

**SOLID LIQUID EXTRACTION FOR THE EVALUATION OF  
ORGANOPHOSPHORUS CONTENT IN APPLE AFTER WASHING  
TREATMENT**

**MUHAMMAD NAZIRUL IKRAM BIN ABD JALIL**

**Final Year Project Report Submitted In  
Partial Fulfillment Of the Requirements for the  
Degree of Bachelor of Science (Hons.) Chemistry  
in the Faculty of Applied Sciences  
University Teknologi MARA**

**JANUARY 2015**

## **ACKNOWLEDGEMENT**

IN THE NAME OF ALLAH THE MOST GRACIOUS THE MOST MERCIFUL

First and foremost, thanks to God Almighty for the guidance and the help in giving me the strength to complete this study. I also would like to thank my supervisor Madam Siti Raihan Binti Zakaria because of her guidance, advice and support in order to me complete this thesis study.

I also would like to thank all the laboratory assistants for giving full cooperation and guidance in handling the instruments and providing all my needs through the study.

Finally, I want to express my sincere appreciation to my beloved parents, family and friends for their support and encouragement in completing this thesis study

Muhammad Nazirul Ikram Bin Abd Jalil

## TABLE OF CONTENTS

	<b>Page</b>
<b>ACKNOWLEDGEMENT</b>	iii
<b>TABLE OF CONTENTS</b>	iv
<b>LIST OF TABLES</b>	vii
<b>LIST OF FIGURES</b>	viii
<b>LIST OF ABBREVIATIONS</b>	ix
<b>ABSTRACT</b>	xi
<b>ABSTRAK</b>	xii
<b>CHAPTER 1 INTRODUCTION</b>	
1.1 Research background	1
1.2 Problem statement	3
1.3 Research objectives	4
1.4 Significance of study	4
1.5 Scope of study	5
<b>CHAPTER 2 LITERATURE REVIEW</b>	
2.1 Chlopyrifos	6
2.1.1 Pathways	6
2.1.2 Metabolites	8
2.1.3 Exposure	11
2.1.4 Toxicity	13
2.1.5 Maximum residue limits	14
2.2 Apple	15
2.3 Removal of chlorpyrifos	16
2.4 Method development	
2.4.1 Solid liquid extraction	17

2.4.2	High Performance Liquid Chromatography	18
2.4.3	UV-Visible detector	19
2.4.4	Suitability of mobile phase	20
2.4.5	Wavelength of chlorpyrifos	20

### **CHAPTER 3 METHODOLOGY**

3.1	Materials and equipment	
3.1.1	Reagents and chemicals	21
3.1.2	Instruments	21
3.2	Experimental work	
3.2.1	Condition of HPLC	22
3.2.2	Qualitative analysis	22
3.2.3	Quantitative analysis	22
3.2.4	Setup for HPLC	23
3.2.5	HPLC analysis of chlorpyrifos	23
3.3	Analysis method	
3.3.1	Preparation of cleaning solution	24
3.3.2	Method of pesticides pollution in apple	25
3.3.3	Sample extraction	26
3.3.4	Preparation of standards chlorpyrifos solution	28
3.3.5	Preparation of mobile phase	28
3.4	Limitations of study	29

### **CHAPTER 4 RESULT AND DISCUSSION**

4.1	Introduction	30
4.2	Solvent system for HPLC analysis	31
4.3	Qualitative analysis of chlorpyrifos	34
4.4	Calibration curve of chlorpyrifos standards	36
4.5	Quantitative analysis of chlorpyrifos in apple samples	39

## ABSTRACT

### SOLID PHASE EXTRACTION FOR THE EVALUATION OF ORGANOPHOSPHORUS CONTENT IN APPLE AFTER WASHING TREATMENT

Apple is a very nutritious fruit but the used of pesticide had cause a concern to human health. Besides, apple had been considered as one of the top list of dirty frozen which is contaminated with pesticides. In this study, the amount of chlorpyrifos in apple after washing treatment and the effectiveness of the washing solution had been determined. The analysis was carried out using Reversed Phase High Performance Liquid Chromatography. The stationary phase used was C<sub>18</sub> column and the mobile phase used is a binary system of acetonitrile-water (70:30). The system was detected using UV-Visible detector at the wavelength of 230 nm and flow rate of 1 mLmin<sup>-1</sup>. The analysis was carried out using simple analytical procedure which is solid liquid extraction (SLE) and the method was successful. The peak of chlorpyrifos appeared at retention time of 2.2 minutes. There were six samples that had been used including the blank. The linearity obtained was 0.9857. The concentration of chlorpyrifos in sample 1, 2, 3, 4, 5 and blank were 12.50 ppm, 8.73 ppm, 7.56 ppm, 5.21 ppm, 6.73 ppm and 4.34 ppm respectively. The removal percentages of chlorpyrifos in apples were ranging from 30% to 59%. The most effective washing solution was acetic acid with 58.32% removal and the least effective washing solution was tap water with 30.16% removal. The washing solution efficiencies depend on the concentration. The concentration of washing solution will enhance the solubility of chlorpyrifos in washing solution and lead to greater removal of chlorpyrifos.