



Microwave-assisted diluted acid digestion for trace elements analysis of edible soybean products



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ABSTRACT

A new method for the decomposition of soybean based edible products (soy extract, textured soy protein, transgenic soybeans, and whole soy flour) was developed to essential (Co, Cr, Cu, Fe, Mn, Ni, Se, V, and Zn) and non-essential (As, Ba, Cd, Pb, and Sr) trace elements determination by ICP OES and ICP-MS respectively. Effects related to the concentration of HNO_3 (2.1–14.5 mol L⁻¹) and the use of hydrogen peroxide on the efficiency of decomposition was evaluated based on the residual carbon content (RCC). It was demonstrated that 2.1 mol L⁻¹ HNO_3 plus 1.0 mL H_2O_2 was suitable for an efficient digestion, since RCC was lower than 18% and the agreement with certified values and spike recoveries were higher than 90% for all analytes. The concentrations of analytes in the samples (minimum–maximum in mg kg⁻¹) were: As (<0.007–0.040), Ba (0.064–10.6), Cd (<0.006–0.028), Co (0.012–102), Cr (0.56–5.88), Cu (6.53–13.9), Fe (24.9–126), Mn (16.4–35.2), Ni (0.74–4.78), Se (<2.90–25), Sr (2.48–20.1), Pb (<0.029–0.11), V (<0.027–20), and Zn (30.1–47.3). Soy-based foods investigated in this study presented variable composition in terms of essential and potentially toxic elements, which can be attributed to different methods of processing.

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1. Introduction

Soybeans are grown for their seeds that are rich in nutritional ingredients, such as proteins (40%), oils (20%), carbohydrates (35%) and essential elements. Due to the increased production of soy-based foods, the determination of trace elements is important in view of toxicity and nutritional assessments (Llorent-Martinez, Fernandez de Cordova, Ruiz-Medina, & Ortega-Barrales, 2012; Yip, Chan, Cheung, Poon, & Sham, 2009). There are previous studies related to the contents of essential and non-essential elements in different species of soybeans and related products in some countries (Fernandez et al., 2002; Hseu, 2004; Karr-Lilienthal, Griesshop, Merchen, Mahan, & Fahey, 2004; Kazi, 2009; Liu, Wang, Liu, Zhao, & Zhan, 2004; Sola-Larranaga & Navarro-Blasco, 2009; Souza, 2005; Wei, Shen, Rui, & Jiao, 2008; Yip et al., 2009). However, despite the large consumption, there are few literature reports regarding the trace elements contents in soybeans produced in Brazil (Carvalho et al., 2011; Gonzalez et al., 2009; Gris

et al., 2008; Mataveli, Pohl, Mounicou, & Arruda, 2010; Sussulini, Souza, Eberlin, & Arruda, 2007; Zhou et al., 2011).

The determination of trace elements in food requires sensitive analytical techniques such as inductively coupled plasma mass spectrometry (ICP-MS) and inductively coupled plasma optical emission spectrometry (ICP OES). However, results obtained employing these techniques may be affected by matrix and/or spectral interferences when working with solutions containing elevated concentrations of dissolved organic compounds. Furthermore, usually before performing measurements by ICP-MS or ICP OES, food samples must be decomposed with suitable methods leading to digests containing reduced amounts of residual carbon compounds and low residual acidity (Korn et al., 2008; Nardi et al., 2009).

Microwave-assisted digestion procedures using closed vessels and various acid mixtures have been employed to decompose organic matrices of biological samples at elevated temperatures and/or pressure. It was already demonstrated that microwave-assisted sample digestion accelerates and minimises contamination and losses of volatile elements (Bakkali, Ramos Martos, Souhail, & Ballesteros, 2009; Cindrić et al., 2012; Guo et al., 2012;

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Millour et al., 2011; Phan-Thien, Wright, & Lee, 2012; Poitevin, 2012; Shaltout et al., 2011, Zanariah, Ngah, & Yahya, 2012).

Nowadays, one of the most important trends in solid sample preparation for elemental analysis is the search for environmentally and friendly processes. Based on green chemistry recommendations, the use of diluted acids for digestion leads to relevant advantages such as cost reduction, production of smaller amounts of residues and reduction of blank values. In this sense, a number of recent applications demonstrated the potential of diluted nitric acid solutions in microwave-assisted digestion (Araujo, Gonzalez, Ferreira, Nogueira, & Nóbrega, 2002; Bizzi, Flores, Picoloto, Barin, & Nóbrega, 2010; Bizzi and Barin et al., 2011; Bizzi and Flores et al., 2011; Bressy, Brito, Barbosa, Teixeira, & Korn, 2013; Castro et al., 2009; Gonzalez et al., 2009; Trevizan, Donati, Nogueira, & Nóbrega, 2006). This approach was recently applied for digestion of black, white, and kidney beans followed by determination of Ba, Ca, Co, Cu, Fe, K, Mg, Mn, Mo, Ni, P, Pb, Se, Sr, and Zn by ICP OES (Santos, Santos, Fernandes, Castro, & Korn, 2013). However, no additional study was performed for evaluating the use of diluted nitric acid for samples containing relatively high oil and protein contents such as soybeans. Castro et al. (2009) proposed a microwave-assisted digestion procedure using diluted solutions ($2 \text{ mol L}^{-1} \text{ HNO}_3$ and H_2O_2) for multielement determination of macro and micro-elements in coffee samples by ICP OES. In another work, the use of diluted nitric acid associated with pressurised oxygen atmosphere was proven to be feasible for milk powder digestion, reducing the volume of reagents and the amount of digestion residues (Bizzi and Barin et al., 2011; Bizzi and Flores et al., 2011). More recently, a procedure using diluted nitric solutions for the decomposition of bean samples was applied for the determination of Ba, Ca, Co, Cu, Cr, K, Mg, Mn, Mo, Ni, P, Pb, Se, Sr, and Zn by ICP OES (Santos et al., 2013). In this work, three experimental factors (acid concentration, temperature, and processing time) were used to select the best conditions for digestion. Results were consistent with previous studies reported in literature where the digestion time and temperature had a significant effect on the residual concentration of nitric acid (Saavedra, Gonzalez, Fernandez, & Blanco, 2004). In all these works, digestion performance was evaluated taking into account the recovery of the analytes, residual carbon content (RCC) and residual acidity (RA) in the final solutions compared to reference values obtained after microwave-assisted acid digestion using concentrated acid.

Taking into account the difficulties involved in soybean digestion due to the high content of proteins and fats, the aim of this study was to optimise and validate a microwave-assisted digestion procedure for determination of essential (Co, Cr, Cu, Fe, Mn, Ni, Se, V, and Zn) and non-essential (As, Ba, Cd, Pb, and Sr) trace elements in soybeans and their products (soy extract, textured soy protein, and whole soy flour) using diluted nitric acid solution.

2. Materials and methods

2.1. Instrumentation

For microwave-assisted acid digestion using concentrated and diluted acid, a closed-vessel microwave digestion system with control of sensor pressure and temperature (ETHOS EZ, Milestone, Sorisole, Italy) was used. This microwave system was equipped with 10 vessels made of perfluoroalcoxi polymer (PFA) with a volume of 100 mL.

An inductively coupled plasma optical emission spectrometer with axial viewing – ICP OES (Vista Pro, Varian, Mulgrave, Australia) equipped with solid state detector, cyclonic spray chamber, and concentric nebulizer was employed for analytes (Ba, Cr, Cu, Fe, Mn, Ni, Sr, and Zn) and RCC determination. The analytes determination was carried out under manufacturer recommended

conditions for radiofrequency applied power (1.3 kW), plasma (15.0 L min^{-1}), auxiliary (1.5 L min^{-1}) and nebulizer (0.7 L min^{-1}) gas flow-rates. The emission lines less affected by spectral interferences and with high intensities and background ratios were selected. The analytical wavelengths (nm) chosen were: Ba II 455.403, Cr II 267.716, Cu I 324.754, Fe II 238.204, Mn II 257.611, Ni II 231.605, Sr II 407.771, Zn I 213.857, and C I 193.025 nm.

A quadrupole ICP-MS XseriesII (Thermo, Germany) equipped with a hexapole collision cell (CC) was used for the determination of analytes in lower concentrations (As, Cd, Co, Cr, Pb and V). The instrument software allows a rapid switch between the standard mode (no gas cell vented to mass analyser chamber) and the CC mode, whilst continuously aspirating the sample. The pre-mixed gases H_2 (7% v/v) in He (H_2O and other impurities < 5 mg/kg) were admitted into CC under rigorous flow control. The measurements were made using nickel sampler and skimmer cones (1.0 mm and 0.7 mm diameter orifices) and standard concentric nebulizer. A glass impact bead spray chamber cooled to 4 °C by a Peltier device and a shielded Fassel torch was used to minimise the plasma potential and thereby obtain a low and narrow initial ion energy distribution. The operating conditions were: radiofrequency applied power of 1.3 kW, nebulizer gas flow-rate of 0.87 L min^{-1} , plasma gas flow-rate of 13 L min^{-1} , auxiliary gas flow-rate of 0.70 L min^{-1} , CCT mode gas flow-rate, 6.5 L min^{-1} ; dwell time 10 ms, sweeps 100, and three replicate readings. Internal standards (Tl, In, Bi, Ge, Rh and Sc) were added to compensate acid effects and instrument drift (calibration concentrations were $0.1\text{--}25 \mu\text{g L}^{-1}$). Measurements were performed using ^{75}As , ^{111}Cd , ^{59}Co , ^{52}Cr , ^{208}Pb , and ^{51}V isotopes.

2.2. Reagents and standards

All reagents used in this work were of analytical grade (Merck, Darmstadt, Germany). Water obtained from a Milli-Q system (Millipore, Billerica, MA, USA) was used for preparing all reagents and standard solutions. Reference analytical solutions for ICP OES and ICP-MS analysis were prepared before use by serial dilution of stock reference solutions containing 1000 mg L^{-1} of each analyte (Multielement solution, Fluka, Buchs, Switzerland) in water.

The RCC determination was performed using yttrium as internal standard (Spex CertiPrep) in a final concentration of 1 mg L^{-1} in digests. Reference solutions for the calibration curve of C were prepared in the range of $25\text{--}500 \text{ mg L}^{-1}$ through serial dilution of a carbon stock reference solution (citric acid in water, $10,000 \text{ mg L}^{-1}$) (Bizzi et al., 2010; Gouveia, Silva, Costa, Nogueira, & Nóbrega, 2001).

2.3. Samples

Commercial soybean non-transgenic samples (named SG, $n = 4$), transgenic samples (named SGT, $n = 2$) and respective products (soy extract (named ES, $n = 3$), textured soy protein (named PTS, $n = 11$), and whole soy flour (named FS, $n = 2$) were purchased from different local markets of Salvador, Bahia, Brazil. All samples were ground in a ball mill, model 8000 M (Spex Sample Prep, Metuchen, NJ, USA) with a set of tungsten carbide vial and ball and sieved in Nylon[®] sieves to particle sizes lower than $500 \mu\text{m}$.

A certified reference material from National Institute of Standards and Technology (NIST, Gaithersburg, MD, USA) of rice flour (NIST 1568a) was used for the evaluation of accuracy since there is no CRM with similar matrix composition available with certified values for metals in soybeans.

2.4. Microwave-assisted digestion of soybeans and derivatives

Microwave-assisted digestion was employed using 250 mg of sample previously ground in a ball mill (model 8000 F, Sample

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