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Analytical Methods

# Determination of nutrients in sugarcane juice using slurry sampling and detection by ICP OES

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ABSTRACT

The fractional factorial and Doehlert designs for optimization of a slurry sampling procedure to determine of nutrients in sugarcane juice by inductively coupled plasma optical emission spectrometry (ICP OES) were applied. External calibration curves were used for direct analysis of the slurry. This procedure allowed determination of Ca, Cu, Fe, K and Mg with limits of detection (LoD) obtained of 2.0, 0.04, 0.2, 1.0 and 1.5 mg $L^{-1}$, respectively. The precision was expressed as relative standard deviation (%RSD), being better than 1.4% (n = 3). Accuracy was confirmed by comparison with sample digestion method. The results for analysis of fourteen sugarcane juices samples demonstrated that the nutrients Ca, Cu, Fe, K and Mg have average contents of 108, 0.506, 6.40, 470 and 114 mg $L^{-1}$, respectively. The proposed analytical method is a good alternative for simultaneous determination of nutrients in sugarcane juice using introduction of slurries and detection by ICP OES.

## 1. Introduction

Sugarcane (*Saccharum officinarum* L.) had been cultivated in about 200 countries, being an important energy matrix in tropical and subtropical countries, such as Brazil. China, India, Thailand, and Australia, together with Brazil, are the world's largest sugarcane producer, accounting for 25% of total production (Brochier, Mercali, & Marczak, 2016; Ramasubramanian, Paramasivam, Jayanthi, & Chandrasekaran, 2014; Sindhu, Gnansounou, Binod, & Pandey, 2016; Suganthi, Bhuvaneswari, & Ramya, 2018; Zuin et al., 2006). From sugarcane, a variety of products can be obtained, such as sugarcane spirits (*cachaças*), sugar and sugarcane juice, etc. Its standardized extract, sugarcane juice, is a non-alcoholic energy drink that is widely consumed in Brazil (Brazil, 2005a; Brochier et al., 2016; Nogueira, Ferreira, Carneiro Junior, & Passoni, 2009; Serafim, Pereira-Filho, & Franco, 2016; Zuin et al., 2006). The juice is a natural source of nutrition, it is important to assess the mineral composition of this beverage (Nogueira et al., 2009; Tormen et al., 2011)

In Brazil, the National Health Surveillance Agency (*Agência Nacional de Vigilância Sanitária* – ANVISA) through the Resolution N° 269 of September 22, 2005, establishes the recommended daily intake (RDI) of minerals for adults, infants, pregnant women, infants aged 0–6 months and 7–11 months, children aged 1–3 years, 4–6 years and 7–10 years. For Ca, the recommended is 1000, 1200, 1000, 300, 400, 500, 600, and 700 mg $day^{-1}$, respectively. For Cu, is 900, 1000, 1300, 200, 220, 340, 440, and 400 mg $day^{-1}$, respectively. For Fe, mass of 14, 27, 15, 0.27, 9, 6, 6, and 9 mg $day^{-1}$, respectively. And for Mg, is 260, 220, 270, 36, 53, 60, 73, and 100 mg $day^{-1}$, respectively. For K, there are no recommended values (Brazil, 2005b). The Decree N° 55,871 of March 26, 1965, Ordinance n° 685 of August 27, 1998 and RDC N° 42 of August 29, 2013 establish maximum limits in non-alcoholic beverages and concentrations for toxics inorganic components, of the elements studied, only copper has established value (30.0 mg $kg^{-1}$) (Brazil, 1965c; Brazil, 1998d; Brazil, 2013e).

Conventionally, atomic spectrometry techniques have been used for analyses of food samples, being requires pre-treatment for complete destruction of organic matter. However, the slurry sampling offer simplification of the sample preparation, a low reagent consumption, minimizes risk of sample contamination and analyte losses from the sample pre-treatment (Amorim et al., 2017; de Andrade, de Brito, dos

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Anjos, & Quináia, 2018).

Slurry sampling procedures employing several spectroanalytical techniques has been used for the determination of the constituent inorganics in food samples. Tai, Jiang, and Sahayam (2016) employed slurry sampling and flow injection analysis (FIA) and chemical vapor generation inductively coupled plasma mass spectrometry (FIA-CVG-ICP –MS) for the determination of As, Hg and Pb in herbs. Amorim et al. (2017) applied slurry sampling and detection by flame atomic absorption spectrometry (F AAS) for the determination of Fe and Mg in cassava starch samples. De Oliveira, Peres, Felsner, and Justi (2017) employed slurries for direct analysis of Pb in raw milk by graphite furnace atomic absorption spectrometry (GF AAS). De Andrade et al. (2018) used slurry sampling and GF AAS for the determination of Cd, Cr, Cu and Pb in yogurt samples.

Several analytical methods also have been employed to determine the composition and possible contaminants in non-alcoholic beverage (Daniel, Santos, Vidal, & Lago, 2015; Jandrić and Cannavan, 2015; Ramasubramanian et al., 2014; Szymczycha-Madeja, Welna, Jedryczko, & Pohl, 2014; Zuin et al., 2006; Sousa, Silva, Baccan, & Cadore, 2005; Santos et al., 2014).

Tormen et al. (2011) and Szymczycha-Madeja & Welna (2013) determined several elements in fruit juices by inductively coupled plasma mass spectrometry (ICP-MS) and inductively coupled plasma optical emission spectrometry (ICP OES), respectively, after simple pre-treatment, and compared their finding against the wet digestion procedure. They realized that the simple pre-treatment procedure was sensitive for determining the composition of the inorganic types of the sample since the concentrations were consistent with the digestion procedure.

Nogueira et al. (2009) determined minerals in sugarcane juice. In this study, the samples were calcined and then solubilized with $HNO_3$ or $HClO_4$ solution. Calcium, Cu, Mg, Mn, and Zn were determined by F AAS, and P concentration was determined by colorimetry analysis.

The advent of chemometric tools has altered the concepts of experimental models, reducing costs and minimizing environmental impacts caused by chemical agents (Kumar, Bansal, Sarma, & Rawal, 2014; Ferreira, 2015; Novaes et al., 2016). Given the multiple factors involved in experimental design set-up, full factorial design is usually unviable, and fractional factorial design is recommended, causing increase of experiments due to the number of factors. (Novaes et al., 2016; Ferreira, 2015; Momen, Zachariadis, Anthemidis, & Stratis, 2007). By fractional factorial design, the number of experiments can be halved, without losing information on the performance of the factors in the process or composition of the response, providing an efficient experimental design is used (Novaes et al., 2016; Reinholds, Bartkevics, Silvis, Van Ruth, & Esslinger, 2015; Ferreira, 2015).

After the evaluation of the factors and their experimental domain, chemometric tools are applied that generate further details on the behavior of variables (Ferreira, 2015). For example, the Doehlert matrix enables design involving a different number of levels and displays high efficiency in terms of the number of experiments needed for optimization (Ferreira, 2015; Novaes et al., 2016; Teófilo & Ferreira, 2006).

Santos et al. (2014) applied factorial and Dohelert designs for the optimization of an analytical method to determine the micronutrient and mineral content of coconut milk by ICP OES following a simple ultrasound-assisted extraction procedure. The procedure was validated by comparing with the sample digestion method and proved to be simple, fast, and efficient. Costa et al. (2013) used experimental design methodology for optimize an analytical method for determination of the mineral element composition of dog and cat foods by ICP OES. Two- and three-level factorial designs were applied to define the optimal proportions of the reagents for microwave-assisted sample digestion and optimize the operational conditions of the spectrometer, respectively.

Considering the importance in the food safety, this work had aim to optimized an analytical method for simultaneous multielementary determination of Ca, Cu, Fe, K, and Mg in sugarcane juice *in natura* using slurry and detection by ICP OES, i.e., after simple pre-treatment.

## 2. Experimental

### 2.1. Instrumentation

The analysis was carried out by inductively coupled plasma optical emission spectrometer with axial configuration (model ES-720, Varian, Mulgrave, Australia) for simultaneous multielementary determination of Ca, Cu, Fe, K and Mg in sugarcane juice.

Operating parameters for spectrometer were as follows: 1200 W of radio frequency (RF) power, 1.0 L $min^{-1}$ of nebulizer gas (NG) flow rate, 15 L $min^{-1}$ of plasma flow rate, 1.5 L $min^{-1}$ of auxiliary gas flow rate, and 0.8 L $min^{-1}$ of nebulization gas flow rate. The solutions (or slurries) were introduced into the plasma using a V-groove nebulizer and Sturman-Masters chamber. Analytical lines of Ca (II) 373.690, Cu (I) 324.350, Fe (II) 234.350, K (I) 766.491, Mg (I) 285.213, Mg (II) 280.265, and Mg (II) 279.800 nm were measured, where (I) is atomic line and (II) is ionic line. Argon gas with a minimum purity of 99.999% (White Martins, São Paulo, Brazil) was used to purge the optics and plasma generation.

The slurry procedure was performed in an ultrasonic bath model Unique UltraCleaner 800A (Unique, São Paulo, Brazil) under optimized conditions. The closed digester block (model TE007A, TECNAL, São Paulo-SP, Brazil), was performed for digestion of the sugarcane juice samples as comparative method.

### 2.2. Reagents, solutions, and samples

This work, all reagents used were analytical grade. All dilutions were performed using deionized water with resistivity 18.2 MΩ $cm^{-1}$ obtained from a Milli-Q system (Millipore, Bedford, USA). All glassware used was previously decontaminated by immersion in a 10% (v $v^{-1}$) $HNO_3$ solution for 24 h and rinsed with deionized water before use.

Nitric acid 65% (m $m^{-1}$) suprapure quality ($HNO_3$, Merck, Darmstadt, Germany) was purified using a sub-boiling distiller apparatus with quartz tube (Kürner Analysentechnick, Rosenheim, Germany) and hydrogen peroxide 30% m $m^{-1}$ ($H_2O_2$, Merck, Darmstadt, Germany) was used to prepared of slurry and digestion samples.

Standard solutions for addition and recovery tests were prepared by suitable dilution of the stock solutions containing 1000 mg $L^{-1}$ of Ca, Cu, Fe, K, and Mg (Titrisol, Merck, Darmstadt, Germany). External calibration curves were also performed using a standard solution (Titrisol, Merck, Darmstadt, Germany) containing 1000 mg $L^{-1}$ of Ca, Cu, Fe, K, and Mg, being diluted according to the working ranges required.

Fourteen sugarcane juice samples were collected from Trades Street in Aracaju City, Sergipe State, Northeast, Brazil on the same day of the analysis and used to evaluate the slurry procedure.

### 2.3. Procedures

#### 2.3.1. Sugarcane juice slurry procedure

The slurries were prepared using 5.0 mL of sugarcane juice sample, immediately after arrival at the laboratory to avoid sample degradation. This aliquot of the sugarcane juice sample was added in tube of 15 mL, followed by 870 μL of $HNO_3$ (65% m $m^{-1}$), 700 μL of $H_2O_2$ (30% m $m^{-1}$), and complete with deionized water up to a total volume of 10 mL. This mixture was then placed in an ultrasonic bath at room temperature for 7 min 40 s and then introduced directly into the ICP OES. This sample preparation and analysis procedure was optimized using Doehlert design. The blank analytical solutions were performed with same procedure as quality control of the reagents, replace the volume of sugarcane juice sample by deionized water (5.0 mL). The analyses were performed in triplicate.

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