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Development a liquid chromatography coupled with tandem mass spectrometry (LC-MS/MS) method for simultaneous determination of 108 pesticide residues in rice

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Background and Aims: In this study, a multi-residue method for simultaneous determination of 108 LC-amenable pesticides, belong to different chemical classes has been developed in rice.
Methods: Pesticides residues were extracted from rice samples based on the modified QuEChERS (Quick, Easy, Cheap, Effective, Rugged, and Safe) sample preparation procedure. **Methods:** Five g of homogenized sample was weighed into a 50 mL centrifuge tube. Ten mL of acetonitrile (MeCN) were added and shaken vigorously. Then, 2 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 triple quadrupole analyzer with multiple-reaction monitoring (MRM). The method was validated using rice samples spiked with 108 pesticides at 3 different levels (n=5) and carbofuran- d3 as internal standard. A matrix-matched calibration curves was established for each pesticide.
Results: The calibration curves for all compounds were linear in the concentration range 0.02-1.0 µg/g with a correlation coefficient range between 0.993 and 0.999.The LOQ and LOD were 0.025 µg/g and 0.008 µg/g for all 108 pesticides, respectively. The mean recoveries of pesticides obtained for three fortification levels (0.025, 0.250and 1.0µg/g) were 72-116% with satisfactory precision (RSD <20%).
Conclusions: For the first time in Iran, an accurate, precise, sensitive and selective multi-residue method was developed for the simultaneous detection, quantification and confirmation of 108 pesticide residues (belong to very different chemical families) in rice using QuEChERS sample preparation procedure and LC–MS/MS.

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