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# High enhancement of macro and micronutrients quantification in Cajuína by ICP OES using ultrasound and multivariate analysis

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## ABSTRACT

A fast, safe, simple, and low-cost analytical method was developed for the determination of macro and microelements in *Cajuína* samples from Brazil combining UAE and ICP OES. A multiplexed approach using experimental designs was applied for method optimization. The optimal conditions were using 4 mL of 8 mol $L^{-1}$ $HNO_3$ and 1.5 mL $H_2O_2$ under 30 min of sonication. The detectability of the method has been improved. The precision was RSD < 5.5% while the accuracy of the method ranged from 87% to 108% as well as the results were in agreement with the reference method. The analysis of 38 samples of *Cajuína* juices presented a mean of Ca, Cu, Fe, K, Mg, Na, P, S and Zn of 8.70, 0.10, 0.97, 1223, 112, 60.1, 83.9, 65.1, and 0.99 mg $L^{-1}$. So, the proposed analytical method demonstrated feasibility for the simultaneous determination of mineral nutrients in similar matrices as beverage.

## 1. Introduction

The cashew tree (*Anacardium Occidentale* L.) is a tropical plant, originally from Brazil, which brings an essential economic contribution to the Northeast region of the country, especially to the states of Ceará, Piauí, and Rio Grande do Norte (Barbosa Ribeiro et al., 2021). According to the Food and Agriculture Organization of the United Nations - FAO, in 2020, Brazil was the largest producer of cashew *ca*. 1,104.130 tons, a production far above the second place, which is Mali with 173000 and in third place Madagascar with 77000 tons (FAO, 2020).

The cashew apple is considered the pseudo fruit of the cashew tree. It has a high nutritional diversity with nutrients such as polyphenols (flavonoids, carotenoids, anacardic acid, and tannins), sugars, organic acids, vitamins, and minerals (copper, zinc, sodium, potassium, calcium, iron, phosphorus, and magnesium) (Lima et al., 2014; Lopes, Miranda, Moura, & Filho, 2012; Honorato & Rodrigues, 2010; Reina et al., 2022). Because of its nutritional potential, the cashew apple has achieved relevance in the food industry in products such as jam, drinks, and juice (Lima, Silva, Neto, & Moita, 2007).

In addition, the cashew apple has been relevant in the food industry to produce several products. Mainly *Cajuína*, a genuine Northeastern Brazilian beverage with a Geographical Indication (GI) certificate from the State of Piauí and is registered as a Brazilian Cultural Heritage by the Institute of National Historical and Artistic Heritage (IPHAN, 2014).

*Cajuína* is prepared from cashew juice, without alcohol, clarified, and sterilized. It has an amber-yellow color resulting from the caramelization of the juice's natural sugars (Galvão, Sousa, Nascimento, Souza, & Nunes, 2022). Despite being nationally disseminated, there are few

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*Abbreviations:* CRM, Certified Reference Material; *di*, Individual Desirability; FAO, Food and Agriculture Organization of the United Nations; FCCD, Face-Centered Central Composite Design; GI, Geographical Indication; ICP OES, Inductively Coupled Plasma Optical Emission Spectroscopy; IPHAN, Institute of National Historical and Artistic Heritage; IUPAC, International Union of Pure and Applied Chemistry; LOD, Limit of Detection; LOQ, Limit of Quantification; MSlof, Mean square of lack of fit; MSpe, Mean square of pure error; OD, Overall Desirability; PCA, Principal Component Analysis; RCC, Residual Carbon Content; RSD, Relative Standard Deviation; SVD, Singular Value Decomposition; UAE, Ultrasound-Assisted Extraction.

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works on this drink; it was reported in the literature that in the samples of *cajuína* commercialized in the state of Piauí, the vitamin C content is below what appears on the label, requiring better quality control in the processing (Lima, Silva, Neto, & Moita, 2007).

Furthermore, *Cajuína* still has an unknown inorganic composition. In this sense, this study focuses on the determination of inorganic constituents present in the *Cajuína*, an essential task for nutritional reasons and product quality. The determination of inorganic constituents in food samples is commonly performed by spectroanalytical techniques, such as inductively coupled plasma optical emission spectrometry (ICP OES) (Pinheiro, Barros, & Nóbrega, 2019; Santos et al., 2022). Depending on the matrix characteristic, this technique requires a sample preparation procedure.

Studies of sample preparation by microwave-assisted digestion or by a digester block are recurrent in the literature (Oliveira, Silva, Bianchi, & Nogueira, 2018; Mullapudi, Krishnan, Gumma, & Dheram, 2019). However, these approaches present some drawbacks, such as using a large volume of reagents that can enhance the contamination risks, especially for analytes present in low concentrations, in addition, to increasing the waste of reagents.

Ultrasound-assisted extraction (UAE) has emerged as an alternative to improve sample preparation. It has been used in different matrices due to its simplicity, efficiency, analyst safety, low cost, use of mild conditions such as temperature, pressure, and mainly the use of diluted acid solutions (Chemat, Zill-E-Huma, & Khan, 2011). What is related to reducing waste production, increasing the interest in developing green chemistry, and ensuring sustainable and non-toxic sample preparation routes (Bizzi et al., 2020; Diehl et al., 2018).

However, different variables need to be optimized to develop a UAE procedure. Thus, multivariate optimization is a powerful tool for evaluating variables that affect the extraction process to obtain the best optimization conditions for all analytes to ensure the accuracy of the method (Ferreira et al., 2018; Pereira & Pereira-Filho, 2018). In addition, multivariate optimization has been used for UAE procedures (Bizzi et al., 2020; Coelho et al., 2022; Costa et al., 2020; Gamela et al., 2019).

Ca, Fe, K, Mg, Mn, P, and Zn were determined in pepper samples using a full factorial design ($2^3$) with a central point to optimize sonication temperature, sonication time, and $HNO_3$ concentration. As can be seen, multivariate optimization presents several advantages, such as (i) optimization of the experiments in a short time; (ii) efficiency of the results; (iii) achievement of more information from a small number of experiments; and (iv) lower reagent consumption (Novaes et al., 2016; Vera Candioti, De Zan, Cámara, & Goicoechea, 2014).

Considering the lack of information in the literature regarding the inorganic composition of *Cajuína*, this study proposed a multivariate optimization of an UAE procedure for the determination of Ca, Cu, Fe, K, Mg, Na, P, and Zn in *Cajuína* juices by ICP OES. Thirty-eight samples of *Cajuína* supplied by the cities of Ceará and Piauí States, Brazil, were analyzed. Besides that, principal component analysis (PCA) was used to evaluate the similarities among the samples.

## 2. Materials and methods

### 2.1. Instrumentation

The determination of Ca, Cu, Fe, K, Mg, Na, P, S and Zn was carried out using a synchronous vertical dual view Inductively Coupled Plasma Optical Emission Spectrometer (ICP OES) (Agilent Technologies, Model 5110, SVDV, Mulgrave, Australia), equipped with sea spray nebulizer, double-pass cyclonic spray chamber, and peristaltic pump. Argon with purity of 99.999% (White Martins-Praxair, Sertãozinho, SP, Brazil) was used for plasma generation, nebulization system and as auxiliary gas.

Instrumental parameters used for the analytical measurements were 1.2 kW of applied radiofrequency power, 12.0 L $min^{-1}$ plasma gas flow rate, 1.0 L $min^{-1}$ auxiliary gas flow rate, 0.7 L $min^{-1}$ nebulizer gas flow rate, 12 rpm peristaltic pump, 15 s stabilization time, and 3 s integration time. The axial viewing position was used for Na (588.995 nm), Ca (422.673 nm), K (769.897), Mg (280.270 nm), Fe (259.940 nm), P (213.618 nm), and Zn (213.857 nm), while radial viewing was used for Cu (327.395 nm) and S (180.669 nm). The samples were microwave-assisted digested in an Ethos 1 system (Milestone, Sorisole, Italy). The UAE procedure was performed using an ultrasound bath (Ultraclean 1400, Unique®, Brazil) operated at a frequency of 40 kHz and a power of 80 W.

### 2.2. Reagents, and standard solutions

Deionized water (resistivity > 18.2 MΩ cm at 25°C) obtained from a Milli-Q water purification system (Millipore, Bedford, USA) was used to prepare all solutions. All glassware used was washed with soap and soaked in 10% of $HNO_3$ and rinsed with deionized water before use.

Nitric acid (Synth, Diadema, SP, Brazil) used to sample preparation was previously purified using a sub-boiling acid distiller (Model BSB-939-IR, Distillacid, Berghof, Berlin, Germany), and the solution 30% (m/m) of $H_2O_2$ (Sigma Aldrich, Darmstadt, Germany). Standard solutions were prepared by dilution of 1000 mg $L^{-1}$ stock solution of Ca, Cu, Fe, K, Mg, Na, P, S and Zn (Fluka, Buchs, St. Gallen, Switzerland), which were used to build calibration curves.

### 2.3. Samples

Thirty-eight samples of *Cajuína* produced in 2018 were commercially acquired in different cities of Ceará and Piauí, Brazil, as shown in Fig. S1 - Supplementary material. In addition, certified reference material (Peach Leaves, NIST 1547) from the National Institute of Standards and Technology (Gaithersburg, MD, USA) was used to evaluate the efficiency of the sample preparation reference method.

### 2.4. Experimental design for ultrasound-assisted extraction

The randomly chosen sample (ID: 29, Teresina - PI) of *Cajuína* was used to optimize the UAE procedure for the determination of inorganic constituents. A volume of 1.5 mL of *Cajuína* was transferred to a 15 mL conical polypropylene tube, and a full factorial design type $2^4$ was applied to assess the significance of the effects of the variables, the first value being the low level (-1) and the according to the highest (+1): (i) sonication time (30 and 60 min), (ii) $HNO_3$ concentration (5 and 7 mol $L^{-1}$), (iii) $HNO_3$ volume (3 and 5 mL) and (iv) $H_2O_2$ volume (1 and 3 mL). These investigated parameters were based on preliminary tests and reports in the literature (Costa et al., 2020; Gamela et al., 2019; Santos et al., 2014) (Supplementary material - Table S1). All extraction procedures were performed at room temperature *ca.* 25°C.

Considering the multiple responses, the overall desirability (OD) was calculated to get the best conditions and guarantee the compliance criteria for all responses simultaneously and provide the best compromise values in the desirable joint response. It was required to calculate the individual desirability (*di*) (Vera Candioti et al., 2014).

To develop a simple procedure to extract all possible analytes, *di* was calculated considering each analyte's minimum and maximum concentration as described in Eq. 1. Where *y* corresponds to the experimental response, *L* (lower value) and *T* (high value) which is equivalent to the minimum and maximum concentration of each element. The *s* corresponds to the weight, and, in this sense, *1* was assigned to all analytes determined. The OD was calculated using the arithmetic mean of di for each experiment.

$$di = \left(\frac{y - L}{T - L}\right)^s \quad (1)$$

After the results obtained in the full factorial design $2^4$, it was possible to identify the significant variables for the inorganic extraction in *Cajuína* juices. Therefore, a face-centered central composite design (FCCD) was carried out to obtain an empirical model that explained the

**Table 1**
Variables involved in the FCCD and obtained responses.

| | | Experiments | | | Variable coded | | Response | | | | | | | OD |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | $X_1$ | $X_2$ | $X_4$ | Ca | P | Cu | Fe | K | Mg | Na | S | Zn | |
| Design | E1 | -1 | -1 | -1 | 9.55 | 80.21 | 0.13 | 0.49 | 1145.23 | 98.68 | 18.38 | 43.04 | 0.89 | 0 |
| | E2 | +1 | -1 | -1 | 10.08 | 93.72 | 0.16 | 0.56 | 1343.50 | 115.97 | 21.59 | 49.28 | 1.07 | 0.67 |
| | E3 | -1 | +1 | -1 | 11.44 | 90.67 | 0.14 | 0.57 | 1290.45 | 111.67 | 20.72 | 46.40 | 0.96 | 0.53 |
| | E4 | +1 | +1 | -1 | 9.82 | 93.95 | 0.15 | 0.59 | 1343.25 | 116.11 | 21.40 | 48.41 | 1.03 | 0.63 |
| | E5 | -1 | -1 | +1 | 9.69 | 95.55 | 0.15 | 0.58 | 1353.41 | 117.31 | 21.53 | 49.15 | 1.04 | 0.63 |
| | E6 | +1 | -1 | +1 | 9.67 | 95.29 | 0.15 | 0.56 | 1350.07 | 116.75 | 21.46 | 48.90 | 1.02 | 0.58 |
| | E7 | -1 | +1 | +1 | 9.70 | 97.07 | 0.15 | 0.60 | 1376.26 | 119.23 | 21.92 | 49.53 | 1.03 | 0.68 |
| | E8 | +1 | +1 | +1 | 9.39 | 94.89 | 0.15 | 0.57 | 1359.75 | 117.62 | 21.52 | 48.20 | 1.01 | 0 |
| Central Point | E9 | 0 | 0 | 0 | 10.31 | 95.10 | 0.15 | 0.61 | 1355.42 | 117.03 | 21.59 | 49.01 | 1.11 | 0.77 |
| | E10 | 0 | 0 | 0 | 11.33 | 97.57 | 0.16 | 0.62 | 1397.48 | 120.62 | 22.41 | 50.65 | 1.00 | 0.89 |
| | E11 | 0 | 0 | 0 | 10.39 | 96.90 | 0.17 | 0.58 | 1379.47 | 118.95 | 21.89 | 50.42 | 1.08 | 0.83 |
| Axial Points | E12 | -1 | 0 | 0 | 9.69 | 91.73 | 0.15 | 0.56 | 1316.57 | 113.85 | 20.99 | 47.86 | 1.04 | 0.52 |
| | E13 | +1 | 0 | 0 | 9.85 | 85.36 | 0.14 | 0.53 | 1225.16 | 105.81 | 19.39 | 45.21 | 0.93 | 0.25 |
| | E14 | 0 | -1 | 0 | 9.95 | 93.40 | 0.15 | 0.56 | 1333.17 | 115.41 | 21.27 | 48.57 | 1.03 | 0.61 |
| | E15 | 0 | +1 | 0 | 10.19 | 92.42 | 0.15 | 0.56 | 1329.90 | 114.74 | 21.12 | 48.34 | 1.13 | 0.65 |
| | E16 | 0 | 0 | -1 | 10.03 | 96.43 | 0.15 | 0.60 | 1382.50 | 119.33 | 21.97 | 50.39 | 1.07 | 0.76 |
| | E17 | 0 | 0 | +1 | 9.92 | 93.61 | 0.15 | 0.58 | 1334.96 | 115.49 | 21.40 | 48.55 | 1.02 | 0.61 |

data and indicated the best working condition, 17 experiments, 14 authentic experiments, and three replicas at the central point (Table 1, *item 3.2*), the parameters of the empirical model obtained were evaluated by ANOVA. The variables and levels analyzed are presented in Table S2 - Supplementary material.

### 2.5. *Reference method*

Microwave digestion was used for comparison purposes with the developed method because it is widely used in several laboratories and in cashew or similar samples (Lima et al., 2014; Jalbani et al., 2010). In this sense, 1.5 mL of sample was transferred to PFA viels, and 6.0 mL of diluted $HNO_3$ (7 mol $L^{-1}$), along with 2.0 mL of 30% (m/m) of $H_2O_2$ were added to the samples. The samples were submitted to the following heating program: (1) 15 min to reach 200°C; (2) 20 min maintained at 200°C. I in both steps the applied power was adjusted to 1000 W. After cooling to room temperature, the content was transferred to volumetric flasks and was diluted to 14 mL with ultrapure water. In addition, to avoid the degradation of the introduction sample system, the samples were three times diluted, with ultrapure water.

### 2.6. *Figures of merit*

The detection (LOD) and quantification (LOQ) limits were experimentally calculated. According to the International Union of Pure and Applied Chemistry (IUPAC), the LOD was calculated from the mean of standard deviation of the intensity obtained by ten authentic replicas of the blank ($n = 10$) and divided by the slope of the analytical calibration curve. The LOQ was calculated as 3 times the LOD. The precision of the measurements was evaluated by the relative standard deviation (RSD).

Addition and recovery studies were carried out to evaluate the recovery of the acid decomposition method developed. Standard additions were added at the beginning of the decomposition process, referring to three concentrations levels, low, medium, and high, for each studied analyte.

### 2.7. *Statistical analyzes*

One sample of *Cajuína* was used for the optimization of UAE conditions, and all data treatment were performed using Matlab R2015a Software. All analyses were done in triplicate. All experiments were performed randomly. The comparisons were all accomplished with a confidence level of 95%. Tukey test was done to evaluate the significance of the differences between mean values for each assay.

## 3. Results and discussion

### 3.1. *$2^4$ factorial design*

Some approaches allow the practical application of ultrasonic irradiation to aid in extracting metallic species from various solid samples. The intense disturbance imposed by the propagation of acoustic waves in the medium promotes ruptures produced by microjets in the collapse of the cavitation bubble (Santos et al., 2014; Sanwal et al., 2022). The ultrasound-assisted extraction procedure is based on leaching of analyte within the cell matrix from powdered samples by the action of high-intensity sonication in an acid solution, which the extraction of the analytes occurs due to a phenomenon known as acoustic cavitation. Ultrasound accelerates sample preparation as it decreases the concentration of the solvent gradient at the interface between the solid and the liquid. In addition, it can increase the sample's surface area due to the solid erosion (AlYammahi et al., 2022; Korn et al., 2008).

The combination of ultrasonic irradiation and dilute acids has been shown to be an effective strategy for developing greener, faster, and cheaper sample preparation procedures. Despite the simplicity of ultrasound-assisted extraction, the information peculiarities of samples that are submitted to ultrasonic irradiation can be severely compromised, as the collapse of the cavitation bubble results in a substantial increase in local temperature and production of free radicals, which can cause analyte loss and gross errors of analysis (Chemat et al., 2011). In this sense, is necessary study the extraction conditions to which the sample will be submitted.

In this context, the variables sonication time, $HNO_3$ concentration, the volume of $HNO_3$, and $H_2O_2$ were chosen due to their significance in the sample preparation process. From the experimental domain´s definition, optimizing the inorganic extraction present in *Cajuína* occurred aiming at the maximum extraction. Table S1, Supplementary material, presents the design matrix and the global desirability and concentrations of inorganic analytes present in *Cajuína* juice. With Ca, P, Cu, Fe, K, Mg, Na, S and Zn results, it can be observed that the yield varied in the range 6.36 to 9.89; 63.40 to 90.42; 0.10 to 0.18; 0.51 to 0.68; 896.90 to 1335.16; 77.88 to 115.88; 15.33 to 22.76; 33.20 to 44.69 and 0.66 to 0.99 mg $L^{-1}$. Making a global average of extraction yield of 8.59, 82.21, 0.14, 0.59, 1194.30, 103.95, 20.27, 41.26 and 0.87 mg $L^{-1}$ for Ca, P, Cu, Fe, K, Mg, Na, S and Zn, respectively.

The effects of the chosen variables were calculated according to Barros Neto, Scarmino & Bruns (2010). They were possible to calculate ten effects percentages for each variable and their interactions, as depicted in Fig. S2 (Supplementary material). The variables $X_1$ (sonication time), $X_2$ ($HNO_3$ concentration), and $X_4$ (volume of $H_2O_2$) were

**Table 2**
ANOVA for the face-centered composite design.

| Variation source | Square sum (SS) | Degrees of freedom | Square mean (SQ) | $F_{calc}$ (95%) | $F_{tab}$ (95%) |
|---|---|---|---|---|---|
| Regression (R) | 0.928 | 9 | 0.103 | 6.437 | 3.676 |
| Residue (r) | 0.112 | 7 | 0.016 | | |
| Lack of fit (Lof) | 0.104 | 5 | 0.020 | 6.666 | 19.296 |
| Pure error (Pe) | 0.007 | 2 | 0.003 | | |
| $R^2$ | 0.892 | | | | |
| r maximal variation | 0.992 | | | | |

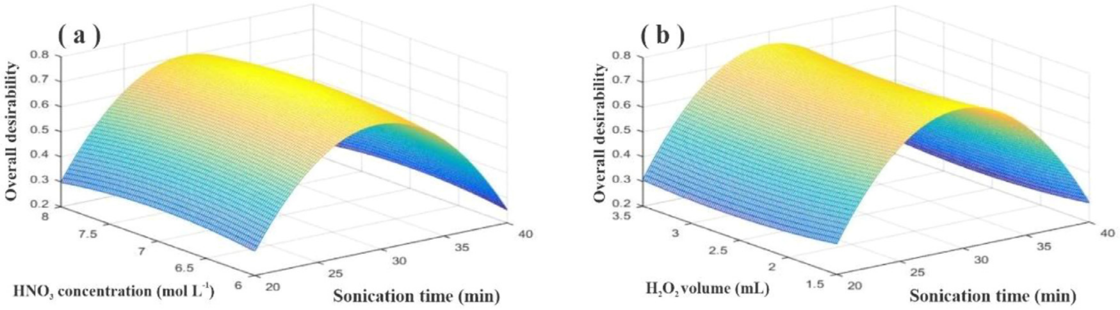

**Fig. 1.** Response surface obtained using FCCD for $HNO_3$ concentration and sonication time (a) and response surface for $H_2O_2$ volume and sonication time (b).

significant at 95% of confidence level, showing to be important in the extraction process. The variable $X_1$ had the most significant influence on the extraction method (25.26%), with its effect value being negative, which indicates that the maximum extraction tends to a low level. The variable $X_3$ (Volume of $HNO_3$) was not significant, so it was set close to its effect value 0.03 for future experiments, corresponding to *ca.* 4 mL.

Given the above, a new FCCD was evaluated with the variables that proved to be significant at new levels to find the optimal condition of the proposed factors for the inorganic extraction from *Cajuína.*

### 3.2. Face-centered central composite design (FCCD)

The FCCD has already been applied for fine-tuning in some studies (Lopes Júnior et al., 2018; Song et al., 2018). Each variable was studied at three different levels, and the results of the experiments are presented in Table 1. In this design, three replicates were measured in a central point to evaluate the error and raise 17 experiments. It is possible to observe that with the choice of new experimental levels for the three significant variables, there was an increase in extraction yield when compared to full factorial design $2^4$ for most of the analyzed analytes, having a global average of 10.06, 93.17, 0.15, 0.57, 1330.39, 114.97, 21.21, 48.35 and 1.03 mg $L^{-1}$ for the Ca, P, Cu, Fe, K, Mg, Na, S and Zn, respectively.

Because of the results obtained with the FCCD tests, the proposed model was evaluated employing ANOVA at 95% of confidence level, and the model obtained does not present lack of fit because the ratio between the mean square of lack of fit (MSlof) and mean square of pure error (MSpe), the $F_{calculated}$ (6.666) was lower than $F_{tabulated}$ (19.296) at 95% confidence level, as shown in Table 2. The quadratic model was able to explain 89% of the variation present in the analyzed tests. The maximum variation explained by the model was 99%, which was adequate to describe the data since only 1% of the errors cannot be explained. The F test for the variance described by the quadratic model related to the variance of residuals showed that the first is significantly greater than the second, since the value of $F_{calculated}$ 6.437 was superior to $F_{tabulated}$ 3.676 at a confidence level of 95%. This means that the model can be used to get predictions.

The polynomial that describes the proposed quadratic model is represented by Eq. 2 and the response surfaces for this model are presented in Fig. 1.

$$OD = 0.7342 - 0.0230X_1 - 0.0090X_4 - 0.2774X_1 * X_1 - 0.0324X_2 * X_2 + 0.0226X_4 * X_4 - 0.1500X_1 * X_2 - 0.1875X_1 * X_4 - 0.1275X_2 * X_4 \quad (2)$$

Fig. 1 shows the response surface for the optimized procedure of extraction built using Eq. 2. The response surface obtained by plotting the variables concentration of $HNO_3$ and sonication temperature, represented in Fig. 1a, shows that the increase in extraction efficiency is tending toward the central point on the time axis, as for the concentration of $HNO_3$, the maximum of the curve is situated on the positive side which is close to 8 mol $L^{-1}$. The use of diluted acids is desirable in analytical methods based on ICP-optical emission spectrometry, because a diluted acid is not as aggressive to the spectrometer's receiving input system which can overcome the non-spectral type interference that is commonly caused by deficiencies during sample transport. In addition, the use of diluted receipts for the sample digestion process is in accordance with the principles of Green Chemistry as it is environmentally-friendly (Farias et al., 2022). In this context, more efficiency is obtained with 30 min sonication time. A fast analysis method is desirable as it significantly reduces analyst work and costs.

Examinig the response surface obtained by plotting $H_2O_2$ volume *versus* sonication time (Fig. 1b), it is possible to observe that the maximum point of curvature is found in the negative section of the volume axis of $H_2O_2$. Solving Eq. 2 this visualization is confirmed, whose optimal condition for this variable is the volume of 1.5 mL. Therefore, the use of a lower volume of $H_2O_2$ contributes to reducing costs and generating less waste. In this sense, the best conditions of UAE for the determination of inorganic constituents, used in further experiments were (i) 30 min of sonication time, (ii) 4 mL of $HNO_3$ 8 mol $L^{-1}$, and (iii) 1.5 volume of $H_2O_2$.

The veracity of the developed procedure (UAE) was evaluated using the spike and recovery test. The recoveries of the analytes, performed in low, middle, and high levels, presented recoveries values between 87 and 108% (Table S3 - Supplementary material).

The ultrasonic energy applied under optimal conditions acts on the incidence of low-frequency mechanical waves that result in cavitation, a phenomenon responsible for the formation and collapse of microbubbles, which reflect in specific areas of high pressure and temperature, facilitating the extraction process. During the extraction procedure, the action of cavitation favors the breaking of bonds between the metal and the organic matrix present in the sample. The breaking of bonds is re-

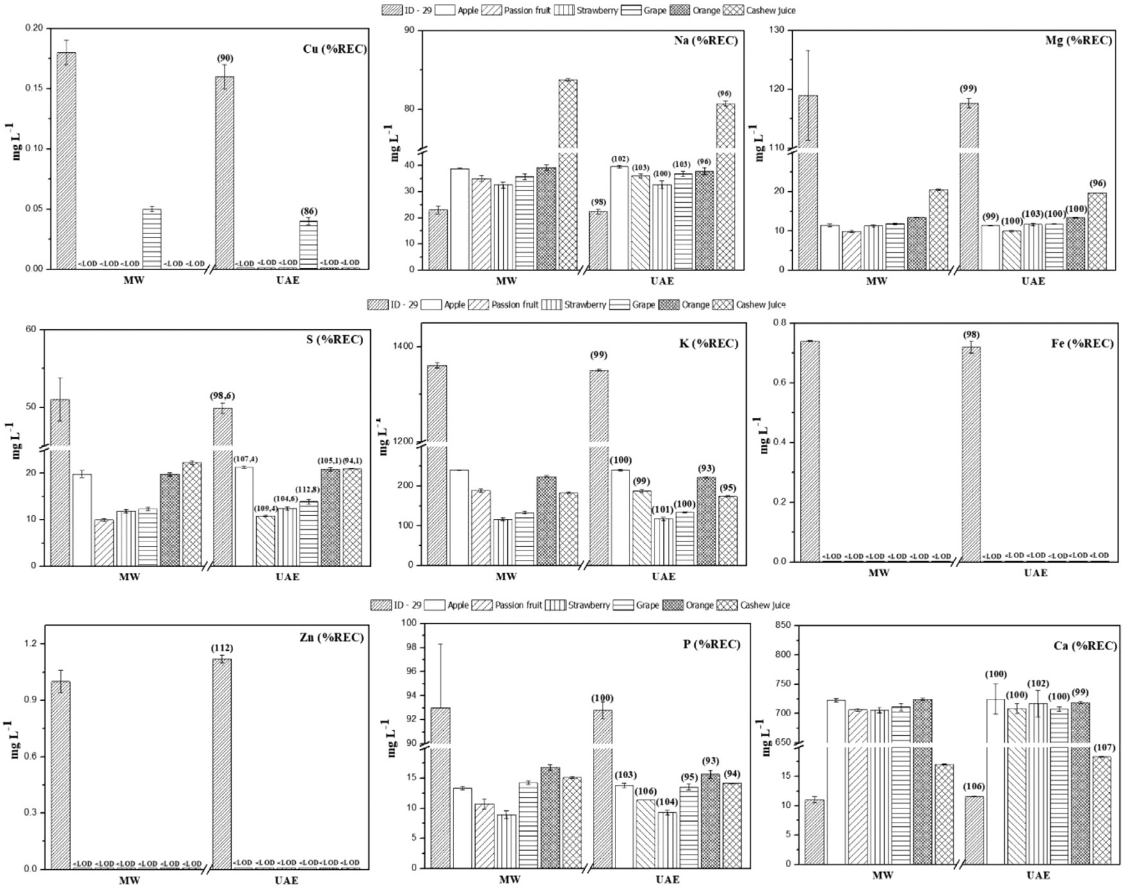

**Fig. 2.** Concentration values of Ca, Cu, Fe, K, Mg, Na, P, S and Zn obtained in *Cajuína* (29) and different fruit juices employing the optimized procedure (UAE) and microwave digestion (MW, reference method).

lated to the production of H· and OH·, highly reactive free radicals, which are released into the solution during the sonication process (Diehl et al., 2018; Santos, Andrade, Galvão, & Felsner, 2019; Machado, Faccio, & Pistón, 2019).

In literature the ultrasound-assisted extraction has already been applied for the determination of micro and macro minerals in non-centrifugal sugar by FAAS, where the best condition was achieved with the proportion of 60:40% (v/v) of $HNO_3$:$H_2O_2$ as an extraction solution, 0.100 g of non-centrifugal sugar and 60 min of sonication (Santos, Andrade, Galvão, & Felsner, 2019). Multiple response optimization of ultrasound-assisted procedure was also applied for multi-element determination in Brazilian wine samples by microwave-induced plasma optical emission spectrometry. The optimum conditions established were concentrations of nitric acid (2.3 mol $L^{-1}$) and hydrogen peroxide (9%) and the accuracy confirmed by spike tests with recoveries ranging from 104 to 118% and comparative analysis of the results obtained using the proposed method and digestion total procedure (Carneiro & Dias, 2021).

In this context, the proposed method based on ultrasound-assisted extraction becomes a viable alternative, because it is a relatively simple methodology, which allows for faster sample preparation and less complexity, in addition to being low cost when compared to other extraction methods used in chemical analysis laboratories, such as microwave-assisted extraction.

### 3.3. Quality control and analytical performance

The evaluated figures of merit of the proposed procedure were the limits of detection (LOD) and quantification (LOQ), presented in Supplementary material, Table S4. The veracity of the reference method, evaluated by the use of certified reference material, is shown in Fig. S3 - Supplementary material. It is possible to observe that the proposed procedures presented LOD and LOQ much lower when compared to those obtained by microwave acid digestion (reference method) (Table S4 - Supplementary material).

The veracity of the reference method, the microwave digestion procedure, presented results between 81 and 111% of concentration recovery of all analytes according to the values certified in the CRM (Fig. S3 - Supplementary material). Statistical evaluation, using Student's t-test, showed no significant difference between the values obtained and the certified values, with a confidence level of 95%, with the value $t_{calculated}$ (1.87) and $t_{tabulated}$ (2.26), confirming the method veracity.

The efficiency of the digestion procedure was also evaluated by determining the residual carbon content (RCC), which is an essential factor that can affect the analytical results. The RCC results were 19.6% for CRM, 4.1% in *Cajuína* for the microwave reference method, and 10.8% in *Cajuína* for the proposed UAE. Therefore, both approaches presented acceptable RCC results and within the range described in the literature (Costa, Silva, Gouveia, Nogueira, & Nóbrega, 2001; Santos et al., 2019). Fig. 2 presented the concentration values in *Cajuína* sample (ID - 29) and different commercial fruit juices obtained using the optimized conditions of UAE, which were compared with the reference method. Statistical evaluation, using Student's t test, showed that there is no significant difference between the values obtained and the certified values, with a confidence level of 95%, with the value $t_{calculated}$ (1.00; 1.98; 1.14; 1,24; 0.13; 1.59; 1.52) to *Cajuína* (29), apple, passion fruit, strawberry, grape, orange, cashew, respectively and $t_{tabulated}$ (2.26) confirming the method veracity. The UAE results are in agreement with the reference method, as it presented excellent recoveries, showing that the UAE can be used as a method for analyzing inorganic elements in *Cajuína* and similar sample juices.

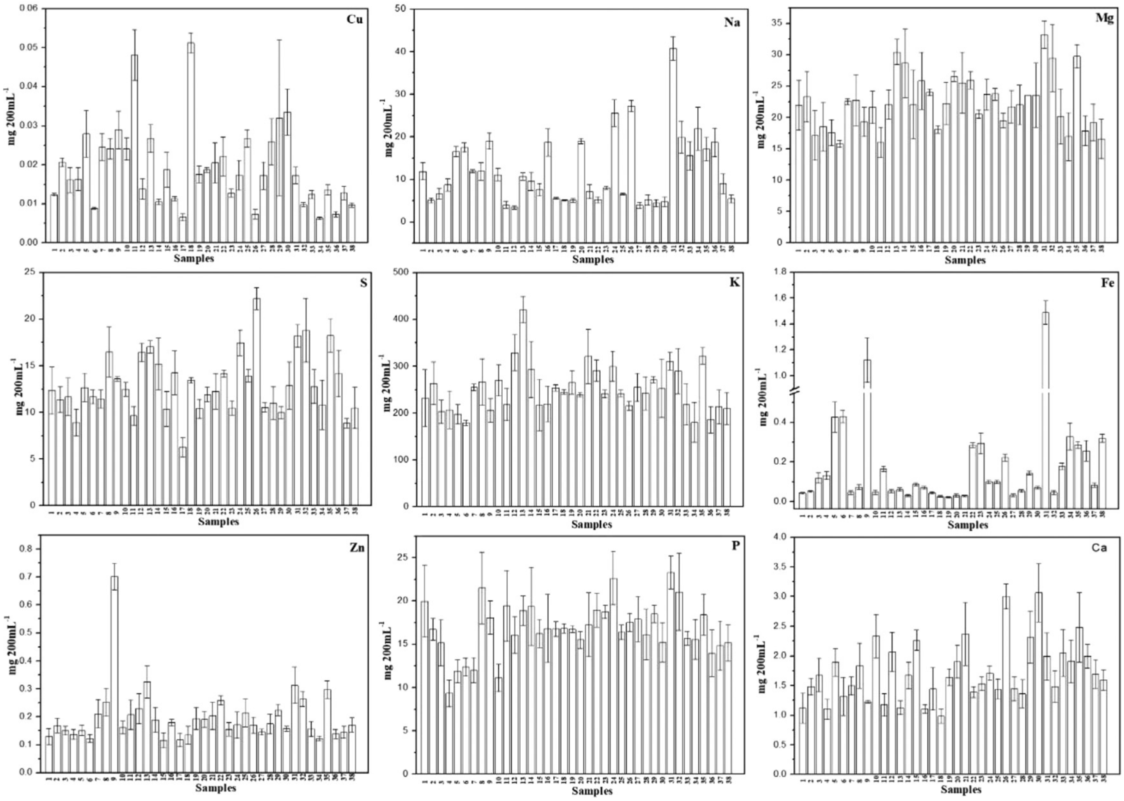

**Fig. 3.** Concentration values Ca, Cu, Fe, K, Mg, Na, P, S and Zn obtained in *Cajuína* employing UAE (mg 200mL$^{-1}$).

The optimized procedure was used to determine the concentration levels of Ca, Cu, Fe, K, Mg, Na, P, S, and Zn in 38 samples of *Cajuína* and they are expressed in mg 200 mL$^{-1}$ as it is the portion normally consumed (Fig. 3). The concentration values varied from (in mg 200 mL$^{-1}$): 0.98 to 3.4 for Ca, 0.0006 to 0.052 for Cu, 0.02 to 1.49 for Fe, 23.6 to 420.8 for K, 15.8 to 33.2 for Mg, 3.44 to 40.8 for Na, 9.4 to 23.4 for P, 6.2 to 22.2 for S, 0.2 to 0.8 for Zn.

Compared with the inorganic elements in different juice fruits described in the literature, *Cajuína* is rich in macro and microelements and can be considered a nutritional source. As examples, Souza and collaborators found contents (mg 200mL$^{-1}$) of Ca between 2 and 19.4; Cu from 0.019 to 0.10; Fe <0.0054 to 0.74; Mg from 3.5 to 20.06; Na from 0.66 to 7.98; and Zn <0.003 to 0.196 in different juices, such as orange, tangerine, apple, peach, lemon, pineapple, red berries, and passion fruit (Souza, Barciela-Alonso, Aboal-Somoza, & Bermejo-Barrera, 2021). Demir and collaborators determined concentrations of essential and non-essential elements in orange, peach, apricot and cherry juices finding a content of Ca from 10.76 to 35.4; Cu from 0.0014 to 0.0166; Fe from 0.0132 to 0.178; K from 168.4 to 216; Mg from 5.54 to 14.66; Na from 0.61 to 4.6; and P from 7.34 to 201.8 mg 200 mL$^{-1}$ (Demir, Kipcak, Dere Ozdemir, & Moroydor Derun, 2020).

### 3.4. Characterization of Cajuína by principal component analysis

The *Cajuína* obtained in different cities of Ceará and Piauí States were evaluated using principal components analysis (PCA). The PCA consists of singular value decomposition (SVD), where the X matrix is decomposed into two new matrices, T (scores) and P (loadings), *i.e.*, using T and P, it is possible to identify patterns in the original matrix X that usually contains the samples in the rows and variables in the columns, and in our specific case are the concentration of the analytes.

Due to different variations in the magnitude of the concentration values of the analytes, the data were pre-processed using autoscaling, in which the data were mean-centered, then each value was divided by the standard deviation. This tool is an unsupervised method that correlates different variables for the evaluation and characterization of the analytical data, identifying trends and sample clusters. A data matrix of 38 × 9 (samples *vs* variables) was generated, and the first four principal components (PC1, PC2, PC3 and PC4) accounted for 34% 17%, 16% and 10% of the total explained variance, respectively (Fig. 4).

Fig. 4a shows the obtained loadings with positive values in PC1, which indicate a tendency to form a correlation among the analytes (Ca, Cu, Fe, K, Mg, Na, P, S, and Zn), followed by a negative value for Cu in PC2, indicating an inversion correlation on the set of the samples. However, Fig. 4b shows the scores plot of PC1 versus PC2, and is not possible to clearly distinguish a separation among the samples. The difficult of the separation of these samples can be explained mainly due the near concentration values of the analytes in the samples. Freitas and collaborators (2020) also used PCA to analyze the difference between different cashew clones and observed the separation of these samples resulting from some factors such as cashew flavor, cashew aroma, sweet aroma, softness, and juiciness, however, the element's determination was not carried out.

Other PCs were evaluated if there is a difference and similarities among the samples from the cities of Ceará and Piauí states. Fig. 4c shows the loadings plot for PC3 versus PC4, where some elements as K, Mg, Na, S and P are more centralized, what become hard to identify a possible separation between the samples, and it can be confirmed by scores, depicted in Fig. 4d. Nevertheless, Cu, Fe and Zn with positive values in PC3, and Ca with negative value in PC4 are more highlighted and are responsible by the separation of some samples, as for example for those represented by number 9, 11, 18 and 29 in positive PC3, and 26 and 30 in negative PC4. Samples 13 and 31 differ from the others due to differences in K, Fe, and Na concentrations, which can be attributed to the incorrect cleaning of the *Cajuína* packaging bottle. Therefore, the C*ajuína* samples present lower concentration values of the elements with high similarities for most of analyzed samples even those obtained in different cities, what explain the poor separation using PCA.

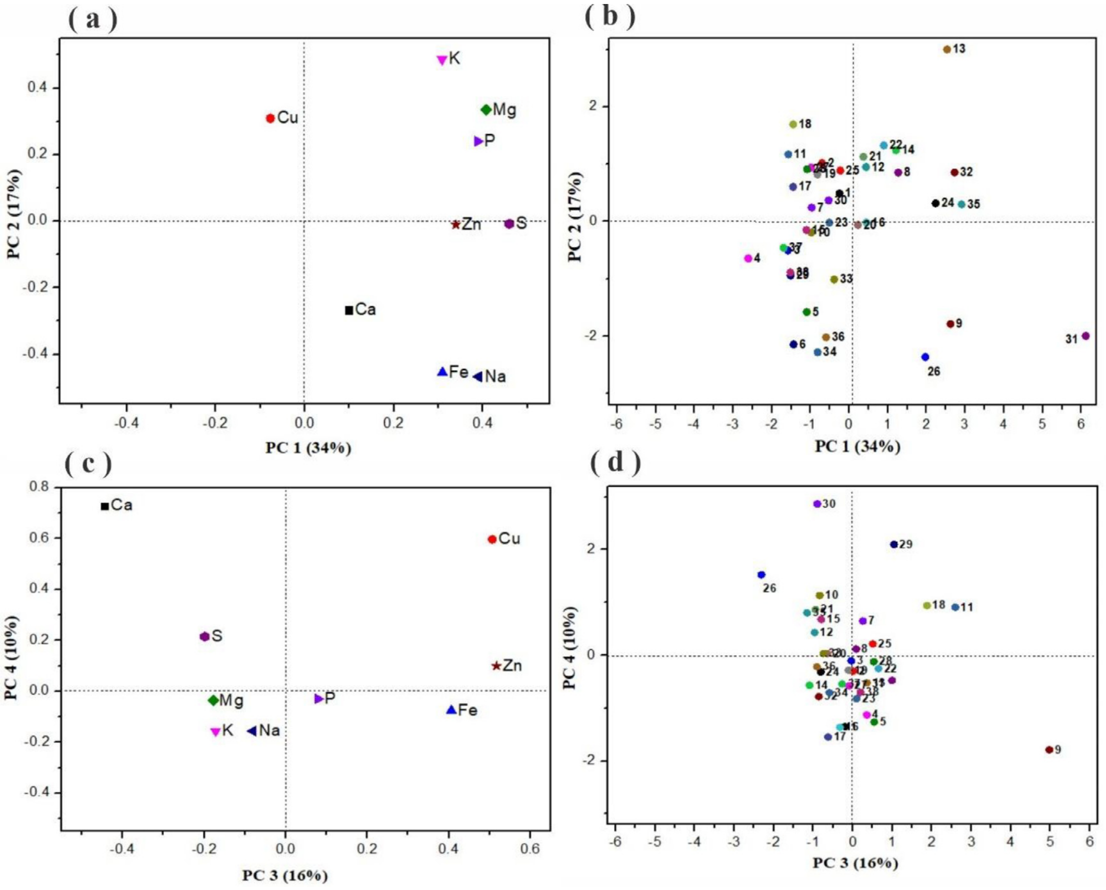

**Fig. 4.** Loadings (a) and Scores (b) for PC1 versus PC2, and, loadings (c) and Scores (d) for PC3 versus PC4.

These similarities and differences can be attributed to several reasons, as plants can present changes in the element's absorption and availability. This availability depends on the soil cation exchange capacity and varies considerably with soil types, mineral composition, and pH. Other factors must also be considered such as fertilization, irrigation water, and mycorrhizal fungi in the root zone. Because the same plant species can have different mineral compositions, thus it depends on cultivation conditions (Reina, 2022; Simpkins, 2000).

## 4. Conclusions

The use of combined multivariate analysis and ultrasound-assisted extraction allowed identifying the most important variables and establishing a better condition for the extraction of macro and microelements in samples of *Cajuína* juices. So, the optimal conditions were 30 min of sonication time, 4 mL of 8 mol $L^{-1}$ $HNO_3$ and 1.5 mL of $H_2O_2$. The proposed method showed good accuracy and precision. Through PCA was not possible observe a good separation and groups of clusters were not formed due to the to the variability of the samples from different cities.

The optimized method produced robust results comparable to the reference method, making it an alternative to microwave digestion for sample preparation. Based on our findings, we can state that the proposed method is simple to perform, fast, viable, precise, and accurate, and can be used as an alternative for routine analysis of Ca, Cu, Fe, K, Mg, Na, P, S and Zn in *Cajuína* and matrices of similar juices.

## Declaration of Competing Interest

Authors declare no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

## CRediT authorship contribution statement

**Tiago Linus Silva Coelho:** Formal analysis, Conceptualization, Visualization, Writing – original draft. **Darlisson Slag Neri Silva:** Writing – review & editing. **Luiz Brito de Souza Filho:** Methodology, Visualization. **Jardel Menezes Rocha:** Methodology, Visualization. **Julymar Marcano de Higuera:** Methodology, Formal analysis, Writing – original draft. **Ivero Pita de Sá:** Methodology, Formal analysis, Writing – original draft. **Raimundo Rafael Gamela:** Data curation, Writing – original draft. **Ana Rita de Araujo Nogueira:** Conceptualization, Project administration, Supervision, Visualization, Writing – review & editing. **Cícero Alves Lopes Júnior:** Conceptualization, Visualization, Writing – review & editing. **Edivan Carvalho Vieira:** Conceptualization, Project administration, Supervision, Visualization, Writing – review & editing.

## Data Availability

The data that has been used is confidential.

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## Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.focha.2023.100265.

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