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Matrix Effects of different food matrices in quantitative analysis of pesticides in chromatography coupled to tandem mass spectrometry

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Injudicious use of pesticides has lead to increase in the pesticide levels in the environment and food. This increase may pose many health hazards to humans and can affect the animals and various ecosystems adversely. Consumption of pesticidecontaminated food via daily diet is a major source of exposure to pesticides. It is necessary to monitor various pesticide residues in food commodities.Most of the pesticide analysis is done by multi-residue analysis methods as it is less time consuming as compared to single residue methods. Availability of more sensitive chromatographic techniques and tandem mass spectrometry coupled to chromatography (GC, GC-MS/MS, LC-MS/MS) has improved the selectivity and sensitivity of pesticide analysis and their identification and quantification in agricultural products. The quantitation by chromatography and/or chromatography coupled to mass spectrometry is affected by matrix in the multi-residue analysis leading to suppressed or enhanced results due to matrix effects. The matrix effect of a compound is the change in signal in a solvent solution compared with signal in matrix due to presence of co-extracted compounds, or changes in eluent properties, such as surface tension, viscosity, volatility – all factors known to affect the ionization process. The suggested ways to reduce the matrix effect are (i) sample clean-up (ii) introduction of additives into the mobile (iii) internal standards or isotopically labeledexternal standards prepared in sample matrix, (iv) dilution of matrix to reduce matrix effects. But it is difficult to obtain a blank matrix for every sample type and will immensely decrease the number of analysis per day. In the present study, we investigated matrix interferences by QuEChERS sample preparation to establish whether (i) dilution of matrix can eliminate the need of matrix standards in LC-MS/MS (ii) the commodities can be grouped together, thus reducing the number of matrices needed to make the calibration standards. Matrix effects were studied by comparing of slopes of calibration curves of matrix (diluted with mobile phase) and solvent based standards. Present study showed that matrix effects were dependent on both, nature of commodity as well as analyte. Maximum matrix effect variability was observed in capsicum. Most of the pesticides showed ion suppression in tomato, capsicum and cumin matrices. In brinjal matrix most of the pesticides showed slight ion enhancement, though the extent of ion enhancement was very less.Due to similar nature of matrix effect of tomato and capsicum these two commodities can be grouped together. Cumin matrix was most difficult to analyse. As 4X dilution did not completely eliminated matrix effects a comparison was made between 10X diluted matrices of cucumber and brinjal. Recovery study was also undertaken in brinjal was done and compared with both the matrices. Matrix effect did not vary much between the two matrices. Analyte variability was also less. Most of the pesticides showed recoveries in acceptable range of 70-130% with calibration curvesfrom both matrices. To compensate for matrix effect it is suggested that (i) tomato and capsicum matrix which show similar trend can be grouped together, (ii) 10 times diluted cucumber matrix can be used to prepare calibration curves for quantitation of pesticides on LC-MS/MS from different fruiting and cucurbits vegetable matrices with LOQ of 0.05 mg kg-1. Further studies can be done with other vegetables like chillies, onion, garlic, spinach, fruits, cereals, pulses and spices to find candidate matrix for quantitation. To understand the effect of mode of ionization on matrix effect, the matrix effects in LC-MS/MS and GC-MS/MS were compared.

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