

Área: ANA

## Matrix Solid-Phase Dispersion combined with HR-CS FAAS for determination of Cu and Mn in oilseed flour samples

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### Highlights

- Green MSPD/HR-CS FAAS method for rapid Cu and Mn determination in oilseeds
- Low sample mass and energy consumption, high safety, and low waste generation
- Application of the AGREE metric

### Abstract

For the first time in the literature, a green method employing matrix solid-phase dispersion (MSPD) combined with high-resolution continuum source flame atomic absorption spectrometry (HR-CS FAAS) was developed for the sequential determination of Cu and Mn in oilseed flour samples. The main MSPD parameters (type of solid support, extraction solution, and stirring type and time) were evaluated and optimized. The optimized conditions were 0.25 g of sample, 0.25 g of diatomaceous earth (DE) as the solid support, 5 mL of 0.5 mol L<sup>-1</sup> HNO<sub>3</sub> as extracting solution, 1 min of maceration, 1 min of manual stirring, and 10 min of centrifugation at 4000 rpm. The optimization was performed using Brazil nut flour (A). Then, the method was applied to nine different oilseed flour samples, including almond (B), peanut (C), hazelnut (D), cashew (E), macadamia (F), nutmeg (G), pecan (H), pistachio (I), and soybean (J) flours. The Cu concentrations obtained using the proposed method (MSPD/HR-CS FAAS) ranged from 2.3 to 10.3 µg g<sup>-1</sup> (RSDs below 10%), while the Mn concentrations ranged from 4.9 to 20.0 µg g<sup>-1</sup> (RSDs below 10%) for the samples A, B, C, D, E, F, G, H, I, J, respectively. The accuracy of the proposed method was evaluated by comparison with an acid digestion/HR-CS FAAS procedure, and the agreement values ranged from 80 to 119% for Cu and from 80 to 105% for Mn. The limits of detection (LOD) of MSPD/HR-CS FAAS were 0.36 µg g<sup>-1</sup> for Cu and 0.19 µg g<sup>-1</sup> for Mn, while the limits of quantification (LOQ) were 1.22 for Cu and 0.64 µg g<sup>-1</sup> for Mn, respectively. Compared to other methods reported in the literature, such as microwave-assisted digestion (MAD) and open-vessel wet decomposition, MSPD showed several advantages, including greater operator safety, simplicity, low cost, reduced energy consumption, and lower waste generation, aligning with the principles of Green Analytical Chemistry (GAC). In applying the AGREE metric, the proposed method achieved a score of 0.61, whereas the reference method scored 0.44. Considering that a higher score indicates a greener method, the proposed method proved to be more environmentally friendly. The MSPD/HR-CS FAAS approach exhibited greener performance in principles 7, 9, 10, 11 and 12 of GAC, corresponding to waste generation, energy consumption, use of bio-based reagents, amount of toxic reagents, and operator safety, respectively. This can be explained as follows: i) in the proposed method, the final volume is 5 mL, while in the comparative method it is 30 mL (principle 7); ii) in MSPD/HR-CS FAAS, the only relevant energy source is the HR-CS FAAS itself, whereas in acid digestion/HR-CS FAAS, there is also a block digester operating for more than 150 min (principle 9); iii) MSPD/HR-CS FAAS employs DE derived from renewable sources (principle 10); iv) in acid digestion/HR-CS FAAS, concentrated HNO<sub>3</sub> and H<sub>2</sub>O<sub>2</sub> are used, increasing method toxicity and reducing operator safety, while in the proposed method, only 0.5 mol L<sup>-1</sup> HNO<sub>3</sub> is employed (principles 11 and 12).

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