

Application of Octadecylsiloxane-Coated Fiber in Solid-Phase Microextraction for Determination of Organophosphorus Pesticide Residues in Vegetables

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ABSTRACT

Analysis of organophosphorus (OPPs) insecticide residues in vegetables by using solvent extraction and chromatography is well accepted as an international standard method. However this method requires a lot of organic solvents and leaves much hazardous waste to the environment. Recently, solid-phase microextraction (SPME) and gas chromatography (GC) has been recognized as another suitable method which can solve all the mentioned problems. In this study, SPME was prepared by hydrolysis and polycondensation with octadecylsiloxane (ODS) on fused silica fiber. The efficiency of extraction by ODS SPME was compared with available commercial SPME fibers and the standard GC-FPD analytical method. Results showed that the optimum conditions for analysis of OPPs using SPME/ GC with DB-1 capillary column at 30 m x 0.32 mm I.D., 1 µm film thickness, the optimum temperature program started with 80°C for 30 sec then increased to 300°C after injection at the rate of 30°C/min, the temperatures of GC injector port and PFPD detector were 250°C and 300°C. One gram of blended vegetable sample in 200 ml of ultrapure water had been shaken for 2 hrs and centrifuged for 2 min, then using ODS fiber to immerse in 1 ml of the aqueous extract for 30 min at room temperature. The analytes were desorbed for 4 mins at GC injector port. The optimum conditions were then used for determining the following insecticides: chlorpyrifos, methylparathion, profenofos and prothiofos standard mixtures spiked in vegetable samples. The percentage recoveries were 99.80%, 101.51%, 104.03% and 106.52% with RSD 9.42%, 6.46%, 7.73% and 3.96%, respectively. The detection limits were 5.00, 40.00, 5.00 and 50.00 µg/l, respectively. The precision of the insecticide determination using ODS was not significantly different from using commercial coated fibers. The ODS SPME developed in this study was found to be as effective as polyacrylate(PAC) (1.43-5.56 %RSD).

Analysis of mevinphos, methylparathion, prothiofos and profenofos spiked in tomato samples using ODS SPME /GC-PFPD compared with using AOAC, was not found to be statistically different in percentage of recoveries.