

Development and optimization of a new extraction of pesticides from soil using direct-immersion SPME LC-Tips followed by GC-MS/MS

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Pesticide residues are an important contaminant of agricultural soils, which must be adequately monitored. However, their analysis is challenging, especially because of physical and chemical variability among different soils, but also because pesticide residues are often present in trace amounts.

This work presents a new method for the extraction of ten pesticides from a sandy-loam soil, by direct-immersion solid-phase microextraction (SPME), using a new type of semi-disposable SPME configuration in which the fiber is attached to a micropipette tip. The analytes were: boscalid, diflufenican, epoxiconazole, indoxacarb, metalaxyl, metolachlor, metribuzin, penconazole, tebuconazole and terbutylazine.

For method optimization, a Plackett Burman experimental design was used to assess 10 extraction variables, each at two levels. Afterwards, the extraction time and extraction solvent were optimized separately. The final method consisted of making a soil slurry by adding an aqueous solution (6% methanol v/v) to 2 grams of soil. The fibers were conditioned and pre-wetted to solvate the coated phase and then inserted, for extraction, into the samples with constant shaking. Afterwards, the analytes were desorbed onto 100 μ L of methanol for 30 minutes. After the addition of analyte protectants (ethylglycerol, gulonolactone, and sorbitol; 500 μ g/mL) the extract was analyzed by GC-MS/MS in multiple reaction monitoring mode.

Calibration was performed by extracting spiked soil with analyte concentrations of 0.1-50 μ g/kg. Isotopically labeled penconazole was used as internal standard. Coefficients of determination were between 0.94-0.97 for all analytes. Limits of quantification ranged between 0.1-10 μ g/kg. This method has the potential to be automated and generates almost no residual toxic waste. Thus, it has the potential to be introduced as a greener and simpler alternative to currently used methodologies.