Development of an Analytical Method for Determination of Highly Polar Pesticide Residues In some Food Products

Thesis Submitted

By

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In the Partial Fulfillment for the Requirement of the Master Degree in Chemistry

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Approval sheet

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Abstract

Name: Mostafa Soliman Abd-el-Ghaffar Abd-el-Megid Abd-ellah

Title of the thesis: Development of an Analytical Method for Determination of Highly Polar Pesticide Residues In some Food Products.

Position: Chemist

Degree: M.Sc., Faculty of science, Ain Shams University

A new simple, easy, fast and cheap modified QuEChERS procedure for the determination of diquat in potatoes using reversed phase liquid chromatography coupled with tandem mass spectrometry (LC-MS/MS) in a total run time of 10 min was developed. Different sample preparation parameters (pH modifier type, sample size effect, and elevated temperature effect) have been tested and optimized. Potatoes sample was extracted with acetonitrile in presence of ammonium hydroxide at 80 °C. Phase separation was obtained by shaking the extract with magnesium sulfate and sodium chloride and analysis was done using liquid chromatography–tandem mass spectrometry. Matrix-matched standard calculations were applied to compensate for matrix induced suppression in LC-MS/MS determination. The precision and trueness of the method were determined from recovery experiments on

five replicates of spiked blank potatoes samples at 0.01, 0.05 and 0.1 mg/kg. The obtained recoveries ranged from 74 to 110% and their RSD values was <5% for all the concentrations.

Key words: QuEChERS, Diquat, Potato, Reversed phase liquid chromatography, LC-MS/MS.

Supervisors' approval:

- **Prof. Dr. Mohamed Mahmoud Mohamed Abo-Aly**
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Aim of work

The present study aimed to introduce a sensitive and simple method for the determination of diquat residues in potato that can be used for the routine determination of diquat residues in potato, taking in consideration that the limit of quantification of the developed method must be less than the maximum residue limit of diquat in Egypt.

So, the plan of work was to:

1- Optimization of analytical method for determination of diquat in potato using Liquid Chromatography with Tandem Mass spectrometry (LC-MS/MS).

2- Validation of the analytical method for determination of diquat in potato.

List of abbreviations

CAC: Codex Alimentarius Commission.

CE: Collision Energy.

Diquat: 1,1'-ethylene-2,2'-bipyridylium dication.

DP: Declustring Potential.

ESI: Electrospray Ionization.

EU: European Union.

EURL-SRM: European Union Reference Laboratory for Pesticides Requiring Single Residue Methods.

FAO: Food and Agriculture Organization.

GAP: Good Agriculture Practice.

HILIC: Hydrophilic Interaction Liquid Chromatography.

K: Partition coefficient.

$K_{o/c}$: Desorption constant.

$K_{o/w}$: octanol/water partition coefficient.

LC-MS/MS: Liquid chromatography tandem mass spectrometry.

LD50: Median lethal dose.

LiSC: Liquid Separation Cell.

LOQ: Quantification limit.

MRM: Multiple Reaction Monitoring.

PP: polypropylene.

Psi: pound-force per square inch.

Q1: Frist Quadrapole

Q2: Second Quadrapole

QuEChERS: Quick, Easy, Cheap, Effective, Rugged and Safe method.

QuPPe: Quick Polar Pesticides method.

RSD%: Relative standard deviation.

SPE: Solid Phase Extraction.

US-EPA: United States Environmental Protection Agency.

WHO: World Health Organization.

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