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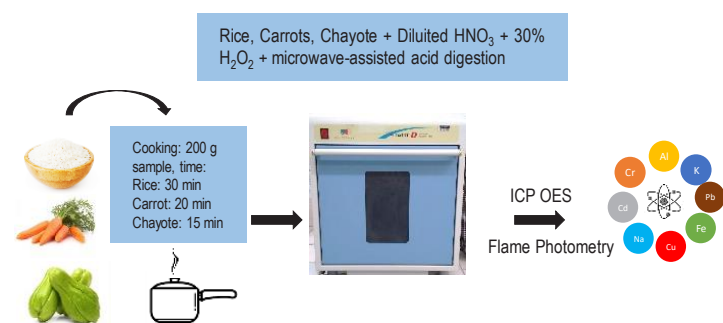
Mineral Composition of Rice, Carrots, and Chayote after Microwave-Assisted Decomposition using Diluted Nitric Acid

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Carrots, chayote, and rice are foods widely consumed by the Brazilian population, so developing analytical methods that allow elemental characterization becomes extremely important. In this work, the sample decomposition method is proposed to prepare samples (raw and cooked) of rice, carrots, and chayote using dilute nitric acid. The samples underwent microwave-assisted acid decomposition (HNO₃/H₂O₂)

for subsequent determination by ICP OES for metals Al, Cd, Cr, Cu, Fe, and Pb and flame photometer for K and Na. Two decomposition methods were used. Method 1: 260 and 500 mg of sample, 3.5 mL of HNO₃ 1 mol L⁻¹, 1.0 mL of H₂O₂ 30% m/m and 3.5 mL of ultrapure water. Method 2: 260 and 500 mg of sample, 5.0 mL of HNO₃ 1 mol L⁻¹, 2.5 mL of H₂O₂ 30% m/m and 0.5 mL of ultrapure water. A factorial design (2²) was also performed to know the effects of some variables on digestion efficiency. Based on the accuracy study using certified reference materials and the analysis of residual acidity and dissolved organic carbon, the condition that presented the best decomposition results was using 5 mL of HNO₃ 0.5 mol L⁻¹ and 2.5 mL of H₂O₂ 30% for 100 mg of sample. Quantification limits ranged from 0.14 (Cr) and 3.4 mg kg⁻¹ (Pb), and the correlation coefficient was close to one in all cases. It was also observed that the cooking process increased the concentration of Al, Cd, and Cr and decreased Cu and K for all foods.

Keywords: vegetable food, cooking effect, minerals, diluted acid, microwave-assisted digestion

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INTRODUCTION

The demand for plant-based foods is on the rise due to their ability to provide essential nutrients for the human body to function properly.¹⁻⁴ Brazil, one of the main responsible countries for meeting this global demand, currently it occupies third position in the ranking of the largest food producers, contributing significantly to global food security.⁵ Among the prominent products in production and consumption are rice, carrots, and chayote. The country stands out especially in rice production, being the main producer outside the Asian region, with an annual harvest that surpassed the 10 million tons mark during the period from 2021 to 2022, while consumption reached the 10,800 tons mark.⁶ Carrot production is carried out in more than 25 countries and in Brazil, the state of Minas Gerais has an extension of approximately 25 thousand hectares annually, resulting in a yield of around 1 million tons and an average consumption of 5.3 kg per capita per year.⁷⁻⁹ Chayote, although it is a constant presence in various culinary cultures around the world, has its main producing region concentrated in Central America. In Brazil, it is among the ten most consumed vegetables, highlighting its relevance in the national diet.¹⁰

The widespread adoption of these foods is driven by their beneficial nutritional properties for human health. In foods of plant origin, micro and macronutrients are found. There are two types of micronutrients: vitamins and minerals. Minerals are inorganic substances (Ca, K, Mg, Na, P, Zn among others) that are present in different proportions in the tissues and fluids of the human body, and they are of vital importance for the correct functioning of the organism.^{11,12} For example, iron plays a critical role in oxygen transport and the immune system along with copper, while potassium is closely related to blood pressure regulation.^{13,14}

Vegetables can be consumed raw, cooked, or roasted, with boiling being the most common preparation method. Thus, there is a risk that ingesting foods of plant origin will not necessarily meet the body's nutritional requirements, due to the losses associated with different preparation methods designed to improve its palatability. In this sense, there is a growing interest in studying the effects of culinary processing on the nutritional and sensorial quality of vegetables.¹⁵⁻¹⁹

Due to the nutritional losses that can be caused by cooking methods, several studies have been conducted with the aim of evaluating the impact of these processes in the presence of metals, including Al, As, B, Ba, Cd, Co, Cr, Fe, Li, Mg, Mn, Na, Ni, Pb, Se, and Zn, present in samples of rice, carrots, and chayote after being subjected to different preparation methods, such as cooking, steaming, boiling under pressure, and steaming under pressure.²⁰⁻²⁷ Although the results of these studies have confirmed nutritional losses due to the diversity of cooking methods, there is no trend in the results indicating which mineral is more prone to leaching.

Through reliable analytical methods, with adequate precision and accuracy, it is possible to know the mineral profile of foods and, consequently, carry out their nutritional evaluation.^{28,29} The closed systems for sample preparation in food prevent contamination and analyte loss; in addition, they provide control over temperature, pressure, and execution time. Therefore, microwave-assisted digestion has proven to be very efficient and safe in oxidizing organic matter.^{28,30} However, the use of concentrated acids in the process generates more waste. Previous studies have suggested using diluted acids (notably HNO₃) combined with H₂O₂ as a solution to this problem, as it maintains digestion efficiency, resulting in digests with low residual acidity and lower standard deviations of blank analytical signal. A brief and simple explanation for the efficiency of this new methodology is the regeneration of HNO₃ during the digestion process.^{28,29,31}

The application of Principles of Green Chemistry is increasingly prevalent in analytical methods, as demonstrated by da Silva and colleagues who used diluted HNO₃ for the determination of nine minerals in rice.³² Also, UI-Haq and colleagues assessed the health risks associated with the presence of trace metals in vegetables such as carrots, cauliflower, pumpkin, and spinach. They combined ultrasound-assisted extraction with diluted acids and 10% H₂O₂. This procedure not only exhibited excellent efficiency but was also conducted in less time and with lower reagent consumption.³³

Given the importance of the determination of minerals in food, this work described the sample preparation using wet decomposition, making use of diluted HNO₃ with H₂O₂ for subsequent quantification of Al, Cd, Cr, Cu, Fe, K, Na, and Pb in samples of carrot (*Daucus carota*), chayote (*Sechium edule*),

and rice (*Oryza sativa*), raw and cooked. The criteria for the selection of the samples were based on their consumption and availability indexes. This is the first study to our knowledge to apply microwave-assisted digestion with diluted acids in the preparation of chayote and carrot samples for subsequent multi-elemental determination. This approach allowed the evaluation of nutrient content and the identification of potentially hazardous metal levels based on the standards set by the Brazilian Health Surveillance Agency (ANVISA).³⁴

MATERIALS AND METHODS

Instrumentation

A microwave oven, model Start D (Milestone, Italy), with a capacity of ten polytetrafluoroethylene vessels, was used to digest the samples. The maximum power of 1200 W was applied, and one of the vessels (reference vessel) was equipped with a temperature sensor.

A flame photometer, model DM-61 (Digimed, Brazil), was used to quantify the Na and K in the digested samples. The following operating conditions were employed for this instrument: sample flow rate of 5.0 mL min⁻¹; response time of 8 s; propane-air flame, using dust-free and oil-free air as the oxidizer, at a pressure of 20 psi and a flow rate of 9 L min⁻¹, supplied by a membrane compressor, model 089-CL (Fanem, Brazil); burner with electronic ignition and safety valve to interrupt the flow of fuel in case of lack of air via pressure switch; wavelength selection by interference filters with a passband lower than 12 nm, at wavelengths of 589 nm (Na) and 766 nm (K); measuring range from 0 to 100 mg L⁻¹; and resolution of 0.1 or 1 mg L⁻¹.

The concentration of the metals (Al, Cd, Cr, Cu, Fe, and Pb) and dissolved carbon was measured using an inductively coupled plasma optical emission spectrometer, model Optima 7000 DV (Perkin Elmer, United States). The following conditions were employed: radiofrequency (RF) power of 1.3 kW, nebulizer gas flow rate of 0.8 L min⁻¹, auxiliary gas flow rate of 0.2 L min⁻¹, plasma gas flow rate of 15 L min⁻¹, sample flow rate 1.0 mL min⁻¹, cross-flow nebulizer, and Scott spray chamber. The following emission lines (nm), in axial plasma view, were controlled: Al (396.153), Cd (228.802), Cr (267.716), Cu (327.393), Fe (238.204), and Pb (220.353).

Reagents and solutions

For the sample preparation procedure, HNO₃ P.A. was employed at concentrations of 0.5, 1.0, and 1.5 mol L⁻¹ (Química Moderna, Brazil) and H₂O₂ P.A. 30% w w⁻¹ (Dinâmica Química Contemporânea, Brazil). Also, distilled-deionized water with specific resistivity of 18.2 MΩ cm from a Milli-Q purification system (Millipore, United States) was used to wash the glassware and prepare the solutions.

The reference solutions used in flame photometry were prepared from metal salts: potassium chloride (Nuclear, Brazil) and sodium chloride (Química Moderna, Brazil). The concentrations of the solutions used in the procedure were 2.0, 5.0, 10.0, 20.0, 30.0, 40.0, 50.0, 60.0, 80.0, and 100 mg L⁻¹ of K and Na. For the ICP OES, the solutions used were from multi-elemental standards (Inorganic Ventures, United States) in concentrations of 0.01, 0.1, 0.5, 1.1, 2, and 4 mg L⁻¹ of Al, Cd, Cr, Cu, Fe, K, Na, and Pb.

To determine the dissolved organic carbon (DOC), anhydrous D-glucose P.A. (Synth, Brazil) was used as a standard, and a stock solution of 50,000 mg L⁻¹ of glucose was prepared. From dilutions, solutions were prepared at concentrations of 100, 500, 1000, 2500, and 5000 mg L⁻¹ of glucose.

The digests' residual acidity (RA) was determined by titration in an aqueous medium using a standardized sodium hydroxide solution and phenolphthalein acid-base indicator. The solutions had a concentration of 0.1 mol L⁻¹ and 0.05 mol L⁻¹.

Samples

Food samples were purchased in supermarkets in the municipality of Vitória de Santo Antão (state of Pernambuco, Brazil). As a pre-treatment before starting the digestion, the vegetables and the parboiled rice were washed in running water, followed by immersion in 200 mg L⁻¹ NaClO for 15 min for disinfection. These foods were then rinsed under running water, and the carrots and chayote were peeled and cut into

slices with an average thickness of 2 cm. Subsequently, 200 g of sample was subjected to wet cooking, in a volume of filtered water corresponding to 3 times the amount of food, in grams.

The pan used was made of aluminum, and the cooking times were 15 min (chayote), 20 min (carrot), and 30 min (rice) under boiling at a temperature of approximately 100 °C. These time and temperature conditions were necessary for the food fibers to become soft, without undergoing disintegration. After cooking, the samples' raw and cooked portions were ground in a household multiprocessor and inserted in an oven with a controlled temperature of 65 °C for approximately 24 h. After drying, the samples were crushed again, this time in a mill (Model MA 630/1, Marconi, Brazil), sieved (60 mesh or 250 µm), and stored in plastic bottles. In the tests, a sample of each matrix was used, in raw and cooked form, totaling six samples.

Microwave-assisted acid digestion

Two analytical methods were tested in the present study. In Method 1, the parameters were determined based on tomato³⁵ and rice³⁶ analysis methods, and 260 mg of the rice sample was weighed, in triplicate, on an analytical balance, directly in the PTFE vessels of the microwave oven. Then, in a laminar flow hood, 3.5 mL of 1 mol L⁻¹ HNO₃, 1.0 mL of 30% H₂O₂ (w w⁻¹), and 3.5 mL of ultrapure H₂O were added.

Subsequently, the vessels were capped and placed in a microwave oven and submitted to a four-step heating program: (i) 4 min to reach 90 °C, (ii) 2 min hold at 90 °C, (iii) 6 min to reach 180 °C, and (iii) 6 min hold at 180 °C. The same procedure was conducted, in triplicate, together with the blanks, for all raw and cooked carrot and chayote samples and repeated with 500 mg of sample.

The same procedures described in Method 1 were performed in Method 2. This was developed based on research stating that volumes of HNO₃ between 0.4 and 2.0 mL are recommended for the composition of up to 200 mg of organic sample.²⁸ Since the maximum indicated ratio is 2 mL for 200 mg, 5 mL is recommended to digest 500 mg of sample. Regarding H₂O₂, no standardized methodologies recommending fixed volumes of this reagent were found in the literature; however, the quantities used in some matrices like those studied in the present work were found. Therefore, observing an increase of 1.5 mL for nitric acid (from 3.5 to 5 mL), it was decided to use a volume of H₂O₂ with the same increase, changing from 1 mL (Method 1) to 2.5 mL. Thus, Method 2 corresponds to the following condition: 5 mL of 1 mol L⁻¹ HNO₃, 2.5 mL of 30% H₂O₂, and 0.5 mL of H₂O.

At the end of the heating program, the digestion vessels, before being opened, were kept in a fume hood until they reached room temperature for approximately 1 h to avoid the loss of volatile elements. After the digestion, the formed solutions were transferred to 15 mL volumetric flasks and diluted to this volume with ultrapure water.

After the digestion procedure, washing runs were performed with the used vessels. In this process, 9.0 mL of nitric acid (50% v v⁻¹) was added to the vessels, and heating was started in a microwave oven following the program: (i) 10 min to reach 160 °C, and (ii) 5 min hold at 160 °C. After the washing runs, the vessels were washed with ultrapure water and immersed in a 10% HNO₃ bath (v v⁻¹) for storage until the subsequent use, when they were rinsed with ultrapure water and dried. The glassware was washed with neutral detergent and ultrapure water at least three times and allowed to dry at room temperature.

RESULTS AND DISCUSSION

Evaluation of the application of Methods 1 and 2

With the application of Method 1, which corresponds to digestion with the addition of 3.5 mL of 1 mol L⁻¹ HNO₃, 1.0 mL of 30% H₂O₂ (w w⁻¹), and 3.5 mL of ultrapure H₂O, it was observed that in the tests carried out with the mass of 260 mg, the digested samples of all foods presented a very cloudy visual appearance and contained particles in suspension. The qualitative results were considered more undesirable for the digested samples from the 500 mg sample than those obtained after the 260 mg sample. The digested from Method 1 contained more particles in suspension, were brown, and had residues of undigested samples. This leads to the conclusion that the tested methodology did not yield adequate decomposition.

The need to develop a new method was observed from the unsatisfactory results of applying Method 1. With the application of Method 2, it was noted that the appearance of the digested solutions with a mass of 260 mg became clearer than 500 mg. Specifically, chayote samples subjected to Method 1 digestion showed the lowest rate of decomposition. This means that the amount of oxidizing reagent present in the reaction medium was not adequate, resulting in the presence of undigested sample residues in suspension.

Factorial design

Although more satisfactory results had been obtained with the application of Method 2 and the 260 mg sample, an attempt was made, through the application of a factorial design, to try to reach a more economical procedure that would produce more diluted acid residues than some methods already proposed for vegetable food.³⁷⁻³⁹ The 2² factorial design was carried out using the chayote sample as a reference because it was the sample with the lowest digestion efficiency, based on the visual aspect, with the application of methods 1 and 2. The volume of added nitric acid remained constant (5 mL), while the volume of peroxide was varied, keeping the total volume constant (8 mL). Ultrapure water was added to complete this final volume. The amount sampled was held constant at 100 mg.

With the realization of the factorial design, it was possible to conduct an initial study searching for the conditions in which the variables significantly influence digestion. Inserting the results obtained in the Statistica software, version 12.5 (StatSoft, USA), it was possible to obtain the Pareto chart allowed for assessing the magnitude of influence of each factor on the responses (DOC and RA).

Starting the study with the values obtained for the residual acidity, the effect of the nitric acid concentration was the only significant variable on the final residual acidity of the non-hydrogen peroxide digest (Figure 1A). Evaluating the Figure 1A, it is observed that nitric acid influences residual acidity. At the same time, Figure 1B shows that increasing the volume of peroxide leads to a decrease in dissolved carbon. This demonstrates that the effects negatively influence the response; taking them from the lowest to the highest level causes a decrease in the response.

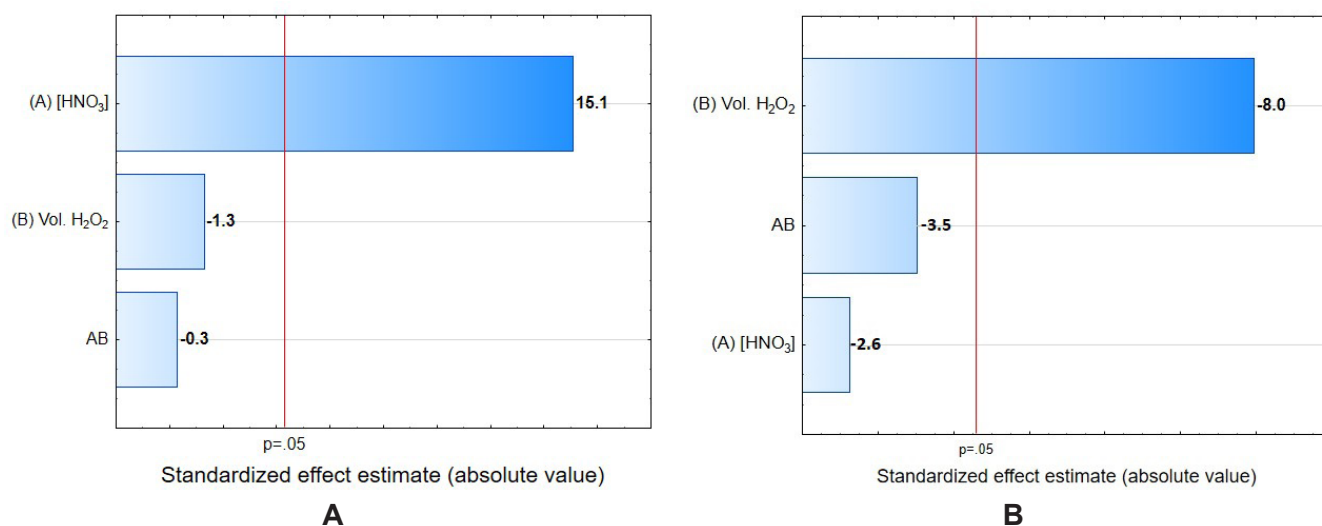


Figure 1. A: Pareto chart of residual acidity results. B: Pareto chart obtained from the analysis of the dissolved carbon results.

The solutions obtained after carrying out the planning were clean. However, it is worth mentioning that obtaining a clear solution for the sample only indicates that the sample may have been completely digested. Some organic compounds that have been partially digested are soluble (do not give turbidity) and colorless (do not give color to the digest). The results obtained of the dissolved organic carbon and residual acidity are shown in Table I.

Table I. Dissolved organic carbon and residual acidity obtained for the digested samples of the factorial design

Essay	HNO ₃ (mol L ⁻¹)	H ₂ O ₂ (mL)	Dissolved organic carbon (mmol L ⁻¹) ^a	Residual acidity (mol L ⁻¹) ^a
1	0.5	2.0	13.7 ± 6.1	0.220 ± 0.003
2	1.5	3.0	5.86 ± 0.42	0.560 ± 0.008
3	0.5	3.0	< LOD	0.190 ± 0.013
4	1.5	2.0	12.4 ± 2.0	0.600 ± 0.008
5	1.0	2.5	6.07 ± 0.35	0.370 ± 0.014
6	1.0	2.5	8.67 ± 1.44	0.400 ± 0.007
7	1.0	2.5	5.81 ± 0.30	0.350 ± 0.014

^aMean value ± standard deviation (n = 3). < LOD: Below limit of detection.

It is observed that the test carried out in the condition of lower volume of hydrogen peroxide was the one that presented the highest amount of dissolved carbon, while the test carried out in the central point condition (essays 5-7) and essay 3 presented the lowest dissolved carbon values. Therefore, to optimize the procedure, it was possible to conclude that by taking the lowest concentration of nitric acid and adding to the volume of hydrogen peroxide at the central point, it would be possible to obtain a digested with low residual acidity and low dissolved carbon content. The center point condition for hydrogen peroxide volume was chosen instead of essay 3 because 0.5 mL less is used, and the dissolved carbon values are similar. The optimized condition for sample preparation was: 5 mL of HNO₃ at a concentration of 0.5 mol L⁻¹, 30% H₂O₂ at a volume of 2.5 mL, 0.5 mL of water, and 100 mg of sample.

Method validation

The method was validated by defining the following figures of merit: linearity (correlation coefficient), limits of detection (LOD) and quantification (LOQ), and accuracy SRM NIST 1568a (Rice Flour); CRM-Agro C1001a (Brown Rice); CRM USP/Embrapa Agro C1002a (Biodynamic Brown Rice); CRM USP/Embrapa Agro C1004a (Tomato Pulp); CRM USP/Embrapa Agro C1006a (Soy Flour) and CRM USP/Embrapa Agro C1007a (Green Coffee). The LOD and LOQ were determined using INMETRO validation guide (LOD = 3.3s/b e LOQ = 10s/b) where “s” is the standard deviation from blank determinations and “b” is the slope of the analytical curve.⁴⁰

The values of the correlation coefficients were close to one (Table II), demonstrating that the method is linear for the quantification of chemical elements of interest. The LOD ranged from 0.043 to 1.0 mg kg⁻¹ and the LOQ from 0.14 to 3.4 mg kg⁻¹ for Cr and Pb, respectively.

Table II. Equations of the analytical curves of the studied elements, their respective correlation coefficient values, and limits of detection and quantification

Analyte	Analytical curve equation ^a	Coefficient of correlation (r)	LOD ^b (mg kg ⁻¹)	LOQ ^b (mg kg ⁻¹)
Al	y = 309122x – 22721	0.9997	0.37	1.1
Cd	y = 167035x + 2647	0.9999	0.15	0.45
Cr	y = 168069x – 377.65	0.9999	0.043	0.14
Cu	y = 324013x – 8541.4	0.9999	0.20	0.62

(continues on the next page)

Table II. Equations of the analytical curves of the studied elements, their respective correlation coefficient values, and limits of detection and quantification (continuation)

Analyte	Analytical curve equation ^a	Coefficient of correlation (r)	LOD ^b (mg kg ⁻¹)	LOQ ^b (mg kg ⁻¹)
Fe	$y = 183729x - 2660.8$	0.9996	0.15	0.46
K	$y = 543745x - 163955$	0.9955	0.13	0.41
Na	$y = 2000000x - 498788$	0.9957	0.18	0.54
Pb	$y = 8071.8x + 550.25$	0.9999	1.0	3.4

^ay: analytical signal; x: concentration, in mg L⁻¹.

^bIn calculating LOD and LOQ, the sample mass (100 mg) and the final volume of the digested samples (15 mL) were considered. That way, $LOD (mg\ kg^{-1}) = 150 \times LOD (mg\ L^{-1})$.

The accuracy of the method was checked using Rice Flour SRM NIST 1568a and Brown Rice CRM-Agro C1001a. The recommendations of the European Reference Materials (ERM) application note on comparing a measured result with a certified value were followed.⁴¹ The results obtained from the studies carried out with the certified materials (Table III) revealed a remarkable congruence between the obtained values and the expected parameters, thus indicating the effectiveness and reliability of the method used during the analysis process. The reliability of the method in question plays a crucial role, as it ensures that the results obtained are representative and consistent, offering, therefore, a solid basis for subsequent analyzes or for taking decisions that are based on the results.

Table III. Determined and certified values obtained after the analysis of five reference materials

Analyte	CRM USP/Embrapa Agro C1001a; Brown Rice		CRM USP/Embrapa Agro C1002a; Biodynamic Brown Rice		CRM USP/Embrapa Agro C1004a; Tomato Pulp		CRM USP/Embrapa Agro C1006a; Soy Flour		CRM USP/Embrapa Agro C1007a; Green Coffee	
	DV (mg kg ⁻¹) ^a	CV (mg kg ⁻¹)	DV (mg kg ⁻¹) ^a	CV (mg kg ⁻¹)	DV (mg kg ⁻¹) ^a	CV (mg kg ⁻¹)	DV (mg kg ⁻¹) ^a	CV (mg kg ⁻¹)	DV (mg kg ⁻¹) ^a	CV (mg kg ⁻¹)
Al	*	*	*	*	*	*	*	*	*	*
Cd	*	*	*	*	1.23 ± 0.1	1.23 ± 0.08	*	*	*	*
Cr	*	*	*	*	*	*	*	*	2.23 ± 0.06	2.04 ± 0.26
Cu	4.1 ± 0.1	4.5 ± 0.3	1.83 ± 0.14	1.87 ± 0.23	7.73 ± 1.3	7.94 ± 0.67	11.4 ± 1.1	12.8 ± 2.8	18.76 ± 1.5	20.1 ± 1.2
Fe	22.7 ± 5.4	17.0 ± 1.3	11.8 ± 6.7	13.3 ± 1.3	27 ± 4.7	30.9 ± 2.0	123 ± 10	137 ± 17	39.7 ± 3.4	44.1 ± 4.0
K	1921 ± 41	1852 ± 319	1917 ± 1692	2058 ± 86	21744 ± 680	26100 ± 2100	18250 ± 1451	17762 ± 1068	18950 ± 440	17510 ± 960
Na	122 ± 37	117 ± 28	*	*	479 ± 35	304 ± 23	*	*	*	*
Pb	<LOD ^b	<0.010 ^b	*	*	*	*	*	*	*	*

DV: Determined value; CV: Certified value.

^aMean value ± standard deviation (n = 3).

^bCRM Rice Flour SRM NIST 1568a

NR: value not reported in the certificate of analysis.

< LOD: Below limit of detection.

*Undeclared or unmeasured value.

Elemental determination by ICP OES and flame photometry

With the optimized condition established, digestions were performed for the rice, carrot, and chayote raw samples and after cooking, as per Table IV, which contains the concentrations for the elements Al, Cd, Cr, Cu, Fe, K, Na, and Pb. The determinations were carried out by ICP OES for metals Al, Cd, Cr, Cu, Fe, and Pb and flame photometry for metals K and Na.

It is worth noting that cooking increased the concentration of micro-contaminants Al, Cd, and Cr (Table IV). However, the Pb could not be measured for rice and carrots and could be considered the same content for chayote raw and after cooking. Meanwhile, for elements considered essential (Cu, Fe, and K), the concentration decreased after cooking for most samples. This behavior was only not verified for Na.

Table IV. Analyte concentration, in $\mu\text{g g}^{-1}$, in samples of carrot, chayote, and rice raw and after cooking

Sample	Elemental concentration ^a							
	Al	Cd	Cr	Cu	Fe	K	Na	Pb
Raw carrot	9.73 ± 1.92	< LOD	0.50 ± 0.05	9.66 ± 1.25	40.40 ± 5.96	8931 ± 197	2841 ± 24	< LOD
Cooked carrot	65.96 ± 2.56	0.28 ± 0.06	0.66 ± 0.02	5.91 ± 0.08	3.29 ± 0.45	7818 ± 130	4257 ± 50	< LOD
Raw chayote	10.97 ± 0.44	< LOD	0.33 ± 0.19	5.22 ± 2.22	5.19 ± 0.77	16591 ± 336	245 ± 11	1.25 ± 0.67
Cooked chayote	49.26 ± 2.60	0.28 ± 0.34	0.85 ± 0.35	2.45 ± 0.21	6.96 ± 0.27	10279 ± 336	1991 ± 39	1.12 ± 0.28
Raw rice	8.13 ± 0.15	< LOD	0.13 ± 0.07	4.17 ± 1.69	79.31 ± 2.01	1636 ± 9	29.82 ± 1.70	< LOD
Cooked rice	13.98 ± 3.81	0.23 ± 0.07	0.31 ± 0.15	3.98 ± 0.47	< LOD	1631 ± 9	24.64 ± 8.98	< LOD

^aMean value ± standard deviation (n = 3).

< LOD: Below limit of detection.

Normative Instruction of ANVISA n^o 160 (2022)⁴² determines the maximum tolerated limits (MTL) of micro-contaminants in food, including Cd and Pb levels for rice, carrots, and chayote. The maximum permitted concentration of Cd for rice, carrots, and chayote is 0.40, 0.10, and 0.05 mg kg⁻¹, respectively. The maximum levels of Pb are 0.20, 0.10, and 0.10 mg kg⁻¹ for rice, carrots, and chayote, respectively. It was observed that the Pb concentration was higher than the limit allowed by ANVISA, in the raw chayote sample and after cooking. The Cd concentration was also higher than allowed by law in the cooked chayote sample. In contrast, the raw carrot and rice samples contained concentrations below the tolerated limit.

In the case of iron in carrot and rice samples, a noticeable decrease in this metal can be observed in the samples after cooking. A study from 2016 evaluated the effects of peeling and cooking on the mineral content of carrots. The quantification of iron showed that both peeled and unpeeled carrots experience a decrease in iron content after cooking.⁴³ However, for the chayote vegetable, a slight increase of approximately 25% in the concentration can be observed. According to Perelló et al., the reduction depends on cooking conditions such as temperature, time, and cooking medium.⁴⁴

The Pb content was below the LOD in carrot and rice samples before and after cooking. The ingestion of lead by food is one of the forms of contamination. Lead can adhere to food if it is grown in contaminated soil or even irrigated with contaminated and thus reach the consumer. Different factors may have raised the levels of these metals. For example, soil that may be contaminated, water used in the cooking process, or using pesticides and fertilizers while growing food. A study carried out in the municipality of Vitória de Santo Antão (Pernambuco, Brazil) which evaluated the soil, plant, and irrigation water, showed that the levels of Fe and Mn were much higher than those found in reference soils in Pernambuco. The studied soils also showed contamination by the metals Cr, Pb, and Cd.⁴⁵

Among the determined analytes, it was observed that the cooking process increased the concentration of Al, Cd, and Cr. To find out whether the increase in the concentration of these metals came from the pan or water used in the cooking process, a study of the cooking blank was carried out. This study consisted of reproducing the conditions used during cooking (aluminum pan, 30 min, and 100 °C), but in the absence of samples. The results obtained are illustrated in Figure 2 (A, B, and C). With regard to Al, the analysis of the blank revealed a concentration of 0.867 mg L⁻¹, noting that the carrot was the sample most significantly affected by the presence of Al (Table IV). The significant presence of Al in the cooking blank was due to the pan used in the process, made of aluminum.

In Brazil, although there are no regulations on safe levels of Al in foods, Resolution No. 285 of May 21, 2019, prohibits using food additives containing Al in various food categories. The World Health Organization defined a Provisional Tolerable Weekly Intake of 2 mg kg body⁻¹.^{46,47} Thus, the value obtained does not exceed the tolerable level, but it is observed that the use of aluminum pans by the population is a possible medium of the increase in aluminum intake. Similar results were obtained by researchers who observed aluminum migration in the cooking process and that leaving food stored in aluminum containers leads to increased levels.⁴⁸

In relation to Cd and Cr (Figure 2), the concentrations measured in the cooking blank study were below LOD. These findings indicate that the origin of the Cd and Cr obtained in higher concentrations in cooked vegetables (Table IV) was not the aluminum pan. A possible source of these elements is the filtered water used during cooking.

Several aspects influence the increased transfer of metals found in kitchen utensils to the human diet. Examples include foods with a low pH, which play a significant role in the migration of these metals into the food, as well as the duration of pot use and cooking time. Additionally, according to one study, the nature of the original materials employed during the cooking process may also result in elevated levels of other metallic elements in the residues released by the cookware, such as Cd, Mn, and V.^{49,50}

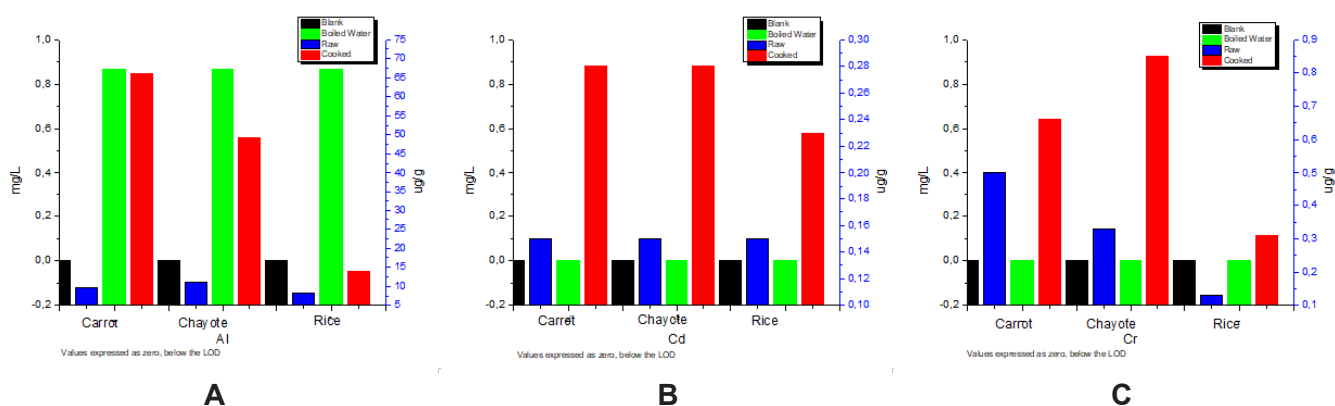


Figure 2. Concentrations of Al (A), Cd (B), and Cr (C) obtained in the study of the cooking blank for unboiled distilled-deionized water (blank) and boiled water, in mg L⁻¹, compared to the content of these elements in vegetables raw and cooked, in µg g⁻¹. Values expressed as zero on the plot were below the LOD.

As for Cu, the permitted limit is 10.0 mg kg⁻¹ for vegetables; therefore, the levels follow the legislation. However, loss of Cu, Fe, K, and Na after cooking was observed. It is known that Fe, K, and Na are considered essential for the proper functioning of the organism, so it is crucial to evaluate the conditions of food preparation, given that the cooking process goes far beyond the choice of condiments. Therefore, more attention should be paid to food preparation. Care must be taken in the use of household utensils and cooking water, as they may become contamination artifacts. It is necessary to highlight that consuming steamed food can be a viable way to avoid possible contamination, as well as the loss of essential minerals for the healthy functioning of the human body. The consumption of steamed foods has advantages such

as (i) less loss of nutrients, such as vitamins (mainly water-soluble ones, vitamin C and Complex B) and minerals; (ii) keeping fragile food intact; (iii) preservation of color, aroma, and natural texture of foods, among others.

Studies were realized to analyze the rice sample using diluted reagents for sample digestion (Table V). This feat becomes possible due to the nitric acid's regeneration process, with the presence of oxygen that allows the oxidation of the organic matter even if the concentration of HNO_3 is much lower than 14 mol L^{-1} .²⁸ No digestion procedures were found employing diluted nitric acid for the carrot and chayote samples.

Table V. Microwave-assisted digestion methods published in the scientific literature used diluted nitric acid in food preparation

Samples (number of samples collected)	Origin of samples	Digesting reagents	Analytes	Quantification method	Ref.
Rice: parboiled (n=10), white (n=8), and brown (n=5)	Salvador, Brazil	MWAD: 250 mg of sample; 7 mL of $4.5 \text{ mol L}^{-1} \text{HNO}_3$; 1 mL of 30% H_2O_2 .	Al, As, Ca, Cd, Cu, Fe, K, Mg, Mn, Mo, Se, and Zn	ICP OES and ICP-MS	51
Vegetable: Root (n=3), Stalk (n=2), Solanaceous (n=3), Melon (n= 4), Leafy (n=8), and legume (n=2)	Hunan Province	DIM: 1 g placed in a muffle and incinerated for 4 h at $480 \text{ }^\circ\text{C}$. Next, the sample was withdrawn from the oven and cooled. Then, 2 mL of $5 \text{ mol L}^{-1} \text{HNO}_3$ was added and evaporated to dryness in a heating plate. Then, the sample was placed in a oven and heated to $400 \text{ }^\circ\text{C}$ for 15 min before being removed (from the oven, cooled, and moistened with four drops of distilled water). Then, 2 mL of 37% HCl was added, and the sample was evaporated to dryness, removed, and then, 5 mL of $2 \text{ mol L}^{-1} \text{HCl}$ was added.	Pb, Cd, Cu, Zn, and total As	ICP OES and AFS	52
Carrot, potato lotus root, white radish, sweet corn, long grain rice, soybean, fleshy prawn, eastern oyster, kelp, and common carp	Guangzhou, China	MWAD: 0.5 g of sample, 12 mL of concentrated HNO_3 , and 4 mL of concentrated H_2O_2 .	Cd	ICP-MS	53
Samples of vegetable sausages (n= 27) and samples of vegetable burgers (n= 39)	Canary Islands, Spain	DIM: 7 g of the sample were dried in an oven at $70 \pm 5 \text{ }^\circ\text{C}$ for 24 h. Subsequently, 65% of HNO_3 was added and placed in a muffle at $400 \pm 25 \text{ }^\circ\text{C}$ for 24 h, with a progressive rise in temperature of $50 \text{ }^\circ\text{C/h}$. The white ashes achieved were dissolved in 1.5% HNO_3 until a final volume of 25 mL.	B, Ba, Co, Cr, Cu, Fe, Li, Mn, Mo, Ni, Sr, V, Zn, Al, Cd, and Pb	ICP OES	54
Meat, meat products, fish, vegetables (lettuce, tomatoes, cauliflower, green beans, onions, peppers, carrots, and eggplant), tubers, chicken eggs, milk, dairy products, soy, yogurt, cereals, legumes, oils and fats, and industrial baking. All samples (n=1)	Catalonia, Spain	MWAD: 0.10 g of sample, 3 mL of 65% HNO_3 , 3 mL of 30% H_2O_2 and 2 mL of ultrapure water.	As, Cd, Hg, and Pb	ICP-MS	55
Different baby food samples (n= 95)	São Paulo, Brazil	MWAD: A sample of 1 g of infant purée or 3 g of infant drink, 8 mL of 65% HNO_3 , and 2 mL 30% H_2O_2 .	Al	ICP OES	56

(continues on the next page)

Table V. Microwave-assisted digestion methods published in the scientific literature used diluted nitric acid in food preparation (continuation)

Samples (number of samples collected)	Origin of samples	Digesting reagents	Analytes	Quantification method	Ref.
88 samples from 26 different vegetables and derived products	Rio de Janeiro, Brazil	WD (Ra and Pb): 10 g of ash sample was digested with HNO ₃ and HCl and co-precipitated as Ba(Ra,Pb)SO ₄ by adding H ₂ SO ₄ and BaCl ₂ . WD (Th and U): 0.2 g of the sample ash was dissolved with 65% HNO ₃ . After 72 h of digestion, the samples were diluted (1:20) with water.	Th, U, Pb, ²²⁶ Ra, and ²²⁸ Ra	ICP-MS	57
Chayote (n = 92)	Argentina city (Bella Vista, Corrientes, Monte Caseros, and Santo Tome)	MWAD: 500 mg sample, 5 mL 65% HNO ₃ , and 2 mL of 30% H ₂ O ₂ .	Al, As, Ba, Ca, Cd, Co, Cu, Fe, K, Mg, Mn, Na, Pb, Sr, and Zn	ICP OES	58
Meat, meat products, fish, vegetables (lettuce, tomatoes, cauliflower, green beans, onions, peppers, carrots, and eggplant), tubers, chicken eggs, milk, dairy products, soy, yogurt, cereals, legumes, oils and fats, and industrial baking. All samples (n=1)	Catalonia, Spain	MWAD: 0.5 g of sample, 9 mL of 22% HNO ₃ , and 0.5 mL of 30% H ₂ O ₂ . Hg was directly determined by amalgamation with gold in an elemental mercury analyzer based on a sample amount of 0.1 g.	As, Cd, Hg, and Pb	ICP-MS	59

MWAD: Microwave-assisted acid digestion; DIM: Dry incineration method; WD: Wet Digestion; ICP-MS: Inductively coupled plasma mass spectrometry; ICP OES: Inductively Coupled Plasma Optical Emission Spectrometry.

Based on these data, the current work is the first that employed diluted nitric acid (0.5 mol L⁻¹) to prepare carrot and chayote samples for later elemental determination. The data contained in Table V show that the lowest concentration used was 4.5 mol L⁻¹ in the sample preparation of different foods.⁴⁹

CONCLUSIONS

In this work, a method of digesting rice, carrots, and chayote was proposed using diluted nitric acid, aiming to determine the multi-elemental composition of these foods. Defining a mineral composition for these foods before and after cooking was possible. The procedure employed diluted nitric acid (0.5 mol L⁻¹), which made the procedure economical and ecologically correct. The parameters to select the digestion conditions were low residual acidity and low dissolved carbon content. This method allowed digesting of the samples quickly and efficiently and subsequently quantifying the analytes by ICP OES. Through the method, it was possible to assess that cooking food is a possible factor in increasing the concentration of aluminum, cadmium, and chromium metals, as well as promoting nutritional losses.

Conflicts of interest

The authors declare no competing interests.

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