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DEVELOPMENT OF PALM METHYL ESTER MICROEMULSIONS AS AEROSOL INSECTICIDES

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By

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DEVELOPMENT OF PALM METHYL ESTER MICROEMULSIONS AS AEROSOL INSECTICIDES

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Palm methyl ester microemulsions are used as a basis for preparing aerosol insecticides against domestic insect pests due to their favourable characteristic such as renewable resources, biodegradable, non-flammable, and less or non-toxic to end users. The study focuses on the development of microemulsion-insecticides using palm-based materials as the oil phases, emulsifiers and/co-emulsifier, and apply them as a basis for preparing aerosol insecticides for household uses. The work includes utilization of palm-based materials containing mixed Tween 80 and Dehydol LS2 (Tween 80:DLS2) at ratios of 70:30, palm methyl esters (PME), deionized water (DH_2O), and 1-propanol as a co-surfactant (Tween 80:DLS2/PME/DH_2O/1-propanol) for preparing water in oil (W/O) and oil in water (O/W) microemulsion (µE) solutions. It involves screening process, ternary phase diagram study, physico-chemical characterizations of microemulsions, formulating and preparing microemulsion-insecticides as aerosol-insecticides, and bioefficacy test of the formulated products on insect pests. The optimum compositions and concentrations for both W/O-µE and O/W-µE solutions showed very clear appearance and thermodynamically stable at ambient and high temperatures (45ºC) as well as low in viscosity and very small particles with nano size. Ternary phase diagrams systems containing mixed Tween 80:DLS2/PME/DH_2O/1-propanol showed large isotropic regions. The optimum compositions for W/O-µE solution were at the range of 20% mixed Tween 80:DLS2 (70:30); 20%-25% PME; 47.5%-40% DH_2O; 12.5%-15% 1-propanol while for O/W-µE solutions, the optimum compositions were at the range of 7.5%-12.5% mixed Tween 80:DLS2 (70:30); 5%-7.5% PME; 77.5%-70% DH_2O; 10% 1-propanol. Both systems were used to formulate W/O-µE and O/W-µE aerosol insecticides to control flying insects such as *Aedes Aegypti* mosquitoes and crawling insects such as *Periplaneta Americana* cockroaches respectively. The physical stability and the mist behaviour of the palm methyl esters aerosol insecticides were studied. There were very fine mist (30-50 microns) suspended longer in the air (6-8 seconds) observed with no water droplet released and no foam formation. These properties made them very suitable to be applied as flying insect killer (FIK) and crawling insect killer (CIK) aerosol-spray products. The bioefficacy test on both products showed good performance in knocking down insects by 100%, 24 hours of mortality rate as compared to the Malaysian Standard Aerosol. In conclusion, this system consisting mixed non-

ionics surfactants, PME, and non-toxic co-surfactant has great potential to be used in preparing palm methyl esters aerosol insecticides which are more environmental and end-users friendly.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk Ijazah Master Sains

PENGHASILAN MIKROEMULSI METIL ESTER KELAPA SAWIT SEBAGAI RACUN SERANGGA AEROSOL

Oleh

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Mikroemulsi metil ester kelapa sawit digunakan sebagai asas untuk menyediakan racun serangga aerosol bagi mengawal serangga perosak domestik disebabkan ciri-cirinya yang bagus seperti sumber yang boleh diperbaharui, mesra alam, tidak mudah terbakar, dan kurang atau tidak berbahaya kepada pengguna. Kajian ini menekankan penghasilan mikroemulsi-racun serangga menggunakan bahan berasaskan kelapa sawit sebagai minyak, pengemulsi dan/pengemulsi bersama dan digunakan sebagai asas untuk menghasilkan racun serangga aerosol untuk digunakan di rumah. Kajian ini termasuklah penggunaan bahan berasaskan kelapa sawit yang mengandungi campuran Tween 80 dan Dehydol LS2 pada nisbah 70:30 (Tween 80:DLS2), metil ester kelapa sawit (PME), air ternyahion (DH_2O), dan 1-propanol sebagai surfaktan bersama (campuran Tween 80:DLS2/PME/DH_2O/1-propanol)untuk menyediakan larutan mikroemulsi (μE) air dalam minyak (W/O) dan larutan mikroemulsi (μE) minyak dalam air (O/W). Ia melibatkan proses saringan, kajian gambarajah tiga fasa, pencirian sifat kimia fizik mikroemulsi, memformulasi dan menghasilkan mikroemulsi-racun serangga sebagai racun serangga aerosol, dan ujian bioefikasi produk yang diformulasi terhadap serangga perosak. Komposisi dan kepekatan optimum untuk kedua-dua larutan W/O-μE dan O/W-μE menunjukkan larutan yang sangat jelas dan stabil secara termodinamik iaitu stabil pada suhu biasa dan suhu yang tinggi (45ºC) dan juga rendah kelikatan serta zarah yang sangat kecil dengan saiz nano. Sistem gambar rajah tiga fasa yang mengandungi campuran Tween 80:DLS2/PME/DH_2O/1-propanol menunjukkan kawasan isotropik yang besar. Komposisi optimum untuk larutan W/O mikroemulsi (μE) berada pada julat 20% campuran Tween 80:DLS2 (70:30); 20%-25% PME; 47.5%-40% DH_2O; 12.5%-15% 1-propanol manakala bagi larutan O/W mikroemulsi (μE), komposisi optimum berada pada julat 7.5%-12.5% campuran Tween 80:DLS2 (70:30); 5%-7.5% PME; 77.5%-70% DH_2O; 10% 1-propanol. Kedua-dua sistem ini telah digunakan untuk memformulasi W/O-μE dan O/W-μE racun serangga aerosol masing-masing untuk mengawal serangga jenis terbang seperti nyamuk *Aedes Aegypti* dan serangga jenis merayap seperti lipas *Periplaneta Americana*. Kestabilan dan sifat fizikal kabus racun serangga aerosol berasaskan metil ester kelapa sawit telah dikaji. Kabus yang sangat kecil (30-50 microns) dilihat stabil di udara (selama 6-8 saat) tanpa sebarang titisan air dibebaskan dan tiada pembentukan buih. Ciri ini menjadikan larutan ini sangat sesuai

untuk digunakan bagi membunuh serangga terbang (FIK) dan membunuh serangga merangkak (CIK) (produk semburan aerosol). Ujian bioefikasi pada kedua-dua produk menunjukkan prestasi yang baik dengan membunuh serangga pada kadar kematian 100% dalam masa 24 jam berbanding dengan Aerosol Standard Malaysia. Kesimpulannya, sistem ini yang terdiri daripada campuran surfaktan bukan ionik, PME, dan surfaktan bersama yang tidak berbahaya mempunyai potensi yang besar untuk digunakan dalam penyediaan racun serangga aerosol berasaskan metil ester kelapa sawit yang lebih mesra pengguna dan alam sekitar.

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The thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirements for the degree of Master of Science. The members of the Supervisory Committee were as follows:

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LIST OF ABBREVIATIONS

PME	Palm methyl esters
W/O	Water-in-Oil
O/W	Oil-in-Water
µE	Microemulsions
DH_2O	Deionized water
FIK	Flying insect killer
CIK	Crawling insect killer
AOT	Aerosol OT (Sodium diethylhexyl sulfosuccinate)
S_1	Primary surfactant
S_2	Secondary surfactant
Co-S	Co-surfactant
HLB	Hydrophilic-lipophilic balance
EO	Ethylene oxide
LC	Liquid crystal
Wt %	Weight percent
w/w	Weight per weight
L_1	Isotropic liquid region
L_c	Liquid crystalline region
M	Multilayer region
EC	Emulsifiable concentrates
EW	Water-based emulsion
MEW	Water-based microemulsion
CPO	Crude palm oil
PKO	Palm kernel oil
PKC	Palm kernel cake

PFAME	Palm fatty acid methyl esters
PK-MCT	Palm kernel-medium chain triglycerides
GNI	Gross national income

CHAPTER 1

INTRODUCTION

1.1 Background of Research

The palm oil industry has brought great economic benefits to Malaysia. The current production of 19 million metric tons (Mmtons) of crude palm oil (CPO) adds 8% to the country's gross national income (GNI) (Zwart, 2013). In addition, Malaysia is one of the main producers and exporters of palm oil in the world, in which about 90% of the oil is used for foods and 10% for oleochemicals or non-food applications (Ismail, 2006). The Malaysian palm oil industry has flourished with a favourable growth in export demand due to the limited supply of world oils and fats. The high demand due to depleting world stock levels results in higher prices of oils and fats as well as palm oil products (Basri *et al*., 2013).

Zwart (2013) reported that oil palm plantations in Malaysia cover close to 5 million hectares (Mha) and with the plantations in Indonesia and other parts of the world, the total oil palm acreage reached more than 16 Mha in 2011. The plantations yield crude palm oil (CPO), palm kernel oil (PKO) and palm kernel cake (PKC) as the products that have been of prime interest. These are traditional ingredients for a wide variety of food, feed and non-food products. In Malaysia, CPO production in 2011 reached 18.91 Mmtons, 2.39 Mmtons for PKC and 2.15 Mmtons for PKO. A consistent and predictable supply of palm kernel oil and palm oil has led to the development of the oleochemical industry in the country (MPOB, 2014).

Most oleochemicals produced in Malaysia are basic oleochemicals such as fatty acids, fatty alcohols, soap noodles, methyl esters, and glycerines. For 2014, exports of oleochemical products rose by 2.3% to 2.22 million tonnes. The statistics for the major oleochemical products exported were fatty acids (0.82 million tonnes or 36.7% of total oleochemical exports), followed by fatty alcohols (0.46 million tonnes or 20.7%), soap noodles (0.41 million tonnes or 18.5%), methyl esters (0.29 million tonnes or 13.0%) and glycerines (0.23 million tonnes or 10.4%). The Malaysian Palm Oil Board (MPOB) through Advanced Oleochemical Technology Division (AOTD) has been actively carrying out research and development activities in downstream products to develop palm-based products (MPOB, 2014).

Oleochemicals are derived from natural oils such as palm and palm kernel oils. These oils and their derivatives have many advantages over petrochemicals (which are derived from petroleum) in that they are renewable, biodegradable, non-flammable and usually less irritant to eyes and skins of end users. Thus, palm oil and its derivatives such as palm fatty acid methyl esters (PFAME), also called as palm oil methyl esters (POME) and palm kernel-medium chain triglycerides (PK-MCT) have potential to be utilised as the oil phases in the study of microemulsion formulations for practical applications (Ismail, 2006). In this work, the development of microemulsion system using palm-

based materials as the oil phases, emulsifiers and/co-emulsifier for aerosol insecticides formulations was studied.

Microemulsions are transparent optically isotropic (clear) and thermodynamically stable solutions, whereby the droplets size is very small (≤100nm) and have low viscosity (Tadros, 2005). These properties have attracted many researchers, formulators, manufacturers, and end-users to study and apply this system for producing high value-added products. Palm methyl esters microemulsions are used as a basis for preparing aerosol insecticides against domestic insect pests due to their favourable characteristic such as renewable resources, biodegradable, non-flammable, and less or non-toxic to end users.

The global specialty or non-agricultural insecticide market is estimated at more than $3.0 billion at the formulated product marketer level in 2003 (Kline & Company, Inc (2004). The leading companies are Bayer, Aventis, Dow, FMC, Syngenta, S.C. Johnson, Scotts, and Sumitomo. While the United States is the largest market segment, accounting for an estimated 60% of the total, Pacific Rim and selected Latin American countries have shown growth when their economies have supported it. These areas continue to offer growth opportunities as per capita incomes rise and human health issues are accorded more importance.

Basic insecticide producers have come to rely on the specialty segment for profitable growth at a time when the two largest crop segments, corn and cotton, have become depressed (or soon will be) due to the introduction of insect resistant seeds. In this environment, specialty non-agricultural insecticide markets are perceived to offer a more stable, profitable outlet for a small but increasing percentage of active ingredient volume. In Malaysia, the sales of household insecticides reflected increasing affluence of the Malaysian society at large as shown in Figure 1.1 (Lee Nai Pin, 2009). With the outbreak of dengue fever and the desire of the citizens to control mosquitoes particularly Aedes, the sales of aerosols and mosquito coils increased particularly in the Klang Valley. Mosquitoes are vectors of many life threatening diseases in humans.

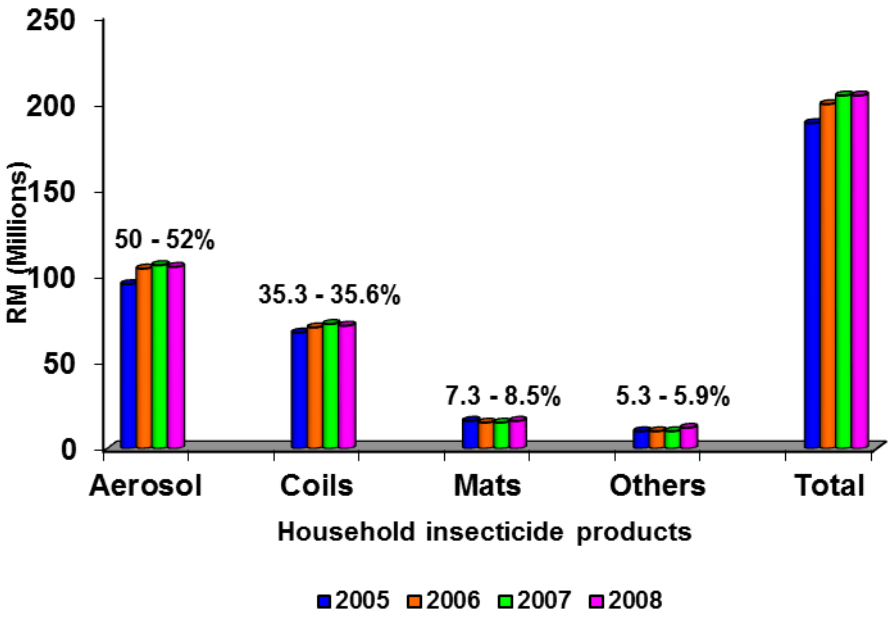

Figure 1.1: The Malaysian Household Insecticides Market (2005 to 2008) in RM Millions (Source: Lee Nai Pin, 2009)

1.2 Statement of Problems

Insecticides comprise about 20% (w/w) of the total pesticides marketed in Malaysia in which more than 55% of the insecticides are in the form of solvent-based formulations or emulsifiable concentrates (EC). The conventional aerosol insecticides consist of 100% solvents derived from petroleum resources, such as xylene, kerosene, toluene and other petroleum-based solvents. (MCPA, 2002). The formulations tend to cause medical problems (e.g., skin and eye irritations) to the end-users or operators, and they are highly flammable and non-biodegradable. In addition, petroleum products are known to be depleting in resources and the prices increased significantly over the years. Therefore, there is increasing demand for safer and more convenient pesticide formulations such as water-based emulsion (EW) or microemulsion (MEW) instead of EC-aerosol insecticide formulations (Ismail, 2007). In recent years, most of Agrochemical companies attempt to formulate products in forms that can be applied globally. Furthermore, there has been dramatic shift from solvent-based to water-based aerosol insecticides due to increasing requirement for safe and more convenient pesticide formulations to ensure easy application and its effectiveness in killing the insect pests.

1.3 Scope of Study

Palm methyl esters microemulsions are used as a basis for preparing aerosol insecticides against domestic insect pests due to their favourable characteristic such as renewable resources, biodegradable, non-flammable, and less or non-toxic to end users. The studies

emphasized the development of microemulsion-insecticides using palm-based materials as the oil phases, emulsifiers, and/or co-emulsifier, and apply them as a basis for preparing aerosol insecticides for household uses. The work include utilization of palm-based materials containing anionic or mixed non-ionic surfactants, palm methyl esters (PME), deionized water (DH_2O), and 1-alkanol as a co-surfactant for preparing water in oil (W/O) and oil in water (O/W) microemulsion (μE) solutions. It involves screening process, ternary phase diagram study, physico-chemical characterizations of microemulsions, formulating and preparing microemulsion-insecticides as aerosol-insecticides, and bioefficacy test of the formulated products on insect pests.

The optimum compositions and concentrations for both W/O-μE and O/W-μE systems were determined. Ternary phase diagrams systems were carried out to determine suitable surfactant or mixed surfactants and/or co-surfactants for W/O-μE and O/W-μE systems as a base for palm methyl esters aerosol insecticides formulations. The physical stability at ambient and high temperatures, particles size, viscosity and the mist behaviour of the palm methyl esters aerosol insecticides were studied. Both systems were used to formulate W/O-μE aerosol insecticides to be applied as Flying Insect Killer (FIK) aerosol-spray product to control flying insects such as mosquitoes and houseflies while O/W-μE aerosol insecticides was applied as Crawling Insect Killer (CIK) aerosol-spray product to control crawling insects such as cockroaches and ants respectively. Other than that, the bioefficacy tests on both products were also determined. The invension of this system consisting mixed surfactants, PME, and non-toxic co-surfactant has great potential to be used in preparing palm methyl esters aerosol insecticides which are more environmental and end-users friendly.

Novelty of palm methyl esters aerosol-insecticides:

- Palm methyl esters microemulsions (μE), a nanotechnology system (< 100 nm of particles size) was used instead of solvent-based or macroemulsion (>500 nm particles size).
- The bases are formulated using palm methyl esters, which are renewable, biodegradable and environmentally friendly.
- The inerts (palm methyl esters microemulsions) are also less hazardous to end-users and aquatic organisms.
- An innovative (μE) aerosol-insecticides technology using green palm-based materials.
- Contains propellant which has an ozone depleting potential (ODP) of zero.

1.4 Objectives

- To formulate microemulsion-insecticides using palm oil-based materials as the oil phases, emulsifiers and/or co-emulsifier.

- To formulate aerosol insecticides formed with palm-based microemulsions against insect pests for public used.
- To characterize the physicochemical properties of microemulsion (μE) such as their conductivity, viscosity, pH, and particle size.
- To evaluate the bioefficacy of household insecticides.

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