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Optimization of Focused-Microwave-Assisted Digestion of Bean Samples and Multielement Determination Using Inductively Coupled Plasma Optical Emission Spectromerty

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Legumes are an important source of proteins, complex carbohydrates, some minerals and vitamins. Bean (*Phaseolus vulgaris, L.*) is a legume of great importance and a principal source of dietary protein in the typical diet of the Brazilian population [1]. In this study, a focused-microwave-assisted oven digestion procedure of Fradinho bean (*Vigna ungriculata*), a typical legume used in Bahia-Brazil for a traditional regional food, was optimized. The multielementar determination of Al, Ba, Ca, Fe, Mg, Mn, and Zn was made using an inductively coupled plasma optical emission spectrometer (ICP OES) with axial or radial viewing configuration. Four different digestion strategies were employed to dissolve the sample: microwave-assisted digestion procedures using a focused- and cavity-oven, and others two procedures using conductive heating employing digester block with open vessels and digestion in a Parr bomb. In the focused microwave oven was used HNO_3:H_2SO_4:H_2O_2. Cavity-microwave oven and Parr bomb digestion procedures used a mixture of HNO_3:H_2O_2, and in the digester block with open vessel a mixture of HNO_3:$HClO_4$ was employed. Accuracy was checked using a standard reference material (NIST SRM Corn Bran 8433). Results are in agreement with 95% confidence level according to *t*-Student test. A 200 mg sample was weighed and 2 mL of HNO_3 or 3 mL HNO_3 plus 1 mL H_2O_2 were added in the procedures developed in cavity-oven and Parr bomb, respectively. In open vessel conductive heater, a 250 mg of sample was dissolved in 4 mL of HNO_3 and, after 12 hour, 2 mL of $HClO_4$ were added. For digestion procedure using focused-microwave oven, a factorial design and a Doehlert matrix optimization were applied to evaluate the digestion conditions. The investigated variables to construct the factorial design were: nitric acid volume, sulfuric acid volume, and temperature. About 500 mg of sample mass and an acid mixture of HNO_3:H_2SO_4:H_2O_2 were evaluated. The heating program was performed in 25 min and the reagents acid volumes were changed according to factorial design. The experimental results were processed according to P-values when the analysis of variance (ANOVA) was used. Results presented acid volumes and temperature as significant variables. Percentual recoveries were major than 80% in comparison with elemental concentrations obtained in digestates produced by microwave cavity-oven.

[1]. C.A.S. Pereira, N.M.B. Costa, Rev. Nutr.,Campinas, 15:5-14, 2002.

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