

بسم الله الرحمن الرحيم



-Caron-





شبكة المعلومات الجامعية التوثيق الالكتروني والميكروفيلم





جامعة عين شمس

التوثيق الإلكتروني والميكروفيلم

قسم

نقسم بالله العظيم أن المادة التي تم توثيقها وتسجيلها على هذه الأقراص المدمجة قد أعدت دون أية تغيرات



يجب أن

تحفظ هذه الأقراص المدمجة بعيدا عن الغيار



MONITORING OF COUNTERFEIT SITUATION OF THE MOST DOMINANT PESTICIDES IN EGYPT.

By

GEHAD MOSAD KHATTAB HASAN

B. Sc. Agric. Sc. (pesticides), Ain Shams University, 2006M. Sc. Agric. Sc. (pesticides), Ain Shams University, 2012

A thesis submitted in partial fulfillment

of

the requirements for the degree of DOCTOR OF PHILOSOPHY

in

Agricultural Science (Pesticides)

Department of Plant Protection
Faculty of Agriculture
Ain shams University

MONITORING OF COUNTERFEIT SITUATION OF THE MOST DOMINANT PESTICIDES IN EGYPT.

By

GEHAD MOSAD KHATTAB HASAN

B. Sc. Agric. Sc. (pesticides), Ain Shams University, 2006

M. Sc. Agric. Sc. (pesticides), Ain Shams University, 2012

Under the supervision of:

Dr. Mohamed Ibraheam Abd El-Megeed

Prof. Emeritus of Pesticides Chemistry, Department. of Plant Protection, Faculty of Agriculture, Ain Shams University (Principal Supervisor)

Dr. Walaa Mohamed Abd El-Ghany.

Prof of Pesticides and Vice Dean for Graduate & Research Affairs, Faculty of Agriculture, Ain Shams University

Dr. Ibrahim El-Desoky Mohamed Mohamed.

Head of Research Emeritus of Pesticide Analysis Res., Dep. Cent. Agric., Pesticides Lab., Agric., Res. Center.

ABSTRACT

Gehad Mosad Khattab: Monitoring of Counterfeit Situation of The most Dominant Pesticides in Egypt. Unpublished Ph.D. Thesis, Department of plant protection, Faculty of Agriculture, Ain Shams University, 2021.

This study aims to assess Counterfeit Situation of Pesticides in Egypt through monitoring of the counterfeit of a pesticide widely used in Egypt.

23 samples of different pesticide formulations were collected, which include 9 active ingredients to monitor the Situation of pesticide adulteration in the Egyptian market.

There are 20 unregistered samples through Egyptian Agricultural Pesticides Committee thus representing 86.96% of the total tested samples, four samples of them have registration number and the same name as formulations already registered in Ministry of Agriculture representing 20% of the total unregistered samples, These formulations are (Tinam EC 1.8%), (Mospilan SP 20%), (Score EC 25%) and (Dimethoate EC40 EC 40% code27) the registration numbers of them are (1391), (959), (945) and (1478) respectively. Three samples are Sardo 25% SC), (Chlorofos EC 48%) and (Ictafos EC 48%) were expired representing 13.04% of the total tested samples.

When determination the percentage of the active ingredients in the samples under study, the results were as follows, Only two samples with representing 8.69% of the samples under study were within the permissible limits of % a.i, and these samples are (Tinam EC 1.8%) and (Chlorofos EC 48%) active ingredient content was 1.53 and 50.355% respectively.

Five samples are (Farmactine EC 1.8%), (Occidor WP 50%), (diazinon EC 60%), (dozion EC 60%) and (Dimethoate EC40 EC 40% code28) didn't contain any active ingredient under the condition

analysis. These samples representing 21.74% of the total samples under study.

The percentage of the active ingredient in 16 samples was less than the acceptable limits representing 69.57% of the total tested samples.

The physical properties of the studied samples before and after storage were determined, 17 samples formulated (EC) determined through emulsion characteristics test, six of samples showed good emulsification characteristics before and after storage representing 35.29% of samples formulated (EC) and eleven samples showed poor emulsification characteristics before and after storage representing 64.7% of samples formulated (EC).

Four samples formulated (SP) determined through wet ability characteristics test all samples under testing showed good wet ability characteristics before and after storage representing 17.39% from the total tested samples.

Two samples one of them formulated (WP) and the other formulated (SC), both of them showed good Suspensibility and wet ability characteristics before and after storage representing 8.69% from the total tested samples.

Key words:

Counterfeit Pesticides, Illegal Pesticides, Mass Spectroscopy, IR, Physical properties.

ACKNOWLEDGMENT

Many thanks and Praise be to great Allah most gracious who shined my way and supported me with patience and perseverance to fulfill this humble work.

I wish to express my deep gratitude to Prof. **Dr. Mohamed Ibraheam Abd EL-Megeed** Prof. of Pesticides Chemistry, Dept. of Plant Protection, Faculty of Agriculture, Ain Shams University, for his Supervision, guidance, useful criticism, valuable help and his efforts to fulfill this work. For his interest and efforts on my behalf, I am extremely grateful.

My Sincere appreciation and deep gratitude and thanks to **Dr. Walaa Mohamed Abd El-Ghany**, Professor of Pesticides Chemistry, Dept. of Plant Protection, Faculty of Agriculture, Ain Shams University, for his sharing in supervision, kind unfailing help throughout this work, advice and encouragement in carrying out this work.

No words can adequately express my deep appreciation and gratitude to **Dr. Ibrahim El-Desoky Mohamed,** Head of Research Emeritus of Pesticide Analysis Res., Dep. Cent. Agric., Pesticides Lab., Agric., Res. Center., for his supervision constructive criticism, valuable advice and help, continuous encouragement, cooperation, and kind assistance.

I would like to express my deepest thanks and appreciation to my best friend **Dr. Zakia Kamal** Researcher in Pesticides Formulations Dept., Central Agricultural Pesticides Laboratory, Agricultural Research Center, for her great help and valuable suggestions throughout this work.

A lot of thanks for all my friends specially Dr. Zakia Kamal Dr. Mona Bakry, Dr. Rania Mohamed, Dr. Ezat rafat, Dr. Ismail Ibrahim and Dr. Rasha Mohamed., and to all my friends for helping

me, in Central Agric. Pesticides Lab., Agric. Research center, Ministry of Agric.

Finally my sincere thanks that I feel Indebted to My brother (Ahmed Mosad), who contributed and supported me in any way to fulfill this work.

to my **father** and my **mother**, God rest them soul, and that have a great credit to my success, my mother taught me that first grabbed the pen and my father taught me how can study my lessons and learn .

Thanks for them prayer

God put them in Paradise and more merciful for my parents.

CONTENTS

NO.	Title		
	LIST OF TABLES		
	LIST OF FIGURES		
	LIST OF ABBREVIATIONS		
I.	INTRODUCTION	1	
II.	REVIEW OF LITERATURE	8	
A	The position of counterfeit pesticides around the world.	8	
В	The position of counterfeit pesticides in Egypt	16	
C.	Threats and risks related to the use of counterfeit		
	pesticides	20	
Ш.	MATERIAL AND METHODS	34	
1.	Pesticides used	34	
1.1.	Abamectin formulations	36	
1.2.	Acetamipirid formulations	40	
1.3.	Azoxystrobin formulations	42	
1.4.	Carbendazim formulations	44	
1.5.	Chlorpyrifos formulations	45	
1.6.	Cypermethrin formulations	47	
1.7.	Diazinon formulations	49	
1.8.	Difenoconazole formulation	51	
1.9.	Dimethoate formulations	52	
2.	Quantitative analysis of active ingredients and		
	impurities of the investigated pesticide formulations	54	
2.1.	Standard preparations of the tested pesticides (a.i) and		
	impurities	54	
2.2.	Sample preparations of the tested pesticide		
	formulations	54	
2.3.	Sample preparations of the impurities of the tested		
	pesticide formulations	55	
3.	Determination of active ingredients and impurities		

	contents of the tested pesticides
3.1.	Gas Chromatography
3.2.	High Performance Liquid Chromatography
4.	Determination of other materials present in the
	formulation
4.1.	Gas Chromatography- Mass Spectroscopy
4.2.	Infrared spectroscopy
4.3.	Liquied Chromatography- Mass Spectroscopy
5.	Determination of the main physical criteria
	corresponding to each of investigated formulation
5.1.	Store samples in the oven
5.2	Preparation of standard water
5.3.	Emulsion stability evaluation for formulations (EC)
5.4.	Suspensibility test of (SC and WDG) formulations
6	Questionnaire forms for all workers in the pesticide
	system
6.1.	Farmers
6.2.	Traders
6.3.	Researchers
IV.	RESULTS AND DISCUSSION
1.	Abamectin formulations
1.1.	Abamectin formulation labels
1.2.	Physical Properties of Abamectin formulations
1.3.	Determination of active ingredients content of
	Abamectin formulations by HPLC
1.4.	Identification of other materials present in the samples
1.4.1	LC-Ms analysis of Abamectin formulations
1.4.2	GC-Ms analysis of Abamectin formulations
1.4.3.	Identification of Abamectin by FTIR
2.	Acetamipirid formulations
2.1.	Acetamipirid formulation labels
2.2	Physical Properties of Acetamipirid formulations
2.3	Determination of active ingredients content of

	Acetamipirid formulations
2.4.	Determination of other materials present in the samples
2.4.1.	GC-MS Analysis of Acetamiprid formulations
2.4.2	Identification of Acetamiprid by FTIR
3	Azoxystrobin formulation
3.1	Azoxystrobin formulation label
3.2	Physical Properties of Azoxystrobin formulation
3.3	Determination of active ingredients content of
	Azoxystrobin formulations
3.4.	Determination of other materials present in the samples
3.4.1.	GC-MS Analysis of Azoxystrobin formulation
3.4.2	Identification of Azoxystrobin by FTIR
4	Carbendazim formulation
4.1.	Carbendazim formulation label
4.2.	Physical Properties of Carbendazim formulation
4.3.	Determination of active ingredient content and level of
	impurities in Carbendazim sample
4.3.1.	Determination of active ingredient content of
	Carbendazim formulation
4.3.2.	Level of impurities
4.4.	Determination of other materials present in the samples
4.4.1	GC-MS Analysis of Carbendazim formulation
4.4.2.	Identification of Carbendazim by FTIR
5	Chlorpyrifos
5.1	Chlorpyrifos formulation labels
5.2	Physical Properties of chlorpyrifos formulations
5.3	Determination of active ingredient content and level of
	impurities in Chlorpyrifos samples
5.3.1.	Determination of active ingredient content of
	chlorpyrifos formulations
5.3.2.	Level of impurities
5.4.	Determination of other materials present in the
	samples

5.4.1	GC-MS Analysis of chlorpyrifos formulations	111
5.4.2	Identification of chlorpyrifos samples by FTIR	113
6	Cypermethrin	115
6.1	Cypermethrin formulation labels	115
6.2	Physical Properties of Cypermethrin formulations	116
6.3	Determination of active ingredient content in	
	Cypermethrin samples	116
6.4.	Determination of other materials present in the samples	118
6.4.1.	GC-MS Analysis of cypermethrin formulations.	118
6.4.2.	Identification of Cypermethrin samples by FTIR	120
7.	Diazinon	122
7.1.	Diazinon formulation labels	122
7.2.	Physical Properties of Diazinon formulations	123
7.3.	Determination of active ingredient content and level of	
	impurities in Diazinon samples	123
7.3.1.	Determination of active ingredient content of diazinon	
	formulations	123
7.3.2.	Level of impurities	124
7.4	Determination of other materials present in the	
	samples	125
7.4.1.	GC-MS Analysis of diazinon formulations	125
7.4.2.	Identification of Diazinon samples by FTIR	126
8	Difenoconazole formulation	128
8.1	Difenoconazole formulation label	128
8.2	Physical Properties of Difenoconazole formulation	128
8.3	Determination of active ingredients content of	129
	Difenoconazole formulations	129
8.4	Determination of other materials present in the samples	129
8.4.1	GC-MS Analysis of Difenoconazole formulation	129
8.4.2	Identification of Difenoconazole by FTIR	130
9	Dimethoate	132
9.1	Dimethoate formulation labels	132
9.2	Physical Properties of Dimethoate formulations	133