadings and scores (called principal component), which are able to describe data variability in a smaller dimensional space (Granato et al., 2018; Kumar et al., 2014). In addition, the use of PCA enables identification of relationships between variables and analyzed samples. This application has been used in the analysis of food quality as a powerful tool for sample discrimination based on chemical composition (Alves et al., 2013; Costa et al., 2019; Fidelis et al., 2017; Ribeiro et al., 2010) and physicochemical properties (Conti et al., 2018; Difonzo et al., 2019; Lu et al., 2017; Šamec et al., 2016).

Considering the economic importance and wide consumption of frozen fruit pulps in the state of Bahia (Brazil), the present study aims to evaluate the physicochemical properties and mineral composition of frozen soursop pulps marketed in the southern region of the state of Bahia. The physicochemical properties and mineral composition of frozen soursop pulps were evaluated and compared with quality standards in Brazilian legislation.

2 Materials and methods

2.1 Instrumentation

An Atago (Tokyo, Japan) DR-A1 abbe refractometer was used for determination of soluble solids content. A Hanna Instruments pH 21 Ph-meter (São Paulo, Brazil) was used to measure the pH of the samples. A closed microwave system model Mars-Xpress from CEM (North Carolina, USA) equipped with PFA (perfluoroalkoxy) vessels was employed for sample digestion procedure. A 710-ES inductively coupled plasma optical emission spectrometer – ICP OES Varian (Mulgrave, Australia), equipped with glass nebulizer, cyclonic nebulization chamber and solid-state detector was used for multi-element determination. The ICP OES torch was aligned with a 5.0 mg L^{-1} manganese standard solution and the optical system was calibrated using multi-element standard solution. Emission lines were selected considering the absence of spectral interferences and adequate sensitivity for element determination at high and low concentrations (Thompson & Barnes, 1992). The Supplementary Table S1 shows the ICP OES experimental conditions and emission lines of the elements.

2.2 Reagents and solutions

All solutions were prepared using analytical grade reagents and ultrapure water with a specific resistance of 18.2 MΩ cm^{-1} obtained from Milli-Q purification system, Millipore (Bedford, MA, USA). Sodium hydroxide P.A. from Biotec (Brazil) was used in acidity determination of the samples. Nitric acid 65% and hydrogen peroxide 30% used for sample digestion were obtained from Merck (Darmstadt, Germany). Monoelement standard solutions of 1000 mg L^{-1} from Specsol (Brazil) were used to prepare the calibration curve.

2.3 Sample collection and storage

Thirty-nine frozen soursop pulp samples from 13 different brands were purchased from local markets in the municipalities of Itabuna, Coaraci, Ilhéus and Salvador, located in the state of Bahia, Brazil. In order to evaluate variations between batches of the same sample, three samples of each brand were acquired. All samples were analyzed in triplicate (n = 3), totaling 117 samples. The samples were codified as NP, IF, E, DM, M, N, BF, S, IT, R, BJ, FN, and BP. All samples were stored in a freezer at -2 °C until analysis.

2.4 Determination of the physicochemical properties of soursop pulps

All physicochemical analyses were carried out according to reference methodologies from the Instituto Adolf Lutz (2008): soluble solids content (°Brix) was determined using a refractometer; titratable acidity was determined titrimetrically using NaOH standardized solution at 0.0998 mol L^{-1} and phenolphthalein alcoholic solution 1% (v v^{-1}) as indicator. The values were expressed in citric acid equivalent; pH was measured using a previously calibrated pH meter. All analyses were carried out in triplicate (n = 3).

2.5 Mineral composition

Approximately 4.0 g of soursop pulp was directly weighed in PFA digestion tubes and 4.0 mL of HNO_3 and 1.0 mL of H_2O_2 were added. Analytical blanks were prepared by adding 4.0 mL of HNO_3, 1.0 mL of H_2O_2 and 5.0 mL of ultrapure water. The microwave heating program was carried out in two successive steps: (i) a 15-minute ramp to reach 180 °C; (ii) 20-minute hold at 180 °C. In all steps, the oven power was kept at 1600 W. After the heating program, the vessels were cooled for 5 minutes. Digested samples were transferred to 25 mL-polypropylene flasks and filled up to 12.5 mL with ultrapure water. The elements Ca, Mg, K, S, P, Cu, Fe, Mn, and Zn were determined by inductively coupled plasma optical emission spectrometry (ICP OES). All analyses were carried out in triplicate.

2.6 Validation of analytical methodology

The analytical methodology for multi-element determination was validated considering the following figures of merit: linearity, limit of detection (LOD), limit of quantification (LOQ), precision and accuracy. The linearity was evaluated through R^2 values. LOD and LOQ values were obtained according to IUPAC

recommendations: LOD = 3σ/s e LOQ = 10σ/s, where σ is the standard deviation of the analytical blanks (n = 10) and s is the slope of the calibration curve (Thompson et al., 2002). Precision was assessed using the relative standard deviation (RSD) obtained from three (n = 3) independent determinations in each one of the analyzed samples. The method accuracy for metals determination was evaluated through analysis of SRM1515 (Apple Leaves).

2.7 Data analysis

Microsoft Excel 2013 (Microsoft, USA) was used for mathematical operations and data treatment. Statistica 7.0 software (StatSoft, USA) was used for principal component analysis (PCA).

3 Results and discussion

3.1 Principal Component Analysis (PCA)

Exploratory analysis using PCA was employed to evaluate important correlations between variables and extract relevant information from the dataset. A data matrix composed of 13 columns (referring to the physicochemical properties and mineral content) and 117 rows corresponding to frozen pulp samples were obtained (Table S2).

The initial dataset pretreatment consisted of auto-scaling the original values of variables in order to make all variables present the same weight (Correia & Ferreira, 2007; Granato et al., 2018). A previous evaluation of variable correlations (Table S3) showed a strong correlation in most of the studied variables. The criteria used for extraction of principal components (PCs) were eigenvalues ≥ 1 (Table S4) and scree test ≥ 1 (Figure S1). In this way, five PCs were extracted that explained 86.5% of the cumulative variance. The initial evaluation of loading values in the unrotated principal component matrix showed high loading values in more than one PC, indicating redundant information. As such, a VARIMAX raw rotation was performed to eliminate this redundancy (Table 1).

As can be seen in Table 1, five major PCs affecting frozen soursop pulp quality were identified. The first principal component explained more than 35% of total variance, where K, Ca, Mg and °Brix showed high loadings in this PC. As such, PC1 is characterized by sweeter samples (high °Brix values) containing a high content of major elements such as K, Ca and Mg. Other major elements such as P and S showed high loadings in the second principal component (PC2) followed by high loadings of variable TA (titratable acidity). Thus, PC2 is composed mainly of acidic pulps due to high citric acid content. PC3 and PC4 presented negative loadings of Mn and pH, respectively. As a result, these PCs are characterized by samples containing low levels of manganese and low pH. The last PC explained approximately 8% of total variance and has high Fe and Cu loadings. Thus, PC5 is characterized by samples containing high levels of minor elements such as Fe and Cu.

The score graphs are shown in Figure 1. Figure 1A (PC1 × PC2) shows the discrimination of IT, BF and three S samples due to high positive loadings. These samples were characterized by high concentrations of K, Ca, Mg and high sugar content (°Brix). The separation between two groups composed of S samples may be due to factors such as storage time or influence of different sample batches. In addition, the high content of these major elements in the sample S might indicate influence of fruit growing conditions, place of collection, and soil characteristic. On the other hand, samples NP, IF, E and DM were separated with high negative loadings and, therefore, presented a low concentration of the aforementioned variables. Samples E and S were also separated in PC2, due to high sulfur and phosphorous content. However, these samples were also discriminated according to acidity (TA) showing the acidic character of these pulps. The PC1 × PC3 graph (Figure 1B), shows a separation of BF and IT samples due to low manganese content. This behavior is also observed in S samples from different batches. Although little information is observed in PC4 (Figure 1C), it is possible to observe the separation of NP samples as a function of their high pH value. The influence of different batches can also be observed due to the separation of groups from the same sample (NP and S). The PC1 × PC5 graph (Figure 1D) showed a discrimination of some R and S samples due to high iron and copper content. In contrast, NP and IT

Table 1. VARIMAX rotated factor loadings of the principal component on variables. High loading values are highlighted in bold.

Variable	PC1	PC2	PC3	PC4	PC5
Fe	-0.034573	-0.089910	-0.018713	0.282167	**0.918602**
Cu	0.219636	0.357069	-0.145453	-0.262440	**0.767703**
Zn	0.534128	0.427529	0.544578	-0.224356	0.174464
Mn	0.204478	0.052150	**-0.919615**	0.075678	0.102492
K	**0.752325**	-0.114967	-0.496354	0.259584	0.241380
P	-0.246007	**0.662482**	0.195955	-0.373642	0.126840
S	-0.036661	**0.930799**	-0.102480	-0.001830	-0.034524
Ca	**0.930690**	-0.039001	0.010769	0.118746	0.058788
Mg	**0.955766**	0.069095	-0.194925	0.034954	0.028330
TA	0.435406	**0.632556**	0.226584	0.443317	0.214355
pH	-0.184422	0.047453	0.222564	**-0.896579**	-0.159563
°Brix	**0.743181**	0.036339	0.133086	0.570815	-0.098226
SS/TA	0.534601	-0.395176	-0.033997	0.362871	-0.235461
Total variance (%)	35.45994	20.78962	13.29450	8.87633	8.06879
Cumulative (%)	35.45994	56.24956	69.54406	78.42039	86.48917

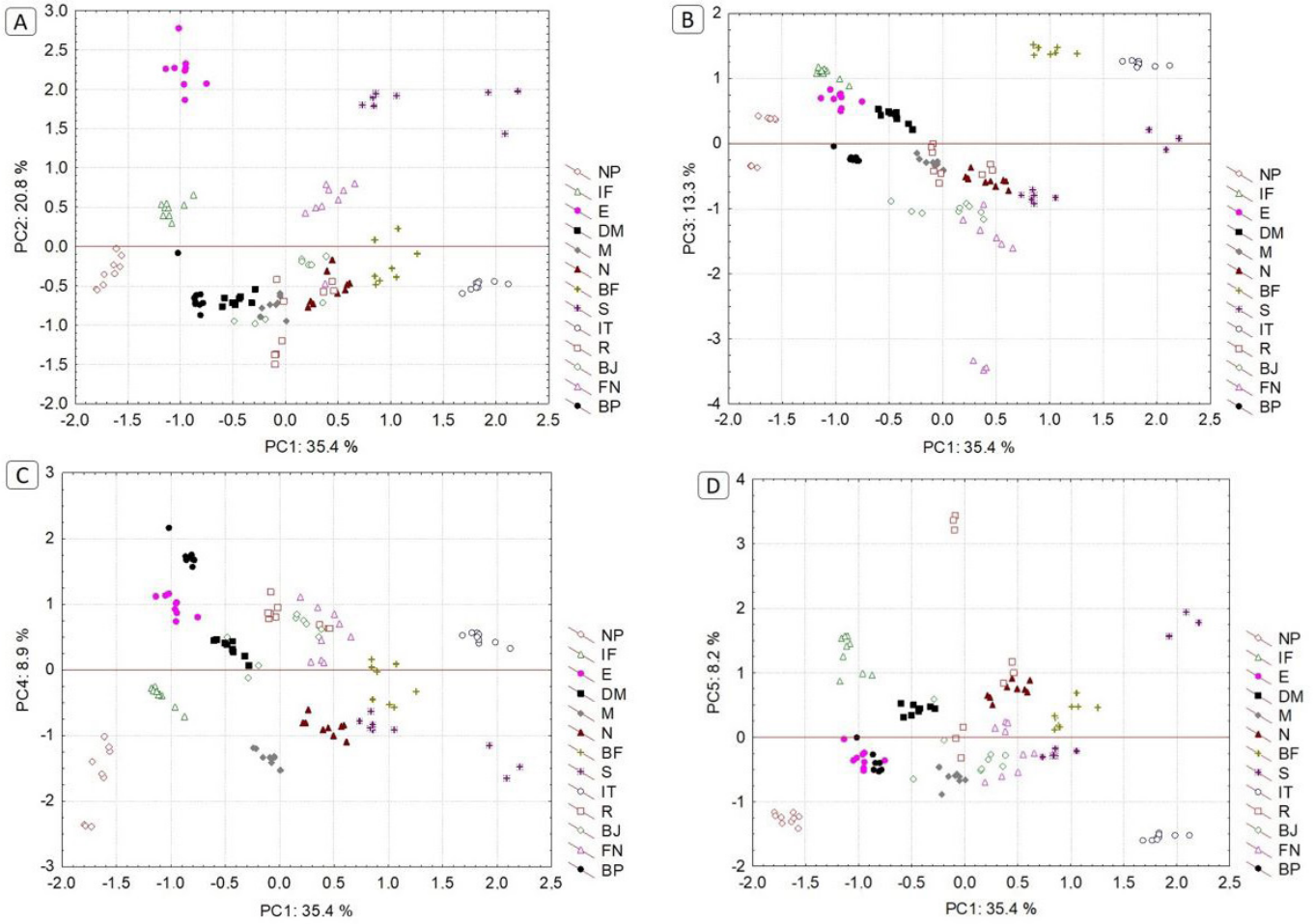

Figure 1. Score graphs composed of commercially-frozen soursop pulps based on physicochemical properties and mineral composition. (A) PC1 × PC2; (B) PC1 × PC3; (C) PC1 × PC4; (D) PC1 × PC5.

samples were discriminated by low concentrations of these minor elements. These samples also presented the influence of different batches.

3.2 Physicochemical properties of the frozen soursop pulps

After preliminary evaluation using PCA, the samples were critically evaluated based on physicochemical properties and mineral composition. Titratable acidity (TA), pH, soluble solids content (SS) and SS/TA ratio of the analyzed soursop pulps are presented in Table 2. The most significant variations were observed in the soluble solids content (°Brix) and SS/TA ratio.

Soluble solids

In general, fruits of the *Annonaceae* family present high soluble solids content with the largest fraction corresponding to soluble sugars. In the soursop fruit, sugar composition mainly includes fructose, glucose and sucrose (Padmanabhan & Paliyath, 2016; Sacramento et al., 2003). The frozen pulps from BF, R, FN, and IT brands presented the highest soluble solids contents (10.23-15.37 °Brix) and, consequently, the highest contents of these sugars. Samples NP, M and IF presented values below the permitted minimum (9.0 °Brix), according to the identity and quality standards established in Brazilian legislation (Brasil, 2000). The soluble solids content in these samples ranged from 4.77 to 7.90 °Brix and, therefore, presented irregularities. This property may vary according to factors such as genetic variety of the fruits or climatic conditions, but the possibility of water addition at the pulping stage should not be ruled out, since this practice is common in the Brazilian northeast to facilitate processing and increase product yield (Bastos et al., 1998). Alamar et al. (2016) reported that water addition in frozen guava and passion fruit pulps was the reason for high moisture and low soluble solids contents in some samples. The other soursop pulps analyzed in the present study presented soluble solids content within the Brazilian regulations.

Titratable acidity and pH

Acidity is an important indicator in taste, color and microbial stability of fruits and their products. High acidity makes the product unacceptable to the consumer even if the sugar content meets the minimum standards (Othman et al., 2014). The most predominant acid in soursop fruit is citric acid, followed by malic and ascorbic acids at lower concentrations (Espinosa et al., 2013;

Table 2. Physicochemical properties of the frozen soursop pulps. Results are expressed as average ± standard deviation (n = 9).

Brand	Physicochemical properties			
	TA (g citric acid 100 g-1)	pH	SS (°Brix)	SS/TA
NP	0.49 ± 0.03	4.57 ± 0.11	4.77 ± 1.24	6.28 ± 1.73
IF	0.83 ± 0.02	4.18 ± 0.02	7.90 ± 0.09	6.16 ± 0.38
E	1.00 ± 0.08	3.93 ± 0.01	9.63 ± 0.10	6.16 ± 0.51
DM	0.80 ± 0.03	4.05 ± 0.02	9.23 ± 0.22	5.68 ± 0.38
M	0.77 ± 0.40	4.28 ± 0.02	6.60 ± 0.09	6.65 ± 0.32
N	0.71 ± 0.05	4.20 ± 0.03	9.37 ± 0.33	8.46 ± 0.65
BF	0.90 ± 0.07	4.01 ± 0.04	10.23 ± 0.58	7.31 ± 0.57
S	0.95 ± 0.10	4.12 ± 0.02	9.77 ± 0.43	5.97 ± 0.47
IT	0.89 ± 0.01	4.03 ± 0.01	15.37 ± 0.18	11.02 ± 0.15
R	0.78 ± 0.07	3.75 ± 0.01	10.51 ± 0.29	8.55 ± 0.65
BJ	0.71 ± 0.09	3.71 ± 0.02	9.53 ± 1.26	8.62 ± 0.75
FN	0.85 ± 0.03	3.85 ± 0.06	11.21 ± 1.09	8.47 ± 1.04
BP	0.78 ± 0.08	3.61 ± 0.01	9.53 ± 0.13	7.88 ± 0.72

TA = titratable acidity; SS = soluble solids; SS/TA = soluble solids/titratable acidity ratio.

Padmanabhan & Paliyath, 2016). Sample NP presented titratable acidity below identity and quality standards (0.6 g 100 g^{-1}). This low value (0.49 ± 0.03) may be a consequence of the dilution of organic acids present in soursop due to water addition in frozen pulp processing (Alamar et al. 2016). The titratable acidity of the other investigated samples ranged from 0.71 to 1.00. Most of the analyzed frozen soursop pulps presented a pH value higher than the minimum required by regulation. Sample NP presented the highest pH value (4.57 ± 0.11), followed by M (4.28 ± 0.02), N (4.20 ± 0.03) and S samples (4.12 ± 0.02). BJ and BP brands presented the lowest pH values (3.71 ± 0.02 and 3.61 ± 001, respectively).

SS/TA ratio

The desirable balance of sweet taste and acidity of the fruit is represented by the ratio between soluble solids in °Brix and titratable acidity (SS/TA ratio) (Ashurst, 2016). This combination of sugars and organic acids provides the soursop's pleasant taste (Pareek et al., 2011) and indicates the maturation index of the fruits. The SS/TA ratio presented a wide range of variation between the analyzed samples. The highest values were found in the frozen soursop pulps from IT (11.02 ± 0.15), BJ (8.62 ± 0.75) and R (8.55 ± 0.65). Samples DM and S showed the lowest average values (< 6.0) and the other brands presented intermediate values (6.16-8.47). A recent study showed that the soluble sugars content and acidity of soursop fruits increase during the ripening process (Othman et al., 2014). Therefore, there is a possibility that frozen pulps with the lowest SS/TA values were subject to water addition during processing or have been produced from soursop fruits with a less advanced maturation index.

3.3 Method validation and mineral content of frozen soursop pulps

The method used for multi-element determination in soursop pulps was validated by evaluating the analytical parameters shown in Table 3. The linearity of analytical curves was assessed through the determination coefficient (R^2) and, R^2 values were higher than 0.999 for most elements, except for Mg (0.9901). LOD and LOQ values ranged from 0.005 to 1.38 μg g^{-1} and 0.016 to 4.55 μg g^{-1}, respectively. These low values show that the method presented high sensitivity and, therefore, is suitable for multi-element analysis of soursop pulps. All RSD values were lower than 9.5%, showing acceptable precision. There is no standard reference material (SRM) for soursop pulp. Thus, the method accuracy was evaluated through NIST standard reference analysis (SRM 1515) of apple leaves (National Institute of Standards & Technology, 2019). As shown in Table 3, there is not a significant difference between experimental and certified (SRM) values. Although calcium presented a high uncertainty (15442 ± 330 mg/kg) compared with certified value (15250 ± 100 mg/kg), these values are not different considering the confidence interval at 95%. Thus, the method may be considered accurate.

After validation, the method was applied to the determination of K, P, S, Ca, Mg, Fe, Cu, Zn and Mn in soursop pulp samples. The results, in μg g^{-1}, are shown in Table 4. High potassium contents were found in all analyzed pulps. Samples FN, R, and S presented the highest contents (1542-1761 μg g^{-1}) and can be considered excellent sources of this nutrient. In samples E, IF and NP, the lowest potassium (544-855 μg g^{-1}) and the highest phosphorus contents were found, ranging from 110 to 139 μg g^{-1}. The other samples presented phosphorus contents lower than 110 μg g^{-1}. Samples NP, E, and FN also presented the highest sulfur contents (101-174 μg g^{-1}). S and IT brands showed the highest calcium (73.8-77.0 μg g^{-1}) and magnesium (>132 μg g^{-1}) contents among the investigated samples. Low calcium contents were determined in NP and IF samples, ranging from 25.3 to 29.4 μg g^{-1}. The lowest magnesium contents also were found in the NP brand (47.2 ± 2.7 μg g^{-1}).

Regarding microelements, the samples that presented the highest iron content were IF, DM, N, and R (> 3.0 μg g^{-1}), while the frozen pulps poorest in this microelement were samples NP, IT and M (1.40 to 1.68 μg g^{-1}). Copper and manganese contents ranged from 0.30 to 0.88 μg g^{-1} and 0.32 to 1.22 μg g^{-1}, respectively. Samples N, S and IF presented the highest copper contents (> 0.75 μg g^{-1}) and the samples from FN, BJ and R brands were

the richest in manganese (0.77-1.22 μg g^{-1}). On the other hand, soursop pulps from NP, BJ, and BP presented the lowest copper content, while the lowest manganese contents were determined in IF and BF samples (< 0.40 μg g^{-1}). Low zinc contents were determined in all the frozen pulps analyzed (< 0.85 μg g^{-1}). Spada et al. (2010) did not find Cu, Zn and Mn in some of the frozen soursop pulps marketed in the state of Rio Grande do Sul, Brazil. The absence of these elements may be related to different growing conditions (Tiburski et al., 2011).

Tiburski et al. (2011) reported that consumption of 300 mL of fruit juice prepared from 100 g of frozen mombin pulp provides approximately 5.8% of recommended daily intake (RDI) for magnesium, 4.6% for phosphorus and 8.2% for potassium. Table 5 shows an estimate of the contribution that frozen soursop pulps provide for daily intake of the essential elements determined in the present study. These values were estimated considering the consumption of 100 g of frozen soursop pulp (one package) and RDI values established in the literature for children and adult

Table 3. Analytical parameters of the method for multi-element determination in frozen pulp samples.

Element	Linear range (mg L^{-1})	Linearity (R^2)	RSD (%)	LOD (mg kg^{-1})	LOQ (mg kg^{-1})	NIST SRM 1515 (Apple Leaves)	
						Experimental values	Certified Values
K	1.46-50	0.9996	0.2-6.9	0.18	4.59	15711 ± 27	16080 ± 210
P	0.16-20	0.9995	0.3-9.1	0.15	0.50	16293 ± 108	NC
S	0.46-20	0.9993	0.2-9.5	0.43	1.44	1867 ± 63	1593 ± 68
Ca	1.46-40	0.9995	0.6-9.7	1.38	4.59	15442 ± 330	15250 ± 100
Mg	0.55-40	0.9901	0.2-9.3	0.52	1.70	2708 ± 81	2710 ± 120
Fe	0.1-2.5	1.0000	0.0-9.2	0.09	0.30	81.7 ± 4.5	82.7 ± 2.6
Cu	0.005-0.5	0.9997	0.3-9.1	0.005	0.015	5.6 ± 0.3	5.69 ± 0.13
Zn	0.016-0.5	0.9998	0.1-6.2	0.015	0.051	12.6 ± 0.4	12.45 ± 0.43
Mn	0.005-0.375	0.9995	0.2-5.7	0.005	0.015	54.2 ± 1.5	54.1 ± 1.1

NC = non-certified value; RSD = relative standard deviation; LOD = limit of detection; LOQ = limit of quantification.

Table 4. Mineral composition of the frozen soursop pulps (in μg g^{-1}). Results are expressed as average ± standard deviation (n = 9).

Brand	Element /μg g^{-1}								
	K	P	S	Ca	Mg	Fe	Cu	Zn	Mn
NP	544 ± 39	110 ± 7	101 ± 23	25.3 ± 1.7	47.2 ± 2.7	1.40 ± 0.21	0.35 ± 0.04	0.39 ± 0.01	0.42 ± 0.19
IF	947 ± 18	139 ± 3	94.7 ± 2.5	29.4 ± 1.2	63.7 ± 1.7	3.32 ± 0.37	0.77 ± 0.03	0.57 ± 0.02	0.32 ± 0.02
E	855 ± 18	123 ± 3	174 ± 5	36.9 ± 6.2	67.3 ± 1.5	2.49 ± 0.15	0.52 ± 0.02	0.66 ± 0.02	0.51 ± 0.01
DM	1325 ± 50	64.0 ± 3.3	75.4 ± 4.1	45.6 ± 2.5	90.8 ± 4.7	3.25 ± 0.08	0.39 ± 0.01	0.47 ± 0.01	0.45 ± 0.01
M	1164 ± 29	67.9 ± 2.0	71.8 ± 2.5	47.4 ± 1.7	85.8 ± 2.4	1.68 ± 0.12	0.43 ± 0.02	0.58 ± 0.01	0.71 ± 0.02
N	1539 ± 54	84.7 ± 5.6	90.7 ± 5.2	50.1 ± 3.6	106 ± 5	3.03 ± 0.10	0.75 ± 0.02	0.50 ± 0.02	0.72 ± 0.02
BF	1392 ± 45	66.4 ± 3.1	84.2 ± 3.9	65.8 ± 3.4	105 ± 5	2.62 ± 0.16	0.54 ± 0.02	0.84 ± 0.03	0.38 ± 0.02
S	1582 ± 39	106 ± 14	172 ± 13	73.8 ± 18.6	132 ± 7	2.38 ± 0.67	0.88 ± 0.20	0.73 ± 0.08	0.73 ± 0.02
IT	1468 ± 35	79.6 ± 1.9	72.7 ± 1.7	77.0 ± 3.8	132 ± 3	1.58 ± 0.03	0.30 ± 0.01	0.64 ± 0.01	0.41 ± 0.01
R	1542 ± 35	66.1 ± 5.2	72.9 ± 5.0	58.8 ± 7.5	92.4 ± 3.4	4.05 ± 1.65	0.68 ± 0.14	0.48 ± 0.07	0.77 ± 0.06
BJ	1536 ± 123	82.6 ± 8.4	82.1 ± 9.8	58.4 ± 4.2	102 ± 10	2.29 ± 0.23	0.53 ± 0.17	0.36 ± 0.03	0.77 ± 0.07
FN	1761 ± 256	80.3 ± 6.8	117.9 ± 24	51.4 ± 3.5	114 ± 9	2.66 ± 0.32	0.58 ± 0.06	0.43 ± 0.02	1.22 ± 0.39
BP	1243 ± 11	44.3 ± 0.8	88.6 ± 2.0	47.3 ± 0.8	70.1 ± 1.0	2.60 ± 0.16	0.34 ± 0.01	0.37 ± 0.004	0.61 ± 0.01

Table 5. Estimated contribution of frozen soursop pulps for recommended daily intake.

Element	Range of concentration/mg 100 g^{-1}	RDI/mg day^{-1}		RDI estimated contribution/%	
		Children[a]	Adults	Children	Adults
Mn	0.027-0.173	1.5	2.3	1.8-11.5	1.2-7.5
Fe	0.114-0.615	9	14	1.3-6.8	0.6-3.2
Cu	0.030-0.114	0.44	0.9	6.8-25.9	3.3-12.6
Zn	0.0325-0.0857	5.6	7.0	0.6-1.5	0.4-1.2
Mg	4.38-13.5	100	260	4.38-13.5	1.7-5.2
K	49.6-208	nd	1600[b]	-	3.8-16.0
Ca	2.43-8.0	700	1000	0.3-1.1	0.2-0.8
P	4.33-14.0	1250	700	0.3-1.1	0.6-2.0
S	6.76-17.8	nd	nd	-	-

[a]childrens 7-10 years (ANVISA); [b]value established in Kohlmeier (2015). nd = not available data; RDI = recommended daily intake.

persons (Brasil, 2005; Food and Agriculture Organization of the United Nations, 2004; Kohlmeier, 2015).

As can be seen in Table 5, frozen soursop pulps can provide up to 11% of manganese and about 13% of magnesium RDIs for children (7-10 years), depending on the product brand. In addition, consumption of a 100 g-portion can supply up to 12 and 16% of the RDI for copper and potassium, respectively, for adult persons. However, contributions from the calcium and zinc RDIs do not reach 1.6% for either age group. These values are similar to those found by Spada et al. (2010), who reported a contribution of 0.5% for calcium RDI and approximately 2.0% for manganese RDI from the consumption of a 100 g-portion of soursop pulp.

4 Conclusions

Based on the evaluation of physicochemical properties such as soluble solids, titratable acidity, pH and SS/TA ratio, it was observed that most of the evaluated frozen soursop pulps presented good quality, except for samples, NP, IF and M. These samples presented soluble solids below the identity and quality standards fixed in Brazilian legislation. Thus, a possible adulteration through water addition may have occurred. Although a detailed bioavailability study is required, the mineral composition showed that frozen soursop pulps are good sources of elements essential to the human organism, except for manganese and zinc, which presented low contents in all investigated samples. Among the thirteen brands evaluated, eight showed high mineral content. Samples FN, S, BF and IT were the richest in major and minor minerals such as K, S, Mg, Fe, Ca, Zn and Cu. The IT and S brands also showed good soluble solids (°Brix) and maturation levels. Therefore, the fruits used in the processing of these frozen-pulps presented excellent quality. Multivariate analysis using PCA showed correlations between different variables and frozen pulp brands.

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Supplementary Material

Supplementary material accompanies this paper.

Table S1. ICP OES operating conditions and spectral lines used for multi-element analysis.

Table S2. Auto-scaled data matrix for principal component analysis.

Table S3. Variables correlations. Strong correlations are highlighted in bold.

Table S4. Eigenvalues of correlation matrix. Were considered only eigenvalues higher or equal to 1.

Figure S1. Scree test graph as criteria for principal component extraction.

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