

Simultaneous analysis of 108 pesticide residues belonging to different chemical classes in rice samples collected from Tehran market using LC-MS/MS

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Background and Aims: In this study, a validated multi-residue method was used for simultaneous determination of 108 LC-amenable pesticides in 130 rice samples (65 imported and 65 domestic) collected from different regions of Tehran, in 2009-2010.

Methods: Five g of homogenized sample was weighed into a 50 mL centrifuge tube. Ten mL of acetonitrile (MeCN) were added and shaken. Two g MgSO₄ and 1.5 g sodium acetate were added and vortexed. Five mL of the extract was concentrated to dryness and dissolved in 0.5 mL MeCN. For clean-up, 20 mg PSA and 60 mg MgSO₄ were added to the extract, vortexed and centrifuged. Finally, 100 µL supernatant was introduced to LC-MS. Chemicals were analyzed simultaneously in a single injection using positive electrospray ionization (ESI+) and with multiple-reaction monitoring (MRM). The amounts of pesticides were determined using interpolation of the relative peak areas for each pesticide to internal standard (carbofuran-d₃) peak area in the sample on the matrix-matched calibration curves.

Results: From the 108 pesticides present in 130 samples, 13 compounds were found in 32 domestic and/or imported samples in concentration ranged 21.4 - 48.6 ng/g. The detected pesticides in domestic samples include bioallethrin (17%), cinosulfuron (10%), cypermethrin (1.5%), deltamethrin (0.8%), imazalil (6.9%), foramsulfuron (1.05%), phosphamidon (10%), spynosinA (7.7%), spynosin D (8.4%), TCMTB (8.4%), triadimenol (6.9%), tricyclazole (8.5%) and in imported samples include cinosulfuron (10.8%), imazalil (4.6%), flutriafol (3.0%), phosphamidon (9.2%), spynosinA (11.5%), spynosin D (10%), TCMTB (10%), triadimenol (10.8%), triasulfuron (5.4%) and tricyclazole (11.5%).

Conclusions: Five samples were contaminated with cinosulfuron, spynosinA, spynosin D, triadimenol and/or tricyclazole at the levels below Iranian maximum residue limits (MRLs) in rice. For other detected pesticides, no MRLs have been set in rice in Iran. Therefore, 27 out of 130 (20.8%) samples were contaminated with illegal pesticides.

Keywords: LC-MS/MS; Multi-residue analysis; Pesticides; Rice