CONTENTS

	page
LIST OF ABBREVIATIONS	
INTRODUCTION	1
REVIEW OF LITERATURE	4
1. Detection of pesticides in water	3
2. Detection of pesticides in soft drinks	40
MATERIALS AND METHODS	51
1. Method (LP)	65
2. Method (SPE)	67
RESULTS AND DISCUSSION	69
1. Optimization of LC-MS/MS parameters	71
a. Optimization of MS/MS	72
2. Optimization of GCMS/MS	85
3. Parameteres of Validation	86
Method 1. Liquid Partioning	88
Method 2. SPE on LCMSMS	96
Limit of Quantitation (LOQ)	139
Measurement Uncertainty	143
SUMMARY	153
REFERENCES	159
ARABIC SUMMARY	

LIST OF ABBREVIATIONS

Abbreviations

Synonyms

μg	Micro-gram, 10 ⁻⁶ g.
APCI	Atmospheric pressure chemical ionization.
API	Atmospheric pressure ionization.
CE	Collision energy.
DP	Declustring potential.
ECD	Electron capture detector.
EI	Electron impact.
ESI	Electo-spray-ionization.
EU	European union.
eV	Electron volt.
FAO	Food and Agriculture Organization.
FINAS	Finnish Accreditation Service
GC	Gas chromatography.
GC-MS/MS	Gas chromatography tandem mass spectrometry.
GPC	Gel permeation chromatography.
HPLC	High performance liquid chromatograph.
IPs	Identification points.
kg	Kilo-gram, 10 ³ g.
K _{OW}	Distribution coefficient between n-octanol and water.
LC-MS/MS	Liquid chromatography tandem mass spectrometry.
LOD	Limit of detection.
LP	Liquid portioning
log _P	The logarithm of the concentrations ratio of the
	unionized solute in the solvents.
LOQ	Limit of quantification.
m/z	Mass-to-charge ratio.
mg	Milli-gram, 10 ⁻³ g.
ml	Milli-liter, 10 ⁻³ liter.
mM	Milli-molar.
ng	Nano-gram.
MRL	Maximum residue limit.
MRM	Multiple reactions monitoring.
MSD	Mass detector.
MSPD	Matrices solid phase dispersion.
NPD	Nitrogen phosphorus detector.
PI	Positive ionization.

PSI	Pound per square inch.	
RSD	Relative standard deviation.	
SPE	Solid phase extraction.	
SPME	Solid-phase micro-extraction.	
WHO	World Health Organization.	

Name of Candidate: Mohamed Abdel-Aziz Mohamed Abdel-Aziz Degree: PhD.
Title of Thesis: Determination Of Some Pesticide Residues In Water And Soft
Drinks
Supervisors: Dr. Mohamed Helmy Belal
Dr. Essam El-Din Abd El-Raofe Eweis
Dr. Sanaa Mohamed Abd EL-Kader El-Sawi
Department: Economic Entomology and Pesticides
Branch: - Pesticides Approval: 12 / 12 /2017

ABSTRACT

An applicable gas & liquid chromatographic (GC- MSMS & LC-MSMS) method was performed. It was found to be specified to determine the residues of certain organochlorine, organonitrogen, carbamate, and organophosphorous pesticides using SPE technique in water and soft drinks (colored and non colored).

The method performance was tested on 402 pesticides representing different types and groups of pesticides. The average recovery of these pesticides using a concentration range of 0.05-1.0 μ g/L (ppb) for water and 0.1-1.0 μ g/L (ppb) for soft drink varied between 70 to 120%. The reproducibility expressed as relative standard deviation (CV) was less than 16.0%. The method showed to be linear at least up to 1.0 μ g/L.

The limit of quantitation (LOQ) in drinking water samples was $0.05\mu g/L$ but in soft drinks (colored and non colored) samples was 0.1 $\mu g/L$. The measurement uncertainty expressed as expanded uncertainty and terms of relative standard deviation (at 95% confidence level) was found to be within the range of \pm 32%.

According to the good results of method in validation and performance test (PT) the method was accredited by the FINAS assessor's team in 2016.

Key words: Pesticide residues, validation, water, soft drinks