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Microextraction techniques at the analytical laboratory: an efficient way for determining low amounts of residual insecticides in soils

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Microextraction techniques allow sensitive measurements of pollutants to be carried out by means of instrumentation commonly available at the analytical laboratory. This communication reports our studies focused to the determination of pyrethroid insecticides in polluted soils. These chemicals are synthetic analogues of pyrethrum widely used for pest control in agricultural and household applications. Because of their properties, pyrethroids tend to strongly absorb to soil particles and organic matter. Although they are considered as pesticides with a low toxicity for humans, long times exposure to them may cause damage in immune system and in the neurological system.

The procedure here studied is based on dispersive liquid-liquid microextraction (DLLME), and permits the determination of fifteen pyrethroid compounds (allethrin, resmethrin, tetramethrin, bifenthrin, fenpropathrin, cyhalothrin, acrinathrin, permethrin, λ-cyfluthrin, cypermethrin, flucythrinate, fenvalerate, esfenvalerate, τ-fluvalinate, and deltamethrin) in soil samples using gas chromatography with mass spectrometry (GC-MS). The analytes were first extracted from the soil samples (4 g) by treatment with 2 mL of acetonitrile, 2 mL of water and 0.5 g of NaCl. The enriched organic phase (approximately 0.8 mL) was separated by centrifugation, and this solution used as the dispersant in a DLLME process. The analytes did not need to be derivatized before their injection into the chromatographic system, due to their volatility and thermal stability. The identification of the different pyrethroids was carried out based on their retention times and mass spectra, considering the m/z values of the different fragments and their relative abundances. The detection limits were in the 0.2-23 ng g-1 range, depending on the analyte and the sample under analysis.

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