UNIVERSITI PUTRA MALAYSIA

DEVELOPMENT OF METHOD FOR DETERMINING GLYPHOSATE RESIDUES IN PALM OIL MATRICES AND SOIL SAMPLES USING HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

MOHAMAD FARIQ FITRI BIN MOHD SALEH

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By

MOHAMAD FARIQ FITRI BIN MOHD SALEH

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfillment of the Requirement for the Degree of Master of Science

November 2015

DEDICATION

Specially dedicated to:
My beloved family, friends, and especially my lovely wife.

Abstract of thesis presented to the Senate of University Putra Malayisa in fulfilment of the requirement for the degree of Master of Science

DEVELOPMENT OF METHOD FOR DETERMINING GLYPHOSATE RESIDUES IN PALM OIL MATRICES AND SOIL SAMPLES USING HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

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Chair: Professor Dzolkhifli Omar, PhD.
Faculty: Agriculture

Herbicides such as glyphosate is commonly used in palm oil plantation to control broad-leaved weeds and grass which can result in loss of yield. The use of pesticides in oil palm plantations has resulted in the growing concern of the presence and danger of the residues in the palm oil as well as in the environment. Glyphosate is one of the common herbicides use to control weeds in the plantation and of concern of its residue in palm oil. Thus, a method was developed in order to monitor its leaching and persistence in oil palm agroenvironment. Although a lot of researches on pesticide residue determination in oil palm matrices had been carried out, the analytical methods for determining the residue of glyphosate in palm oil matrices via pre-column derivatization with FMOC-Cl have limited reports. The use of pre-column derivatization provides numerous advantages in terms of the use of non-complicated instruments, rapid, fewer restrictions, and efficient. The detection and quantification of glyphosate was comprehensively studied by using High Performance Liquid Chromatography with Fluorescence Detector (HPLC-FLD). The instrumentation parameters such as detector wavelength, flow rate of mobile phase, selection of mobile phase was optimized. In the analysis to determine residues of glyphosate in palm oil samples, extraction step based on liquid-liquid extraction (LLE) technique with pre-column derivatization was optimized. Dichloromethane and water were used as the extraction solvent and centrifuged for 30 minute at 3000 rpm. The extraction of glyphosate from soil samples was also investigated and agitation with magnetic stirrer was chosen as the optimized extraction technique. Extraction parameters such as extraction solvent (deionized water, sodium hydroxide and potassium dehydrogen phosphate), solvent volume (15, 25 and 30 mL), and duration of extraction (30,45 and 60 min) were comprehensively investigated and optimized. The optimized conditions

were obtained using 25 mL of potassium dehydrogen phosphate as the extraction solvent and 30 min as the duration of extraction. Recovery of glyphosate obtained from spiked crude palm oil (CPO), crude palm kernel oil (CPKO), soil samples were 85-97, 87-92 and 83-88%, respectively with relative standard deviations of less than 3%. The limit of detection (LOD) and limit of quantification, (LOQ) for glyphosate in spiked CPO, CPKO and soil samples was 0.01 and 0.05 µg/mL, respectively. Validation studies were also carried out and the method developed for analysis of glyphosate in spiked CPO, CPKO and soil samples gave the best reproducibility with low % RSD for all tested matrices.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia Sebagai memenuhi keperluan untuk ijazah Sarjana Sains

PEMBANGUNAN KAEDAH BAGI MENGESAN GLIFOSAT DI DALAM MATRIKS MINYAK SAWIT DAN SAMPEL TANAH DENGAN MENGGUNAKAN KROMATOGRAFI CECAIR BERPRESTASI TINGGI

Oleh

MOHAMAD FARIQ FITRI BIN MOHD SALEH

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Racun rumpai seperti glifosat biasanya digunakan dalam ladang kelapa sawit untuk mengawal rumpai berdaun lebar dan rumput yang boleh menyebabkan kehilangan hasil daripada pohon sawit. Penggunaan racun perosak di ladang kelapa sawit telah menyebabkan kebimbangan yang semakin meningkat dengan penggunaan dan kehadiran sisa-sisa minyak sawit yang bahaya di samping pencemaran terhadap alam sekitar. Glifosat adalah salah satu daripada racun rumpai yang biasa digunakan untuk mengawal rumpai di ladang dan dikhuatiri sisanya terdapat di dalam minyak sawit. Oleh itu,sebuah kaedah telah dibangunkan bertujuan untuk memantau kadar larut lesap dan ketahanan glifosat di dalam persekitaran kelapa sawit. Meskipun banyak kajian telah dijalankan berkaitan penentuan residu racun perosak di dalam matriks minyak sawit, namun penentuan residu glifosat di dalam matriks minyak sawit melalui kaedah derivatisasi pra-turus dengan FMOC-Cl masih tidak pernah dijalankan. Penggunaan teknik derivatisasi pra-turus memberikan banyak kelebihan seperti penggunaan instrumen yang mudah, kaedah yang cepat, kurang masalah dan berkesan. Kaedah pengesanan dan kuantifikasi glifosat dikaji secara komprehensif dengan menggunakan kromatografi cecair berprestasi tinggi dengan pengesan pendarfluor (HPLC - FLD). Parameter intrumen seperti panjang gelombang, kadar aliran fasa bergerak , pemilihan fasa bergerak telah dioptimumkan. Dalam analisis untuk menentukan sisa glifosat dalam sampel minyak sawit , langkah pengekstrakan berdasarkan pengekstrakan cecair -cecair (LLE) dengan pra-derivitasi turus telah dioptimumkan. Dalam analisis untuk menentukan sisa glifosat dalam sampel minyak sawit, langkah pengekstrakan berdasarkan pengekstrakan teknik LLE dengan pra-derivitasi telah dioptimumkan. Diklorometan dan air telah digunakan sebagai pelarut pengekstrakan dan diemparkan selama 30 minit pada kadar 3000 rpm. Pengekstrakan glifosat dari sampel tanah juga dikaji dan teknik pergolakan dengan menggunakan pengacau magnet telah dipilih sebagai teknik pengekstrakan. Parameter pengekstrakan seperti pengekstrakan pelarut (air ternyah ion , natrium hidroksida dan kalium dehydrogen fosfat), jumlah pelarut (15 , 25 dan 30 mL) , dan tempoh pengekstrakan (30,45 dan 60 min) telah diuji secara menyeluruh dan dioptimumkan. Syarat-syarat yang dioptimumkan telah diperolehi dengan menggunakan 25 mL kalium dehydrogen fosfat sebagai pengekstrakan pelarut dan 30 minit sebagai tempoh pengekstrakan. Kajian perolehan semula glifosat

diperolehi daripada minyak sawit mentah (MSM), minyak isirong sawit (MIS), sampel tanah yang telah disuntik dengan larutan piawai glifosat, adalah pada kadar 85-97 , 87-92 dan 83-88 % , masing-masing dengan sisihan piawai relatif kurang daripada 3%. Had pengesanan (LOD) dan had kuantifikasi , (LOQ) untuk glifosat dalam MSM, MIS dan sampel tanah, masing-masing adalah 0.01 dan 0.05 µg / mL. Kajian pengesahan turut dijalankan pada kaedah yang dibangunkan untuk kesemua analisis glifosat dalam MSM, CPKO dan sampel tanah, dan keputusan daripada kajian memberikan kebolehulangan yang baik dengan % RSD rendah untuk semua matriks yang telah diuji.

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I certify that a Thesis Examination Committee has met on the date of viva to conduct the final examination of Norhayu binti Asib on her thesis entitled Genetic Diversity of Tuba plants, and Structural Elucidation, Formulation and Toxicities of their in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U (A) 106] 15 March 1998. The committee recommends that the student be awarded the Doctor of Philosophy.
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LIST OF ABBREVIATIONS

%	Percent
°C	celcius
µg/mL	microgram per mililiter
µl	microliter
ASE	Accelerated solvent extraction
CPKO	Crude Palm Kernel Oil
CPO	Crude Palm Oil
ECD	Electron Capture Detector
FAO	Food and Agriculture Organization
GC	Gas chromatography
GCB	Graphitized carbon black
GPC	Gel Permeation chromatography
HS-SPME	Headspace solid-phase microextraction
HPLC	High Performance Liquid Chromatography
hrs	Hours
IUPAC	International Union of Pure and Applied Chemistry
LOD	Limit of Detection
LOQ	Limit of Quantification
Min	minute
ml	mililiter
MS	Mass spectra
nm	Nanometer
O/W	oil in water
PCA	Principle component Analysis
PCR	Polymerase Chain Reaction
ppm	Part per million
RAPDs	Random Amplified Polymorphic DNA
RNA	Ribonucleic Acid
rpm	revolution per minute
SE	Standard error
Sec	Second
SFE	Supercritical fluid extraction
SPE	Solid Phase Extraction
SPME	Solid Phase Microextraction
spp	species
USE	Ultrasonic extraction
w/w	weight/weight

CHAPTER 1

INTRODUCTION

1.1 General Introduction

Generally, there are two types of agricultural crops in Malaysia, namely food crops and industrial crops. Food crops include vegetables, fruits, and rice; while industrial crops comprise of rubber, cocoa, and oil palm (Fatimah 1997). Oil palm is one of the main commodities of the country's economic driver for the Economic Transformation Programme (ETP) under the responsibility of the Malaysia Ministry of Plantation Industries and Commodities (KPPK) (Che Johari, M. 2011). Palm oil has been widely used in the production of food and non-food products, such as soap and biodiesel. In addition, palm oil has also been listed among one of the 17 vegetable oils received at the meeting of the FAO/ WHO food standard requirements under the CODEX Alimentarius Commission Program (CODEX Alimentarius Commission 1983).

Besides, the palm oil industry has been projected to grow due to high demand all over the world. In 2013, the total area of oil palm cultivation reached 5.64 million hectares, showing an increase of 4.6% compared to 5.39 million hectares reported in the previous years (May Y.C., 2015). Hence, Malaysia, being a major palm oil producer besides Indonesia, has to necessarily ensure that the productivity of palm oil to continue to increase, as well as the production of high quality of palm oil. Moreover, protection efforts concerning the growth of palm trees should be taken in order to avoid any disruption that can affect oil palm production, such as insects, diseases, and the presence of weed in the oil palm agro system (Izwarie, 2011).

The use of pesticide is an alternative method to solve the disruption problem in planted area. Besides, the FAO (2002) had reported that the use of pesticides has increased over the past 35 years with an increase of 4-4.5% every year. Moreover, most smallholders and farmers use pesticides to control, as well as to kill weeds, fungi, and pests, due to high efficiency, besides being low in cost (Faizah & Ayat, 2008). In fact, various types of pesticides are often used in oil palm plantation in Malaysia, such as fungicide, rodenticide, insecticide, and herbicide (Ainie *et al.*, 2007; Halimah, 2000). The frequent types of herbicides applied in oil palm plantations are glyphosate, glufosinate ammonium, dicamba, 2,4-D, and triclopyr, which are used for killing weeds, such as *Mikania mikrantha, Eleusine indica*, and *pennisitum* (Ainie *et al.* 2007; Chung *et al.* 2000).

Apart from that, the increase of consumption and pesticide application in oil palm industry to increase the productivity has posed a risk of pollution to the environment ecosystem, and thus, has raised concerns pertaining to the quality level and the safety of oil palm produced. This was proved with the discovery of lindane and chlorpyrifos residues in a river at Selangor and Tasik Chini in Pahang (Leong *et al.* 2007; Sarojeni, 2008), as well as the findings of glufosinate ammonium (*Basta R*) residues on the soil surface up to a depth of 15 cm (Krebs *et al.* 1987). The seeping of pesticides into water or soil can occur directly either by spraying or mixing the pesticides with soil or indirectly through washing by rain, leaching or run-off (Maznah, 2013). In addition, Seiber (2002) reported that reduction and decomposition of pesticides residue in the environment depend on the rate of composites, such as leaching, hydrolysis, volatilization, and decomposition, by microbes. Furthermore, mobility rate, chemical content, adsorption rate, intake by tree roots, and the wind also can affect the condition of herbicide (Carolina *et al.* 2008).

Following herbicide contamination of soil and water in the oil palm ecosystem, herbicide residues retained in the soil also pose a risk in contaminating palm leaves and oil palm fruits. Usually, oil palm fruits are processed into crude palm oil, palm kernel oil, and other oil as the raw material in the food manufacturing industry. Hence, it is indeed harmful if there are traces of herbicide residual in the oil palm fruitlets since the herbicide residues in oil can negatively affect human with nervous disorder, immunology, carcinogenesis, and shortness of breath (Ren-Xiang *et al.* 2008). Therefore, a proper monitoring methodology must be developed against palm oil produced to ensure that the oil is free from any herbicide residual contamination.

Several methods of analysis of organo-phsphorus pesticides had been developed in various matrices such as fatty food, water, soil, vegetable and human serum. Most of the method are complex, time consuming and uses a lot of chemicals (Tsunoda., 1993; Sancho *et al.*, 1994; Vreeken *et al.*, 1998). Progress in the field of research is growing rapidly, thus, development of the new methods and modification of previous analytical methods is easier with the use of updated instruments and methods. This is because; the previous analytical methods employed old instrumentation, high in cost, and required long periods of analysis.

Although a lot of researches on pesticide residue determination in various matrices had been carried out, the analytical methods for determining the residue of glyphosate via pre-column derivatization with FMOC-Cl have limited reports. The use of pre-column derivatization provides numerous advantages in terms of the use of non-complicated instruments, rapid, fewer restrictions, and efficient.. Thus, the discovery of new methods or the improvement of the existing analytical methods is important to determine glyphosate residues in various environmental matrices.

1.2 Objectives Of The Study

i. To develop an efficient method of extraction, as well as detection of glyphosate residues in crude palm oil (CPO), crude palm kernel oil (CPKO), and soil matrices.

ii. To validate the methods of extraction and detection of glyphosate residues in CPO, CPKO, and soil matrices using field samples.

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