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Determination of Pyrethroid Pesticides by UHPLC-MS/MS. Can it be a real alternative to GC-MS/MS?

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ABSTRACT

The thesis focuses on optimization of UHPLC-MS/MS method and assessment of important analytical parameters such as MEs, Linearity and LODs to assure the reliability of UHPLC-MS/MS method in detecting pyrethroid pesticides in sample extracts of lemon, cherry and lettuce and critically comparing with similar techniques as well as GC-MS/MS.

Matrix effect was assessed by constructing external calibration curves and matrix-matched calibration curves and comparing their slope ratios. The LODs of each analyte were determined using a S/N of 3 measured from the chromatograms of quantitative MRM transitions in both External and matrix-matched calibration curves and linearity was assessed from the obtained correlation coefficient ((R^2).

The result revealed variability among both compounds and matrices. ME ranged from (-33% to -94%)with all the analytes expressing ion suppression. Lettuce showed the strongest suppression (-44% to -94%) followed by cherry (-35 to -85) and lemon (-33% to -71%). The pattern reflects the increasing matrix complexity and co-interferences from pigments, chlorophyll, organic acids and sugars that compete during the ionization process. λ-cyhalothrin in all matrices exhibited the least ion suppression while cypermethrin exhibited the highest suggesting differential ionization behavior based on compound structure.

Higher LODs up to 100 ng/ml were observed across all the matrices in the matrix-matched calibration, indicating the impact of matrix effects, whereas the LODs for the external curve ranged from 1 to 10 ng/ml. All the calibration curves, both external and matrix-matched demonstrated excellent linearity with R2 ranging from 0.9901 to 0.9994 meeting the acceptance criteria set by SANTE/12682/2019 which is ($R2 \geq 0.98$).

The results obtained when compared to other GC-MS/MS methods affirm that the UHPLC-MS/MS method is an alternative to GC-MS/MS for quantification of pesticide residues in food samples especially fruits and vegetables.

To improve analytical reliability and lower matrix interferences in complex food samples, further improvement employing matrix-matched calibration and optimization of clean up sorbents is recommended.