**A comparative approach of simple green sample preparation methods based on optimization strategies for nutrient analysis of food**

Cora Jofré F(1,2), Giacomino V(1), Perez E(1), Rivera F(1), Wagner M(1,2), Zaldarriaga J(1,2), Montemerlo A(2), Camiña J(1,2), Azcarate S(1,2), Savio M(1,2)

(1) Facultad Ciencias Exactas y Naturales, Universidad Nacional de La Pampa, Av.Uruguay 151, (6300) Santa Rosa, La Pampa, Argentina. Tel: 02954-245220 int 7433

(2) Instituto de Ciencias de la Tierra y Ambientales de la Pampa (INCITAP-CONICET-UNLPam), Santa Rosa, La Pampa, Argentina.

E-mail: silvanaazcarate@gmail.com

The chemical composition is regarded as the most important indicator to evaluate food quality and its nutritional value. Elemental composition (major, minor, trace and rare earth elements) reflects the particular environment and can provide uniquely representative fingerprints, making significant progress in authenticity studies. However, when using spectrochemical instrumental methods for inorganic trace analysis in solid samples, it is necessary to obtain a representative solution with the analytes. Sample preparation is a challenging step in the analytical procedure, where wet digestion sample preparation methods are widely used in analytical chemistry. In this context, Green Analytical Chemistry (GAC) searches for cheaper, more efficient and accurate greener alternatives, developing simple and inexpensive methods for analytes qualitative and/or quantitative determination. There is still a constant search for new alternatives for time-efficient pretreatments, reducing operating steps, contamination and reagent concentrations. In order to comply with GAC recommendations, the aim of this work was to develop and optimize analytical strategies for complex samples preparation extracting with dilute reagents and assisted by ultrasound (USAE) and infrared (IRAE) radiations for multielemental determination by Microwave induced plasma atomic emission spectrometry (MIP OES). Likewise, a comparative analysis was carried out between the two procedures under their optimal conditions and on three different samples (animal feed, grapes and pork meat). The experimental optimization of each sample preparation system was carried out through a face centered central composite design (FC-CCD), assessing 4 and 5 factors for IRAE and USAE, respectively. Dissolved organic carbon (DOC), residual acidity (RA) and solid residue (SR) were evaluated as responses employing desirability function. Thus, response surface methodology was implemented to find the best combination of mass, diluted reagents (HNO3 and H2O2), time and temperature in order to minimize the studied responses for elemental extraction in analyzed samples. The selected factors were evaluated in a range of 100-500 mg (sample mass), 15-45 min (heating time), 2-7 M (HNO3 concentration) and 10-30 % (H2O2 concentration) for both designs and the temperature between 30-60°C was considered in the USAE design while for the IRAE it was kept constant at 190°C. The comparison between both methodologies revealed a better performance of IRAE than USAE for the parameters studied, achieving lower values for DOC, RA and SR for the three samples under study. On the other hand, different optimal conditions were acquired for the different samples, evidencing that the sample preparation also depends on the sample under study. For this reason, after their experimental validation, the optimal combinations of IRAE design for each sample were selected to elucidate sample composition through multielemental analysis; results were compared with guidelines values.

Keywords: Agri-food, Green Pretreatment Design of Experiment, Multivariate Optimization, Multielemental Analysis.