



Analytical Methods

Multivariate optimization of simple procedure for determination of Fe and Mg in cassava starch employing slurry sampling and FAAS



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ABSTRACT

A slurry sampling procedure has been developed for Fe and Mg determination in cassava starch using flame atomic absorption spectrometry. The optimization step was performed using a univariate methodology for 200 mg samples and a multivariate methodology, using the Box-Behnken design, for other variables, such as solvent (HNO₃:HCl), final concentration (1.7 mol L⁻¹) and time (26 min). This procedure allowed determination of iron and magnesium with detection limits of 1.01 and 3.36 mg kg⁻¹, respectively. Precision, expressed as relative standard deviation (%RSD), was of 5.8 and 4.1% (n = 10) for Fe (17.8 mg kg⁻¹) and Mg (64.5 mg kg⁻¹), respectively. Accuracy was confirmed by analysis of a standard reference material for wheat flour (NIST 1567a), which had certified concentrations of 14.1 ± 0.5 mg kg⁻¹ for Fe and 40 ± 2.0 mg kg⁻¹ for Mg, and the concentrations found using proposed method were 13.7 ± 0.3 mg kg⁻¹ for Fe and 40.8 ± 1.5 mg kg⁻¹ for Mg. Comparison with concentrations obtained using closed vessel microwave digestion was also realized. The concentrations obtained varied between 7.85 and 17.8 mg kg⁻¹ for Fe and 23.7–64.5 mg kg⁻¹, for Mg. The simplicity, easily, speed and satisfactory analytical characteristics indicate that the proposed analytical procedure is a good alternative for the determination of Fe and Mg in cassava starch samples.

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1. Introduction

Cassava (*Manihot esculenta crantz*) is a staple food in the diet of populations in developing countries, especially those with low incomes (FAO, 2015). It is used mainly because of its availability, low cost and the versatility of products derived from the root, including starch. Cassava starch is a rich source of fiber, carbohydrates, minerals and vitamins. The minerals contained in cassava, specifically iron and magnesium, participate in important biological processes (Montagnac, Davis, & Tanumihardjo, 2009).

Iron and magnesium are essential elements for humans, animals, plants and microorganisms. However, as for all essential minerals, an excess or deficiency can result in chronic or acute disorders (Stein, 1994). Iron is well known for its role in oxygen transport but it is also an essential component of hundreds of enzymes, many of which are involved in energy metabolism. While having a role in brain development and the immune system, deficiency of iron, whether from inadequate intake or decreased absorption, is

the primary cause of nutritional anemia, the most common nutritional deficiency in the world (Nielsen, 1999; Whittaker, 1998).

Magnesium is an essential for more than 300 important biological processes, including the production of ATP. It is present in the body largely in combination with calcium and phosphorus in bones. The remainder is distributed among tissues, functioning as control agent in various chemical reactions that make up the metabolism. To perform this function, magnesium is as an activator of enzymes that function in energy production and proteins that make up the raw material of tissues (Nielsen, 1999).

Preparation of food samples for atomic spectrometry analyses requires pre-treatment of samples involving the complete destruction of organic matter. In order to do that, the laborious procedures involving digestion with use of concentrated acids, high temperatures, and high pressures are used most (Taylor et al., 2014). Slurry sampling appears to be an attractive alternative due to simplification of the sample pre-treatment, decreased operational work and analysis time, low risk of sample contamination and minimization of analyte losses caused by pre-treatment (Ferreira et al., 2010; Magalhães & Arruda, 1998; Vale, Oleszczuk, & Santos, 2006).

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Table 1
Applications of slurry sampling for metals determination in food samples.

Sample	Analyte	Technique	Diluent	Reference
Chocolate	Mn and Zn	FAAS	HNO ₃ (2.0 mol L ⁻¹)	Silva et al. (2006)
Wheat flour	Mn	FAAS	HNO ₃ (2.0 mol L ⁻¹)	Araujo et al. (2007)
Seafood	Cu, Mn, and Fe	FAAS	HNO ₃ (0.5 mol L ⁻¹) + HCl (0.5 mol L ⁻¹)	Silva et al. (2008)
Tea leaves	Zn and Mn	FAAS	HNO ₃ (1.0 mol L ⁻¹) + HCl (2.0 mol L ⁻¹) and Triton X-100 (2.5%)	Bezerra et al. (2010)
Cassava Leaves	Fe	FAAS	HNO ₃ (0.2 mol L ⁻¹) + HCl (3.6 mol L ⁻¹) + H ₂ O ₂ (5%)	Dias et al. (2011)
Milk	Mo	GF AAS	Dilution in ultrapure water	Amorim et al. (2011)
Dairy products	Ca and Mg	HR-CS FAAS	HCl (2.0 mol L ⁻¹)	Brandão et al. (2011)
Sweeteners	Ca, Cd, Cu, Co, Fe, Mg, Mn, Na, Ni, Pb, Se and Zn	ICP OES	HNO ₃ (0.3% v/v)	Sousa et al. (2011)
Rice	Hg	CV AAS	HCl (6.0 mol L ⁻¹)	Silva et al. (2012)
Food	Cr	GF AAS	HNO ₃ (5% v/v)	Dobrowolski et al. (2012)
Herbs	As, Hg and Pb	ETV-ICP-MS	C ₆ H ₈ O ₇ (0.1% m/v) + HCl (0.2% v/v)	Lin and Jiang (2013)
Rice	As	HG AFS	HCl (1.5 mol L ⁻¹) + KI (10%) + C ₆ H ₈ O ₆ (2%)	Santos et al. (2013)
Honey	Cd, Pb and Cr	GF AAS	HNO ₃ (1.0 mol L ⁻¹) + H ₂ O ₂ (3%)	Andrade et al. (2014)
Starch	Fe and Zn	FAAS	HNO ₃ (1.0 mol L ⁻¹) + HCl (1.0 mol L ⁻¹)	Amorim et al. (2015)

Slurry sampling procedures employing atomic spectrometric techniques, such as source graphite furnace atomic absorption spectrometry (GF AAS) (Andrade, Anjos, Felsner, Torres, & Quinãia, 2014; Dobrowolski, Kapusta, & Dobrzynska, 2012), flame atomic absorption spectrometry (FAAS) (Almeida et al., 2013; Araujo, Dias, Macedo, Santos, & Ferreira, 2007; Brandão, Matos, & Ferreira, 2011), inductively coupled plasma optical emission spectrometry (ICP OES) (Dados, Kartsiouli, Chatzimitakos, Papastephanou, & Stalikas, 2014; Santos et al., 2010; Sousa, Baccan, & Cadore, 2011), hydride generation atomic absorption spectrometry (HG AAS) (Macedo et al., 2010; Silva et al., 2014), cold vapor atomic absorption spectrometry (CV AAS) (Silva, Silva, Leão, Matos, & Ferreira, 2012), electrothermal atomic absorption spectrometry (ET AAS) (Amorim, Franco, Nascentes, & Silva, 2011; Souza, Silva, Kelmer, & Oliveira, 2013), electrothermal vaporization inductively coupled plasma mass spectrometry (ETV-ICP-MS) (Hsu, Jiang, & Sahayam, 2013; Lin & Jiang, 2013), hydride generation atomic fluorescence spectrometry (HG AFS) (Santos, Cavalcante, Macedo, Nogueira, & Silva, 2013) and others, have been proposed for analyses of a variety of samples (Vale et al., 2006). However, FAAS is the most simple and economic technique (Amorim, Lobo, Santos, & Ferreira, 2008). Several studies have been reported in the literature that describe development of procedures based on application of suspension samples, as a sample preparation step for determination of metals. The details of some works that applied slurry sampling for food analyses are presented in Table 1.

Multivariate techniques have been applied widely in the optimization of analytical chemistry methods (Barros et al., 2013; Costa et al., 2013; Kumar, Bansal, Sarma, & Rawal, 2014; Santos et al., 2014; Souza et al., 2014; Tarley et al., 2009). Among these, the Box-Behnken design (Ferreira et al., 2007) is among those used most (Kyung-Won, Min-Jin, Min-Jung, & Kyu-Hong, 2015; Li, Fang, & You, 2013; Roosta, Ghaedi, & Daneshfar, 2014; Santos et al., 2009; Trindade, Dantas, Lima, Ferreira, & Teixeira, 2015) but its use in slurry sampling has not been explored sufficiently.

In this paper, the Box-Behnken design was used for multivariate optimization of a simple slurry sampling procedure for cassava starch samples for multi-element determination of iron and magnesium using FAAS, which has not been reported previously.

2. Experimental

2.1. Instrumentation

A Varian model SpectrAA 240FS (Mulgrave, Victoria, Australia) flame atomic absorption spectrometer equipped with an air-

acetylene burner was used to measure absorbance. Hollow cathode lamps (Varian instruments) were used as a light source, at 248.3 nm and 285.2 nm for Fe and Mg, respectively. Deuterium lamp background correction was also used. The flame composition was acetylene (flow rate: 2.0 L min⁻¹) and air (flow rate: 13.5 L min⁻¹). Nebulizer flow rate was 5.0 mL min⁻¹. An ultrasonic bath Unique, Model USC – 1800 (Cortland, Nova York, USA) was used for preparation of the slurry. The ultrasound bath equipment was filled with 2.8 L of water and volumetric flasks were placed in the bath where more intense cavitation had been mapped previously (Nascentes, Korn, Sousa, & Arruda, 2001). A Sartorius model BL D105 (São Paulo, Brazil) analytical balance was used to determine sample mass. Digestion of cassava starch samples was carried out with a microwave oven (Master Sineo Model MDS 40, Shanghai, China). A Quimis T 222 QM centrifuge (São Paulo, Brazil) with 50 mL centrifuge tubes was used to separate solids from the liquid-phase in some experiments. The particle size was measured using a Shimadzu Superscan SS-550 scanning electron microscope (Kioto, Japan).

2.2. Reagents and solutions

All chemicals and reagents used in this study were of analytical reagent grade. Deionized water was used to prepare all solutions. Laboratory glassware was kept overnight in 10% v/v nitric acid solution and, before use, the glassware was rinsed with deionized water and dried in a dust free environment. Iron and magnesium solutions (100.0 mg L⁻¹) were prepared by diluting a (1000 mg L⁻¹) solution (Merck, Darmstadt, Germany). Nitric and hydrochloric acid solutions were prepared by dilution with water from the concentrated acids.

2.3. Optimization of the procedure

The optimization for iron and magnesium in cassava starch using slurry sampling using univariate studies for mass and a Box-Behnken design for the type of extractor (TE), its final concentration (CE) and time (TS). The variables and concentrations used in the multivariate optimization are presented in the Supplementary material. Responses for experimental designs were obtained by evaluating recovery using as a reference the values obtained after microwave assisted digestion with HNO₃ and H₂O₂ (concentrated). The data analysis was performed using the Statistica 8.0 software.

To allow simultaneous optimization of the responses obtained, a mathematical approach developed by Derringer and Suich was used (Derringer & Suich, 1980). It is based on desirability functions applied in optimizing multi-response experiments. The approach

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