Pesticide Residues Determination in Olive and Oilseed Rape Samples by Gas Chromatography-SIM-Mass Spectrometry after Modified QuEChERS Method Coupled to Dual-d-SPE Clean-up

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The challenge of this study was to develop a new procedure for the determination of organochlorine, organophosphate and carbamate pesticides in fatty vegetable matrices such as olives (*Olea europaea cv.* Picual) at different ripeness grade, and therefore different level of fat content, grown in Granada (Spain) and two oilseed rape (*Brassica napus*) varieties (*cv.* Digger and Nelson F1) obtained from Experimental Station Department of Crop Production in Prusy, University of Agriculture in Krakow (Poland). The pesticides extraction was carried out by *Quick, Easy, Cheap, Effective, Rugged and Safe* QuEChERS method, adapted for the analysis of pesticide residues in food matrices with high fat content paying special attention the *clean-up* stage. Pesticides extraction effectiveness was evaluated at two different spiking levels (0,015 mg kg^{-1} and 0,030 mg kg^{-1}) and efficiency of the *dual*-d-SPE *clean-up* stage was evaluated by comparison testing two different d-SPE *clean-up* combinations (SAX/GCB and SAX/C_{18}) at the second *clean-up* step after the addition the combination of three d-SPE sorbents (PSA+GCB+C_{18}) at the first *clean-up* step. The analysis of pesticide residues was performed by Gas Chromatography Ion Trap Mass Spectrometry (GC/IT-MS) working in SIM mode (using Varian 4000 GC-MS (Varian, Inc., USA), consisted of 3800 GC and 4000 Ion Trap MS detector). The linear relation was observed from 0 to 400 ng mL^{-1} and the determination coefficient R^2>0,997 in all instances for all target analytes. Better recoveries were obtained in samples at 0,030 mg kg^{-1} spiking level. The recoveries were in the range 70-120%, with RSD values lower than 17% at 0,030 mg kg^{-1} spiking level for most pesticides.

Keywords: pesticides, GC-MS, QuEChERS, olive, oilseed rape, fatty vegetable matrix

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