Acid extraction of metals from cashew leaves cultivated in Roraima and determination by Inductively Coupled Plasma Optical Emission Spectroscopy

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Resumo:

O cajueiro (Anacardium occidentale), espécie nativa do Brasil, apresenta grande importância econômica, nutricional e industrial, sendo uma valiosa fonte de compostos bioativos e nutrientes. Apesar de seu potencial agroindustrial, especialmente em regiões como o estado de Roraima, estudos sobre a composição mineral das folhas dessa cultura em condições amazônicas ainda são escassos. A análise foliar é uma ferramenta essencial para o diagnóstico do estado nutricional das plantas e para o direcionamento de práticas de fertilização mais sustentáveis. Este estudo teve como objetivo avaliar a eficácia de três métodos de extração ácida na determinação da composição elementar de folhas de cajueiro cultivadas em Roraima. As amostras foram analisadas por espectrometria de emissão óptica com plasma indutivamente acoplado (ICP-OES), técnica reconhecida por sua alta sensibilidade e capacidade multielementar. Os resultados visam identificar o método mais eficiente para extração de nutrientes e fornecer subsídios técnicos para o manejo nutricional da cultura na região Norte, contribuindo para o aumento da produtividade e o desenvolvimento sustentável da cajucultura local.

Palavras-chaves: Caju, ICP, Roraima.

Abstract:

The cashew tree (Anacardium occidentale), a species native to Brazil, is of great economic, nutritional, and industrial importance, being a valuable source of bioactive compounds and nutrients. Despite its agroindustrial potential, especially in regions such as the state of Roraima, studies on the mineral composition of the leaves of this crop under Amazonian conditions are still scarce. Leaf analysis is an essential tool for diagnosing the nutritional status of plants and for directing more sustainable fertilization practices. This study aimed to evaluate the effectiveness of three acid extraction methods in determining the elemental composition of cashew leaves grown in Roraima. The samples were analyzed by inductively coupled plasma optical emission spectrometry (ICP-OES), a technique recognized for its high sensitivity and multielement capacity. The results aim to identify the most efficient method for nutrient extraction and provide technical support for nutritional management of the crop in the North region, contributing to increased productivity and the sustainable development of local cashew farming.

Keywords: Cashew, ICP, Roraima.

Introduction

The cashew tree (Anacardium occidentale), a species native to Brazil, stands out for its economic, nutritional, and industrial importance (LIMA et al., 2025). Its nuts are widely valued for their high content of protein, unsaturated fatty acids, vitamins, and minerals, attributes that give the crop significant agroindustrial potential (KRISHNAPPA et al., 2024).

In this context, assessing the plant's nutritional status becomes essential to support more efficient agricultural practices. Leaf analysis is an essential tool for diagnosing nutrient deficiencies or excesses, enabling corrective interventions in crop management (PEREIRA et al., 2022). Knowledge of the mineral composition of leaves is particularly relevant for formulating more accurate and sustainable fertilization recommendations.

However, studies related to cashew farming in the state of Roraima are still in their infancy, and to date, there are no investigations addressing the elemental composition of cashew leaves under local conditions. This gap highlights the need for research focused on the nutritional characterization of the species in the region. The state of Roraima, located in the North Region of Brazil, has areas where cashew trees are cultivated, an activity that represents a strategic alternative for generating income and promoting food security, especially among family farmers. Given its geographic location and favorable soil and climate characteristics, the state has great potential for expanding the crop.

Therefore, it is essential to develop studies that identify the chemical elements essential for the full development of the plant, contributing to the formulation of technological strategies aimed at increasing productivity and promoting sustainable development in the region. Among the methods used to analyze nutrients in plant tissues, acid digestion stands out for its simplicity, low cost, and efficiency. Extraction with dilute acid solutions represents a gentle and effective approach for releasing macro- and micronutrients in soluble forms (RODRIGUES et al., 2025).

In general, sample preparation—which includes steps such as digestion or extraction—is essential for subsequent multielement analysis, as it aims to mineralize the organic fraction and solubilize the analytes in inorganic forms (ALMEIDA et al., 2025). In this study, three different extraction methods were applied to cashew leaves to evaluate their effectiveness in determining elemental composition. Comparing the methods will not only identify the most appropriate technique for the species under study but also provide unprecedented data on the nutritional status of cashew trees grown in the state of Roraima.

The analyses were conducted using inductively coupled plasma optical emission spectrometry (ICP-OES), a technique widely used to determine mineral elements in plant samples. Recognized for its high sensitivity, accuracy, precision, and broad multielement detection capacity, ICP-OES requires rigorous sample preparation, including complete solubilization (SILVA et al., 2025; SANTOS et al., 2024; FERREIRA et al., 2025). Its application in this study aims to ensure the reliability of the results and expand technical-scientific knowledge about the mineral nutrition of cashew trees under Amazonian edaphoclimatic conditions.

Methodology

Elemental determination was performed using an Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) model PlasmaQuant 9100 from Analytik Jena (Thuringia, Germany). The instrument has a borosilicate glass cyclonic nebulization chamber, a detachable V-Shuttle torch with a 2 mm injector and cap (quartz), a 1 mL/min borosilicate concentric nebulizer, and a PVC pump tube. ASpect PQ 1.3.2.0 software was used for ICP-OES control and analysis. Analytical argon 5.0 (White Martins, Rio de Janeiro, Brazil) was used for plasma generation.

Samples were weighed using a balance (Shimadzu model UX620H, Tokyo, Japan) with a resolution of 0.001 g and a maximum capacity of 620 g. Digital muffle furnace (SPlabor model SP-1200, São Paulo, Brazil) with a maximum temperature of 1200°C used in the dry digestion process. 50 ml porcelain crucible. Quantitative filter paper (Whatman 42 type, 15 mm diameter, Merck, Darmstadt, Germany). Ultrapure water from the Milli-Q system (Millipore, USA) with a resistivity of 18 MΩ cm. HNO3 (P.A., 65% w/w, 1.4 kg/L, Merck, Darmstadt, Germany). ICP multi-element standard solution (1.045 g/cm3 at 20 °C, Merck, Darmstadt, Germany) was diluted to prepare the calibration curve. ICP phosphorus standard solution (1.045 g/cm3 at 20 °C, Merck, Darmstadt, Germany) was diluted to prepare the calibration curve. Calibration standard solutions were prepared by diluting them in water acidified with 1.25% (v/v) nitric acid.

Samples were collected from cashew trees located in the Boa Vista region, Roraima, Brazil. This location has an average annual temperature of 23.5°C and an average annual precipitation of 146.3 mm (NATIONAL INSTITUTE OF METEOROLOGY, 2024). The collected leaves were washed with distilled water and dried at 60°C to constant weight. They were then ground, passed through 20-mesh sieves, and stored for the digestion stage (SILVA, 2009).

The extraction of chemical elements present in plant tissue was performed using three distinct methods. In the first method, a 10 mL aliquot of nitric acid was added to 1 g of the plant sample, followed by digestion at a constant temperature of 140 °C (ZARCINAS, CARTWRIGHT & SPOUNCER, 1987). Stirring was necessary during the process to control foaming. Digestion continued until the volume was reduced to approximately 1 mL of acid remaining. The solution was then cooled and diluted to a final volume of 20 mL using 1% (v/v) nitric acid.

The second method consisted of weighing 1 g of the plant sample, adding 10 mL of nitric acid, and allowing the mixture to stand overnight. Subsequently, the sample was heated to 125 °C for 4 hours. After cooling, the solution was first diluted to 12.5 mL with nitric acid and then to 50 mL with distilled water (HALVIN & SOLTANPOUR, 1980). In the third method, 500 mg of the plant sample was weighed and transferred to a porcelain crucible, which was gradually heated in a muffle furnace until it reached 500 °C, where it remained for 3 hours before being turned off. After the crucible cooled, 25 mL of a 1 M nitric acid solution was added to extract the elements (SILVA, 2009).

Results and Discussions

The analyses performed by inductively coupled plasma optical emission spectrometry (ICP OES) allowed accurate and rapid multielement profiles, since the technique, combined with the sample preparation and digestion methods, presents adequate sensitivity and the capacity for simultaneous determination of several elements (SILVA et al., 2021).

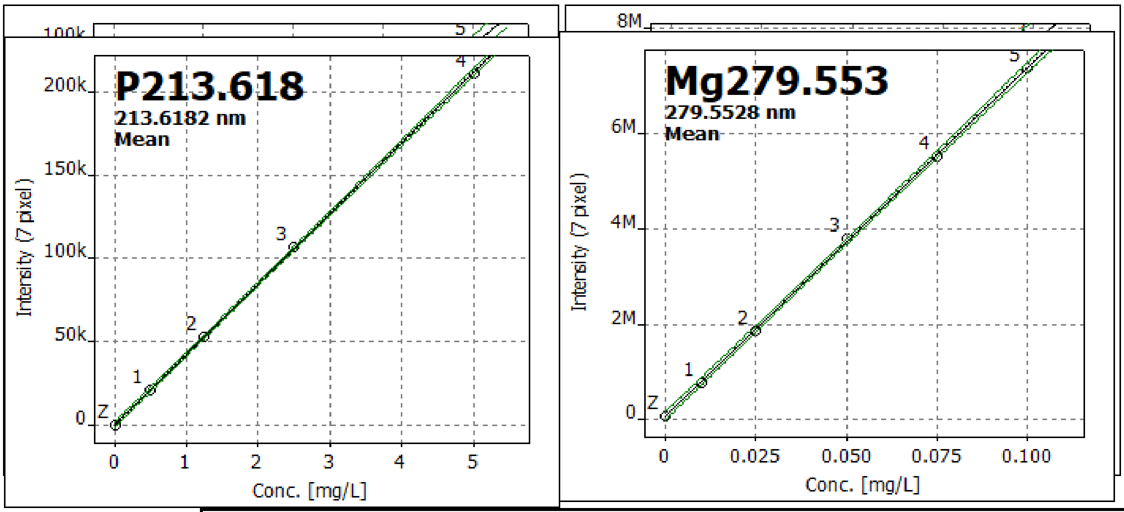
Macronutrients are essential for plant growth and productivity and are absorbed primarily from the soil by the root system (SOUSA et al., 2021). Among these, potassium (K) stands out due to its observed concentrations and its importance in osmotic regulation, enzyme activation, protein synthesis, photoassimilate translocation, and stomatal aperture control. Insufficient K levels are associated with reduced carbon assimilation and decreased crop productivity, while adequate availability, including through biofertilizers, contributes to improved grain quality and greater tolerance to abiotic and biotic stresses (SOUMARÉ et al., 2023; RAWAT, PANDEY & SAXENA, 2022).

Phosphorus (P) plays an equally important role, participating in key processes such as nucleic acid synthesis, phospholipid formation, ATP production, respiration, and photosynthesis. The balanced distribution of its different fractions is crucial for root growth, energy transfer efficiency, and stress tolerance (PANG et al., 2024; WALI et al., 2025; LI et al., 2025).

Magnesium (Mg) plays a physiological role in photosynthesis, carbohydrate partitioning, and protein synthesis. Its deficiency affects the transport of photoassimilates, which can compromise the growth of roots, nodules, and seeds, in addition to reducing nitrogen use efficiency (TIAN et al., 2021; XIE et al., 2021). Finally, calcium (Ca) is essential for cellular integrity and growth regulation, in addition to contributing to stress tolerance. Its low mobility in the phloem explains the occurrence of disorders such as apical necrosis and leaf tip burn when at inadequate levels (Faizan et al., 2025).

Thus, the simultaneous determination of these elements via ICP OES provides robust information to interpret the efficiency of extraction methods and the nutritional status of the plant. The evaluation of the extraction methods began with the production of calibration curves for each element analyzed by ICP. As shown in Figure 1, high linearity was observed in all curves, indicating a good fit between concentration and instrumental response.

Figure 1. Calibration curves of the analyzed elements.



The sensitivity of the method was determined based on the limits of detection (LOD) and quantification (LOQ), which varied according to the specific characteristics of each element. In all cases, a proportional increase in the intensity of the analytical signal was observed as the concentration increased, Figure 2, confirming the reliability of the procedure.

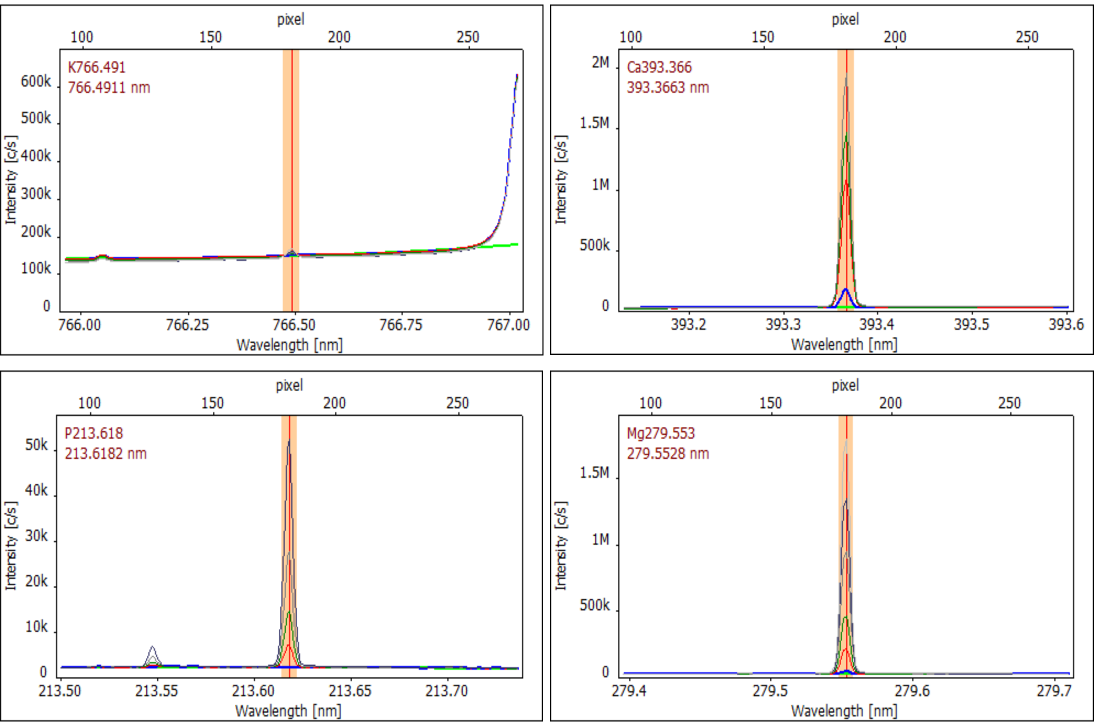


Figure 2. Calibration curves of the analyzed elements.

Table 1 presents the correlation coefficients (R^2), limits of detection (LOD), and limits of quantification (LOQ) obtained for the calibration curves. The curves demonstrated high linearity, with R^2 ranging from 0.9948 to 0.9998. The sensitivity of the method was demonstrated by the low values of LOD, below 0.0380 mg/L, and LOQ, below 0.0048 mg/L, demonstrating the precision and detection capacity of the analytical procedure.

Table 1. Limit of detection (LOD), limit of quantification (LOQ), calibration curve (y) and linearity.

Elements	y	R^2	LOD (mg/L)	LOQ (mg/L)
K	12676.493+860590.03x	0.9982	0.0027	0.0099
P	504.78159+42186.920x	0.9998	0.0380	0.1508
Ca	637313.15+87743792x	0.9948	0.0057	0.0209
Mg	65436.109+73282085x	0.9997	0.0013	0.0048

The chemical elements potassium (K), phosphorus (P), calcium (Ca), and magnesium (Mg) were determined using three different extraction methods. In the first method, 1 g of the sample was used, to which 10 mL of nitric acid was added, followed by digestion at 140 °C (ZARCINAS, CARTWRIGHT & SPOUNCER, 1987). In the second method, 1 g of the sample was also used, with the addition of 10 mL of nitric acid and subsequent digestion at 124 °C for a short period of time (HALVIN & SOLTANPOUR, 1980). In the third method, 500 mg of the sample was calcined in a muffle furnace at 500 °C for 4 h. After cooling, 25 mL of 1 mol/L nitric acid was added to extract the elements (SILVA, 2009). Table 2 presents the results obtained by the three methods.

Table 2. Determination of chemical elements and Standard Deviation (SD).

Elements	Method 1 (mg/kg)	SD	Method 2 (mg/kg)	SD	Method 3 (mg/kg)	SD
K	2664.00	21.06	2997.50	9.80	3285.00	8.60
P	445.20	0.42	442.85	5.26	532.50	0.06
Ca	39.90	0.01	102.30	0.09	102.00	0.02
Mg	50.54	0.00	120.80	0.07	120.45	0.07

Potassium (K) concentration varied among the three methods evaluated, with a smaller difference observed between the results obtained by the second and third methods. For phosphorus (P), there was significant similarity between the first and second methods, although the value obtained by the third method did not present a significant discrepancy in relation to the other two methods.

In the case of calcium (Ca) and magnesium (Mg), the first method resulted in substantially lower concentrations compared to the other methods, suggesting low extraction efficiency for these elements. In the literature, both in old studies and in more recent research, a trend is observed in which the foliar concentrations of macronutrients in the common cashew tree and the precocious dwarf cashew tree follow the decreasing order K > Ca > Mg > P.

However, the results obtained in this work presented the following sequence: K > P > Ca > Mg, suggesting that the extraction methods evaluated were not sufficiently effective for the calcium and magnesium present in the analyzed samples (FREITAS, G. S., FERNANDES, J. R., & SILVA, C. A. (2003); SILVA, FERNANDES, OLIVEIRA & DIAS, 2024).

Final considerations

The evaluation of the mineral composition of cashew leaves grown in the state of Roraima highlighted the importance of choosing the appropriate extraction method to obtain reliable data on the plant's nutritional status. Among the three methods evaluated, variation in element extraction efficiency was observed, especially for calcium and magnesium, reinforcing the need for methodologies compatible with the characteristics of the plant matrix analyzed. The use of inductively coupled plasma optical emission spectrometry (ICP-OES) proved to be an effective tool for multielement analysis, ensuring accuracy and sensitivity in the results obtained. This study represents an initial advance in the nutritional characterization of cashew trees under Amazonian edaphoclimatic conditions and provides support for the development of more efficient and sustainable nutritional management practices.

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