Title Optimisation of a solid-phase microextraction/HPLC/Diode Array method for multiple pesticide screening in lettuce

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Abstract

A new method was developed for the determination of 10 pesticides widely used in lettuce production (acetamiprid, azoxystrobin, cyprodinil, fenhexamid, fludioxonil, folpet, iprodione, metalaxyl, pirimicarb, and tolyfluanid) using solid-phase microextraction (SPME) and liquid chromatography (HPLC) with diode-array detection (DAD). The extraction performance of four different SPME coatings, polydimethylsiloxane (PDMS), PDMS/divinylbenzene (PDMS/DVB), carbowax/templated resin (CW/TPR), and polyacrylate (PA) was evaluated using an interface SPME–HPLC. CW/TPR fibre was selected as the most appropriate for the extraction of majority of these pesticides. Three variables (pH, NaCl% and extraction time) were considered key factors in the optimisation process. Interactions between these analytical factors and their optimal levels were investigated by response surface methodology based on central composite design. The method allowed the determination of azoxystrobin, cyprodinil, fenehexamid, fludioxonil, folpet, iprodione, and tolyfluanid in lettuce at concentrations between 0.8 and 25.6 mg/kg, i.e., bellow the maximum residues levels allowed for those compounds in lettuce. Lettuce samples that suffered pesticide treatments with folpet and fenehexamid were analysed during days to harvest to study the dissipation behaviour of the pesticides used. Concentration of folpet was 92.8; 53.4; 22.9; 17.9; 7.45; 1.85 mg/kg and concentration of fenhexamid was 158.1; 76.2; 31.0; 27.3; 7.24; 0.87 mg/kg, respectively for t_0, t_1, t_3, t_6, t_7, t_9 days, and not detected at t_{14} days for the two pesticides.