Development of an LC-MS/MS method for the determination of pesticides and patulin in apples

A method for the simultaneous determination of 33 pesticides or degradation products together with patulin in apples by liquid chromatography-tandem mass spectrometry (LC-MS/MS) was developed. The method involved homogenization of the apples, extraction with ammonium acetate-acetic acid solution in methanol-water by ultrasonication, filtration, and determination by LC-MS/MS. The repeatability and within-laboratory reproducibility for the three spiking levels 0.02, 0.04 and 0.2 mg kg\(^{-1}\) were between 4\% and 35\%. In general, the repeatability and reproducibility were about 10-20\%. The limits of quantification (LOQs) were between 0.01 and 0.14 mg kg\(^{-1}\). The method was used on incurred samples from parts of the ISAFRUIT project financed by the European Commission under the 6th Framework Programme. Samples were analysed at four different stages: after harvest, after storage (controlled), after a water bath, and after 28 days at room temperature. Pesticide residues were found at all stages, but no significant differences in the concentration were seen between the stages analysed. The concentration decreased significantly only for tolyfluanid after storage at room temperature for 28 days when only 0-6\% of the original amount of tolyfluanid remained in the apples. No patulin was found in the apples stored for 28 days at room temperature and no growth of Penicillium expansum was observed on these apples. However, when the apples were inoculated with a spore suspension of P. expansum, high concentrations of patulin were found.

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