DETERMINATION OF PESTICIDES IN WATER SAMPLES USING DISPERSIVE LIQUID - LIQUID MICROEXTRACTION (DLLME) AND GAS CHROMATOGRAPHY- MICRO ELECTRON CAPTURE DETECTOR (GC-µECD)

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RESEARCH REPORT SUBMITTED IN FULFILLMNT OF THE REQUIREMENT FOR THE DEGREE OF MASTER OF SCIENCE (ANALYTICAL CHEMISTRY AND INSTRUMENTAL ANALYSIS)

DEPARTMENT OF CHEMISTRY FACULTY OF SCIENCE UNIVERSITY OF MALAYA KUALA LUMPUR

2012

UNIVERSITY MALAYA ORIGINAL LITERARY WORK DECLARATION

Name of Candidates: **HUSNA BINTI A. HAMID** (I.C/Passport No.: 830115-03-5522) Registration/Matric No.: SCG100030 Name of Degree: MASTER OF SCIENCE (ANALYTICAL CHEMISTRY AND INSTRUMENTAL ANALYSIS Title of Project Paper/ Research Report/ Dissertation/ Thesis ("this Work"): DETERMINATION OF PESTICIDES IN WATER SAMPLES USING DISPERSIVE LIQUID - LIQUID MICROEXTRACTION (DLLME) AND GAS CHROMATOGRAPHY – MICRO ELECTRON CAPTURE DETECTOR (GC-µECD) Field of Study: ANALYTICAL CHEMISTRY I do solemnly and sincerely declare that: (1) I am the sole author/writer of this work; (2) This work is original; (3) Any use of any work in which copyright exists was done by way of fair dealing and for permitted purpose and any excerpt of extract from, or reference to or reproduction of any copyright work has been disclosed expressly and sufficiently and the title of the work and its authorship have been acknowledged in this work; I do not have any actual knowledge nor do I ought reasonably to know that the making (4) of this work constitutes an infringement of any copyright work; I hereby assign all and every rights in the copyright to this work to the University of (5) Malaya ("UM"), who henceforth shall be owner of the copyright in this work and that any reproduction or use in any form or by any means whatsoever is prohibited without the written consent of UM having been first had and obtained; I am fully aware that if in this course of making this work i have infringed any (6) copyright whether intentionally or otherwise, I may be subject to legal action or any other action as may be determined by UM. Candidate's Signature Date Subscribed and solemnly declared before,

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ABSTRACT

The surface modification of magnetic nanoparticles was carried out in order to change the polarities. The coated particles with octyl group on the surface to ensure the attachment of the functional group and hence improved the extraction process in two step of Dispersive Liquid Liquid Microextraction, DLLME method.

Captured Detector, GC-ECD were used for the extraction and determination of pesticides extracted from water samples (treated waste water, tap water from laboratory and housing area). In this extraction method, CCl₄ was selected as the extraction solvent since it shows the highest relative recovery, 41.20 to 98.30 % and 1-octanol was selected as dispersal solvent with the relative recovery of 29.02 to 81.39 %.

Under the optimum condition, extraction of pesticides residues in treated waste water, tap water (laboratory and housing area) were investigated. Chlorothalonil in treated waste waters shows the highest content (0.6789 µgL⁻¹) compared tap water in laboratory and hosing area, which lies in the values of 0.4781 and 0.4781 µgL⁻¹ respectively. The presence of DDT residues in three different types of water were detected at the level of 0.1762 to 0.7173 µgL⁻¹. The residue of DDE detected in treated waste water was 0.2122 µgL⁻¹ compared to tap water in housing area (0.1762 µgL⁻¹). However, there are no residues of DDE was detected in tap water collected in laboratory. Chloropyriphos shows the higher percentage recovery compared to the other pesticides residues in the ranges of 43.21 to 86.79 %.

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LIST OF ABBREVIATION

DDT Dichlorodiphenyltrichloroethane

DDE Dichlorodiphenyldichloroethylene

LD₅₀ Lethal Dose

WHO World Health Organization

ADI Acceptable daily intake

DNA Double Nucleic Acid

PAHs Polyaromatic Hydrocarbons

PCBs Polychlorinated biphenyls

ND Not Detectable

SPE solid-phase extraction

MIT Molecular Imprinting Technique

SPME Solid-Phase Micro-extraction

SDME Single-Drop Micro-extraction

HF-LPME Hollow Fibre-based Liquid-Phase Micro-extraction

GC Gas Chromatography

HPLC High Performance Liquid Chromatography

DLLME Dispersive Liquid-Liquid Micro-extraction

LLE liquid-liquid Extraction

K Distribution coefficient

AAS Atomic Absorption Spectroscopy

GC-FID Gas Chromatography - Flame Ionization Detector

GC-MS Gas Chromatography - Mass Spectroscopy

GC-ECD Gas Chromatography - electron Capture Detector

mmol Milimol

OCP Organochlorine Pesticides

ng nanogram

 C_2Cl_4 Tetrachloro ethylene

CH₃Cl Chloroform

 C_6H_5Cl Chlorobenzene

ACN Acetonitrile

μg microgram

L Litre

MeOH Methanol

EtOH Ethanol