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Evaluation of vortex-assisted matrix solid-phase dispersion for the extraction of Cu, Fe, and Mn from corn samples with determination by HR-CS GF AAS

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Palavras Chave: *Corn*, *VA-MSPD, Green Analytical Chemistry, HR CS GF AAS.*

Highlights

MSPD and HR-CS GF AAS for the determination of Cu, Fe, and Mn in corn
Simple, low-cost method that meets the principles of Green Analytical Chemistry
Use of a renewable-source solid support

Resumo/Abstract

Brazil ranks third in global corn production, which is used for both animal and human consumption, making the monitoring of essential and toxic elements extremely important. Matrix solid-phase dispersion (MSPD) has been applied to the extraction of different types of analytes, both organic and inorganic, and offers advantages such as requiring small sample amounts, using diluted acids, eliminating the need for a heating step, and allowing the use of solid support from renewable source. Therefore, in this study, MSPD was proposed for the first time for the extraction of Cu, Fe, and Mn from corn samples, followed by determination using high-resolution continuum source graphite furnace atomic absorption spectrometry (HR-CS GF AAS). Initially, 0.5 g of ground and dried corn sample and 0.5 g of solid support (alumina, beach sand, florisil, diatomaceous earth, and silica) were weighed. The mixture was macerated in glass mortar for 5 min, and the resulting homogeneous mixture was transferred to polypropylene (PP) tubes. Then, 10 mL of 1 mol L^{-1} HNO_3 was added, followed by vortex agitation for 3 min and centrifugation for 10 min at 4000 rpm. Subsequently, the supernatant was collected, and the quantification of Cu, Fe, and Mn was performed by HR-CS GF AAS. The solid support that presented the best agreement for all analytes was diatomaceous earth, which was therefore selected for the subsequent steps, showing agreement levels of 35%, 46%, and 98% for Cu, Fe, and Mn, respectively, compared to the reference method. As a comparative method, microwave-assisted digestion (MAD) was carried out using a microwave digestion system (Ethos Easy, Milestone), in which 0.3 g of sample was weighed and 5 mL of concentrated HNO_3 was added. The samples were then subjected to a heating program: *i*) a ramp to 200 °C over 20 min; *ii*) a hold at 200 ºC for 15 min); and *iii*) a cooling step of 20 min. After digestion, the solutions were transferred to PP tubes and diluted to 50 mL with ultrapure water. Quantification was then performed by inductively coupled plasma optical emission spectrometry (ICP OES). A Certified Reference Material (CRM) of rice flour (1568b) was used to evaluate the accuracy of the decomposition procedure, showing agreement between 80 and 110% for Cu, Fe, and Mn. Moreover, as it is a renewable-source material, its use aligns with the principles of Green Analytical Chemistry. Thus, the proposed method proved to be simple and environmentally friendly for the extraction and determination of metals in corn samples. As next steps, other MSPD parameters will be evaluated, such as maceration time, type and concentration of the extracting solution, sample-to-sorbent mass ratio, and type and duration of agitation.

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