

**METHOD DEVELOPMENT ON THE ANALYSIS OF
PESTICIDES IN VEGETABLES USING LIQUID
CHROMATOGRAPHY - MASS SPECTROMETRY (LC - MS)**

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ABSTRACT

A multiclass and multiresidue analytical method using liquid chromatography with atmospheric pressure ionisation mass spectrometry (LC-API-MS) and a heated nebuliser with positive and negative chemical ionisation (PCI/NCI) modes of operation for the trace determination of pesticides in vegetables was developed. Six types of organophosphorus, three types of N-methylcarbamates and one each of a chlorinated and phenylurea pesticide were selected in this study. Different classes of pesticides are extracted together with 5% ethyl alcohol in ethyl acetate (v/v) and cleaned up using a Florisil column elute with 100 ml acetonitrile in a single procedure. No fractional collection is required. The final extract was then concentrated and injected into the LC-MS system without pre- or post-column derivatization. Both pseudo-molecular ion of the positive and negative type- $(M+H)^+$ and $(M-H)^-$ or the characteristic fragment ion of the pesticides were monitored under PCI/NCI using time-scheduled selected ion monitoring (SIM) conditions with an acquisition window of each pesticide for identification and quantification. The heated nebuliser probe temperature, focusing ring and orifice voltages were optimised to achieve identification capabilities with the highest sensitivity. With some modification, the flow injection analysis (FIA) technique has been applicable for pesticide formulations. The FIA system can perform quick single residue analysis (SRM) without any sample pre-treatment process or LC column. This measurement method can be extremely useful for determining pesticide formulations. Detection limits for both techniques were in the region of 1 - 3 μg and recoveries ranged from 70% to 110 %.

ABSTRAK

Kaedah kromatografi cecair menggunakan spektrometri jisim - pengionan tekanan atmosfera (LC-API-MS) dengan penebula panas bagi menganalisis berbagai kelas/jenis sisa pestisid dalam sayur-sayuran telah di lapurkan. Enam jenis organofosforus, tiga jenis N-metilkarbamat, satu pestisid berklorin dan satu lagi pestisid fenilurea telah di pilih dalam kajian ini. Berbagai kelas/jenis sisa pestisid telah di ekstrak dengan pelarut 5% etanol dalam etil asetat dan pembersihan dalam turus Florisil dalam satu prosedur yang sama. Kaedah pemisahan ini tidak memerlukan pungutan pecahan. 1 μ L pengekstrak kemudian di suntik ke dalam sistem LC-API-MS tanpa sebarang pra / pos terbitan.

Kedua-dua nya iaitu pseudo-molekul atau ion serpihan tertentu daripada pestisid di rakamkan dengan pengionan kimia positif atau negatif (PCI/NCI) menggunakan mod perakaman ion tunggal (SIM) serta satu tingkap perolehan data bagi setiap jenis pestisid demi mengenalpasti dan mengkuantitasi pestisid yang terkandung dalam sayur-sayuran. Suhu penebula panas, voltan bulatan tumpuan serta pembukaan diselaraskan supaya mencapai kepekaan yang tertinggi bagi peralatan ini. Dengan sedikit perubahan, kaedah Analisis Suntikan Aliran (FIA) boleh digunakan untuk menentu formulasi pestisid tanpa sebarang proses pra-perlakuan sample. Kaedah pengukuran formulasi pestisid ini merupakan satu cara yang amat pantas dan berkesan. Had pengesanan dan purata pengembalian semula bagi pestisid-pestisid dalam kajian masing-masing adalah 1 – 3 μ g dan 70% hingga 110%.

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