Development of a Semiautomatic and Miniaturized Solid-Liquid Extraction Method Using Ethyl Acetate for the Determination of Around 80 Gas Chromatography Amenable Pesticides in Vegetable Samples

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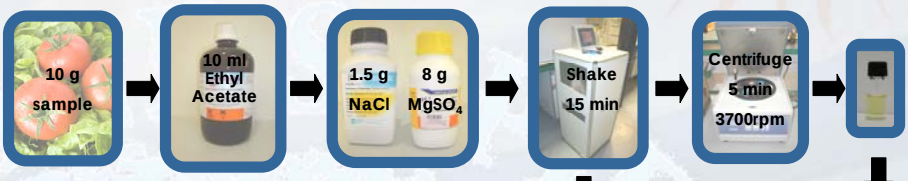
INTRODUCTION

A semiautomatic and miniaturized multirresidue extraction method has been developed using ethyl acetate as extraction solvent. The extraction method is a miniaturized version of the conventional ethyl acetate method, which involves fewer amounts of solvent and also less time consumption in concentration steps. In the proposed method the agitation step is performed by an automatic axial agitator.
Analytical determinations were performed with a GC IT MS system, a MS/MS method has been developed for the determination of the pesticides.
The multirresidue extraction method consist on a solid-liquid extraction with ethyl acetate as solvent, 10 g of sample are extracted with 10 ml of solvent. The agitation step is performed automatically, different parameters like amplitude, rate, acceleration, abruptness, and agitation time were optimized. After 10 minutes of extraction the supernatant was injected directly to the GC IT MS system.
Recoveries were evaluated for all pesticides in tomato matrix at a level of 10 µg/kg yielding recoveries between 70 and 120 %, relative standard deviation calculated with five replicate are lower than 20 % in all cases, except for Methamidophos in which the recovery was lower than 70%.



EXTRACTION: 10 SAMPLES IN 15 MINUTES !!!

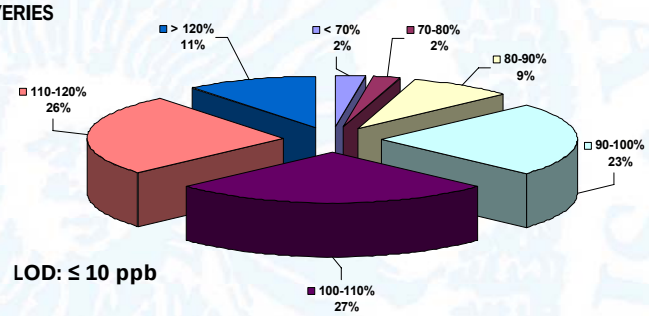
GC-IT-MS/MS

A Varian GC-MS/MS system comprising a CP-3800 gas chromatograph with a 1079 PTV injector, a CP-8400 autosampler and a 4000 ion trap MS was used for the determination of isofenphos methyl.

Optimized conditions

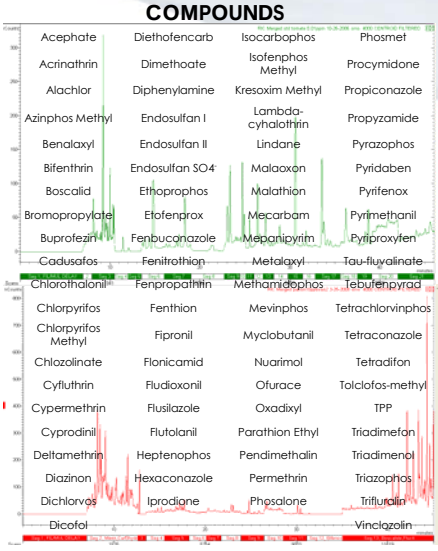
Injection volume: 10µl
Injector temperature programme:
70ºC → 0.50 min;
300ºC at 100ºC/min → 10 min
Column: HP5-MSI 30m x 0.25mm (0.25µm)
Oven temperature programme:
70ºC → 3.50 min
180ºC at 30ºC/min → 10 min
300ºC at 8ºC/min → 5 min
Gas carrier: Helium
Flow: 1 ml/min

RECOVERIES



LOD: ≤ 10 ppb

COMPOUNDS



CONCLUSIONS

The analytical method with the ethyl acetate extraction developed showed a good performance for the determination of the pesticides. The validation parameters, such as recoveries, repeatability, reproducibility and linearity were evaluated for all the compounds in tomato matrix. More than 90 % of pesticides showed a limit of determination lower than 10 µg/Kg, and all of them presented a linear response in the studied range of concentration (10 – 500 µg/Kg).

The whole method was applied for the analysis of 100 samples of vegetables. More than 80% of them contained pesticides at different concentration levels.