UNIVERSITI PUTRA MALAYSIA

DEVELOPMENT OF NANOEMULSION CONTAINING *Parthenium hysterophorus* L. CRUDE EXTRACT FOR PRE-EMERGENCE HERBICIDE FORMULATION

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By

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A nanoemulsion containing *Parthenium hysterophorus* crude extract (PHCE) was developed with the aim to introduced nanoemulsion as delivery system for PHCE in pre-emergence herbicide formulation. *Parthenium hysterophorus* is a noxious plant containing allelochemical parthenin with an ability to inhibit the growth of neighbouring plants. The study on herbicidal activity of *P. hysterophorus* was widely reported however, the study on development of PHCE in herbicide formulation was less explored. Nanoemulsion was proposed as the delivery system for PHCE to increase the efficiency and stability of plant crude extract. The use of natural-based active ingredients and adjuvants in nanoemulsion herbicide formulation was thought to be more environmental friendly compared to conventional synthetic herbicide.

In the initial stage of the study, the local invasive weed known as Rumput Miang Mexico (RMM) was genetically identified as *P. hysterophorus* by using Internal Transcribes Spacer (ITS) region-based. The collected DNA sequence was registered in NCBI GenBank® under accession number of KY249559. The amount of the main allelochemical (parthenin) was determined to be 26.74 mg g^{-1} of dry plant by using UPLC-MS spectroscopy analysis.

In the development of *P. hysterophorus* nanoemulsion (PHNE) formulation, several oil were tested to solubilize PHCE. Form the result, palm kernel oil ester (PKOE) was chosen as the oil phase due to the highest concentration of parthenin (2.43 ± 0.07 mg mL^{-1}) was solubilized as compared to the other tested oils. Phase behaviour of *P. hysterophorus* crude extract (PHCE) was studied by construction of ternary phase diagrams using different surfactants. High

solubilisation capacity (isotropic region) was obtained by mixture of surfactants Tensiofix and Tween 80 (ratio 8:2) compared to the other mixture of surfactants. One spot was selected as pre-formulation from the isotropic region of PKOE: PHCE (3:1)/ Tensiofix: Tween 80 (8:2)/ water system and further become the starting point for composition optimization of PHNE.

Mixture Experimental Design (MED) modelling was used to determine the optimal composition of PHNE. From the optimization process, analysis of variance (ANOVA) showed a fit quadratic polynomial model for four independent variables (PKOE, Tensiofix: Tween 80 (8:2), PHCE and water) in relation to particle size. The model suggested an optimal formulation composition which contained 30.91 % of PKOE, 28.48 % of mixed surfactants (Tensiofix and Tween 80, 8:2), 28.32 % of water and 12.29 % of PHCE with the smallest particle size (140.10 nm) was obtained. The reading of both experimental and predicted particle size in the verification experiment was acceptable with a residual standard error (RSE) less than 2 %.

Under the optimal condition, the particle of final formulation was clearly shown by morphology analysis to be spherical in shape and demonstrated an average particle size which is in agreement with dynamic light scattering analysis. The optimized nanoemulsion demonstrated a good stability (no phase separation) under centrifugation and different storage conditions (25 ± 5 °C and 45 °C). Manipulating time as parameter, nanoemulsion stored for 60 days exhibit monodisperse emulsion with narrow distribution of polydispersity index values (0.07 to 0.08), stable zeta potential values (± 25 mV) and slight increase of particle size (143.0 nm to 218.5 nm). The increase in particle size over time might have been contributed by Oswald ripening phenomenon which was shown by a linear graph from Oswald ripening rate analysis.

In the efficiency test, PHNE was compared with PHCE in terms of surface tension and phytotoxicity against *Diodia ocimifolia* seed. PHNE displayed lower surface tension in comparison to PHCE which suggested that nanoemulsion might have enhanced the ability of the plant crude extract to penetrate through plant seed coat. In addition, PHNE was shown to cause total inhibition of *D. ocimifolia* seed at lower concentration (5 g L^{-1}) than PHCE (10 g L^{-1}). The ability of PHCE to inhibit *D. ocimifolia* seed was almost doubled after being formulated in nanoemulsion system by having half effective dose (ED_{50}) value for PHNE (1.93 g L^{-1}) and PHCE (3.60 g L^{-1}). Similarly to the seed germination, PHNE shown to be more efficient in reducing the growth of radicle and shoots of *D. ocimifolia* in compared to the plant crude extract. The finding of the research could potentially serve as a platform for development of a natural herbicide formulation from crude plant extract for green weed management.

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PEMBANGUNAN NANOEMULSI MENGANDUNGI EKSTRAK MENTAH *Parthenium hysterophorus* L. SEBAGAI FORMULASI RACUN RUMPAI PRA-PERTUMBUHAN

Oleh

NOORUL JANNAH BINTI ZAINUDDIN

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Suatu formulasi nanoemulsi yang mengandungi ekstrak mentah *Parthenium hysterophorus* (PHCE) telah dibangunkan bagi memperkenalkan nanoemulsi sebagai sistem penghantaran bagi PHCE di dalam racun rumpai pra-pertumbuhan. *Parthenium hysterophorus* adalah sejenis tumbuhan beracun yang mengandungi allelokimia parthenin dan berupaya merencat pertumbuhan tumbuhan lain. Kajian mengenai aktiviti racun rumpai PHCE telah banyak dilaporkan namun, kajian tentang pembangunan PHCE sebagai formulasi racun rumpai kurang diterokai. Nanoemulsi telah diperkenalkan sebagai sistem penghantaran bagi PHCE bagi meningkatkan keefisienan dan kestabilan ekstrak mentah tumbuhan. Penggunaan bahan aktif dan bahan tambahan semulajadi di dalam formulasi racun rumpai dianggap lebih bersifat mesra alam berbanding racun rumpai sintetik konvensional.

Pada permulaan kajian, rumput tempatan yang dikenali sebagai Rumput Miang Mexico (RMM) telah dikenalpasti secara genetik sebagai *P. hysterophorus* dengan menggunakan penanda *Internal Transcribe Spacer* (ITS). Jujukan DNA yang diperoleh telah didaftarkan di dalam GenBank ®NCBI di bawah nombor pendaftaran KY249559. Melalui analisis spektroskopi UPLC-MS, sejumlah 26.74 mg g^{-1} bahan allelokimia utama (parthenin) telah diperoleh daripada jisim kering PHCE.

Di dalam pembangunan sistem nanoemulsi *P. hysterophorus* (PHNE), beberapa jenis minyak telah diuji untuk melarutkan PHCE. Keputusan yang diperoleh menunjukkan ester minyak isirong sawit (PKOE) telah dipilih sebagai fasa minyak berdasarkan kebolehannya melarutkan parthenin sebanyak 2.43 mg mL^{-}

^{1} berbanding minyak yang lain. Perlakuan fasa ekstrak mentah PHCE telah ditunjukkan melalui pembinaan diagram tiga fasa menggunakan beberapa jenis surfaktan. Campuran surfaktan Tensiofix dan Tween 80 (8:2) telah menghasilkan kawasan isotropik tertinggi berbanding campuran surfaktan yang lain. Satu titik telah dipilih sebagai pra-formulasi dari kawasan isotropik pada sistem PKOE: PHCE (3:1) /Tensiofix: Tween 80 (8:2)/ air dan seterusnya dijadikan titik permulaan untuk pengoptimunan komposisi PHNE.

Pengoptimunan kompoisisi PHNE telah ditentukan melalui kaedah rekabentuk eksperimen campuran (MED*)*. Melalui kaedah tersebut, analisis varians (ANOVA) telah memberikan sebuah model polinomial kuadratik yang menunjukkan hubungan di antara empat pembolehubah (PKOE, Tensiofix: Tween 80 (8:2), PHCE dan air) dengan saiz partikel. Model yang diperoleh telah mencadangkan komposisi formulasi yang mengandungi 30.91 % PKOE, 28.48 % campuran surfaktan (Tensiofix dan Tween 80, 8:2), 28.32 % air dan 12.29 % PHCE dengan nilai saiz partikel yang minimum (140.10 nm). Pada eksperimen pengesahan, hasil bacaan sebenar saiz partikel adalah selaras dengan bacaan ramalan di mana nilai ralat baki standard (RSE) yang lebih rendah daripada 2 %.

Pada keadaan optimum, partikel formulasi menunjukkan bentuk sfera yang jelas ketika analisis morfologi dan memberikan purata saiz partikel yang selari dengan analisis dari penganalisa saiz (*dynamic scattering light*). Formulasi optimum menunjukkan kestabilan yang tinggi (tiada pemisahan fasa) ketika ujian daya empar dan ujian penyimpanan pada keadaan yang berbeza (25 ± 5 °C and 45 °C). Apabila masa dimanipulasi sebagai parameter, nanoemulsi yang disimpan selama 60 hari mempamerkan emulsi monoserakan dengan purata indeks poliserakan (PDI) yang kecil (0.07 to 0.08), nilai zeta potensi yang stabil (±25 mV) dan sedikit peningkatan pada saiz partikel (143.0 nm kepada 218.5 nm. Peningkatan saiz partikel adalah disebabkan oleh fenomena pematangan Oswald yang ditunjukkan oleh graf linear pada analisis kadar pematangan Oswald.

Pada ujian keefisienan, PHNE telah dibandingkan dengan PHCE dari segi tekanan permukaan dan ketoksikan terhadap biji benih *Diodia ocimifolia*. PHNE memberikan nilai tekanan permukaan yang rendah berbanding PHCE yang mencadangkan nanoemulsi mungkin berupaya meningkatkan kebolehan ektrak tumbuhan untuk menembusi kulit biji benih tumbuhan. Selain itu, PHNE merencat secara menyeluruh pertumbuhan biji benih *D. ocimifolia* pada kepekatan yang lebih rendah (5 g L^{-1}) berbanding PHCE (10 g L^{-1}). Kebolehan PHCE untuk merencat pertumbuhan *D. ocimifolia* telah digandakan hampir dua kali setelah diformulasi ke dalam sistem nanoemulsi dengan menunjukkan nilai dos separa efektif (ED_{50}) bagi PHNE (1.93 g L^{-1}) and PHCE (3.60 g L^{-1}). Selari dengan ujian pertumbuhan biji benih, nanoemulsi turut menunjukkan keefisienan yang lebih baik di dalam menghalang pertumbuhan pucuk dan akar *D. ocimifolia.* Hasil kajian ini berpotensi untuk menjadi platform bagi pembangunan formulasi racun rumpai semulajadi dengan tujuan pengurusan rumpai secara selamat.

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfillment of the requirement for the degree of Doctor of Philosophy. The members of the Supervisory Committee were as follows:

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MOA	Mode of action
ROS	Reactive oxygen species
nAChRs	Nicotinic acetylcholine receptors
HPPD	p-hydroxyphenylpyruvate dioxygenase
CIB	Central Insecticide Board
FDA	Food and Drug Administration
ALS	Acetolactate synthase
PPE	Personal Protective Equipment
Ai	Active ingredient
WP	Wettable powder
WDG	Water-Dispersible Granule
DF	Dry flowable
WSC	Water Soluble Concentrate
E/EC	Emulsifiable Concentrate
AS	Aqueous Suspension
O/W	Oil-in-water
W/O	Water-in-oil
HLB	Hydrophilic lipophilic balance
LC_{50}	50% of lethal concentration
LAPG	Long-chain alkylpolyglucosides
ED_{50}	50% of effective dose
CMC	Critical micelle concentration
MED	Mixture experimental design

DNA	Deoxyribonucleic acid
PKOE	Palm kernel oil ester
MCT	Medium chain triglyceride
PCR	Polymerase chain reaction
ITS	Internal Transcribes Spacer
nBLAST	Nucleotide Basic Local Alignment Search Tool
NCBI	National Institute of Biotechnology Information
TLC	Thin layer chromatography
UV	Ultraviolet
FTIR	Fourier transform infrared
NMR	Nuclear magnetic resonance
$CDCl_3$	Deuterated chloroform
LCMS	Liquid chromatography mass spectrometry
UPLC	Ultra performance liquid chromatography
SQD	Single Quadrupole Detector
APG	Alkyl polyglucoside
ANOVA	Analysis of variance
R^2	Coefficient of determination
DLS	Dynamic light scattering
RG%	Percentage of relative germination
RMM	Rumput Miang Mexico
RNA	Ribonucleic acid
SOR	Surfactant-to-oil ratio
PDI	Polydispersity index

RSE	Residual standard error
DAT	Day after treatment
PHCE	*Parthenium hysterophorus* crude extract
PHNE	*Parthenium hysterophorus* nanoemulsion
GP	Germination percentage
GI	Germination index

CHAPTER 1

INTRODUCTION

1.1 Research Background

Weed has been defined as a group of aggressive unwanted plants that grow competitively together with crops or grown on wasted land. The study on weed management is of fundamental importance for successful crop protection. Although its population only account for less than 1 % of the total plant species on earth, the impact it gave was huge enough (Qasem and Foy, 2001). Interestingly, in a controlled environment and balance ecosystem, weeds should not become a problem because of their own purpose of existence. Thus, human being is responsible for finding a solution for the imbalance growth of these species which caused by rapid development of human activities.

Currently, there are approximately 800 weed species in our nature environment, many of which are invasive (Ahmad *et al.*, 2016). Management of weed species is dominantly by herbicide, integrated with other control measures including cultural practices, prescribed burning, animal grazing, and to a certain extent, followed by manual and mechanical roughing. Reliance upon herbicide as the primary method for weed control was associated with herbicide-resistant weeds and uses of a toxic chemical in the formulation. Thus, plant based herbicide formulation has gained great attention as a source of environmental friendly active component.

Ideally in development of a product formulation containing plant material, single or pure compound is preferable over mixture substances due to its specificity in method development and mechanism of action. However, the use of isolated compound is not always the most viable or successful approach. Isolation process frequently associated with difficulty in the purification steps with most of the compound loss during the process and reduction in terms of reactivity for some isolated compound at the end of isolation process. There were increasing numbers of publication reveal on relation of plant extract with nanotechnology-based system basically for the reason to reduce waste in product development. Moreover, mixture compound could possibly act in multiple targets due to synergism between different compounds (Gertsch, 2011; Zorzi *et al.*, 2015).

Nanotechnology offer an encapsulation of active ingredient (Ai) in various field of agriculture with the aim to enhance the solubility of poorly soluble Ai, to protect the Ai from photodegradation and volatilization, and to increase the uptake of Ai into variety of membrane barrier (Melanie Kah and Hofmann, 2014). However, supporting data about nanoemulsion and herbicide remains scarce. Previous studies have shown the study of nanoformulated product by insect to support the enhanced uptake hypothesis. The lethal concentration (LC_{50}) of nanoemulsion loaded with neem oil against *Culex quiquefasciatus* decreased with particle size which was interpreted as indicating an increased uptake of smaller particles (Anjali *et al.*, 2012). In the second study, the efficacy of a nanoemulsion containing permethrin was significantly higher than that of the pure active ingredient against larvae of *Aedes aegypti* (LC_{50} nanopermethrin = 0.0063 mg L^{-1}, LC_{50} permethrin = 0.0199 mg L^{-1}), which was again indicating an increased uptake of the nanoformulated product (Kumar *et al.*, 2013).

Therefore, in this study *Parthenium hysterophorus* (*P. hysterophorus*) crude extract containing allelochemical was introduced as a new bioactive component in herbicide formulation. *P. hysterophorus* is one of the world's seven most devastating and aggressive weed commonly known as carrot grass and bitter weed (Patel, 2011). The plant contained allelochemical parthenin which play as major phytotoxic compound in the plant. Allelochemical is a chemical compound that influence growth, survival and reproduction of other organism (Dhillon and Mukhopadhyay, 2015). *P. hysterophorus* crude extract (PHCE) was formulated in nanoemulsion as a system to deliver the bioactive compounds through hydrophobic plant surface for natural weed management.

1.2 Problem statement

This study was conducted to develop a natural-based herbicide formulation for green weed management. Weed has become one of the major problem in agriculture industry where it was dominantly managed by using herbicide. However, conventional synthetic herbicide was reported to be toxic towards human health and caused environmental pollution (Masirah *et al.*, 2013). Furthermore, the number of weed resistance species was increasing from time to time (Heap, 2019) as a result from repeated used of the same chemical group of herbicide. Thus, natural-based component such as plant crude extract could become the bioactive ingredient for herbicide formulation in order to reduce its hazardous effect towards environment.

P. hysterophorus is a noxious weed and was widely reported to have the herbicidal properties by releasing allelochemicals to the neighbouring plants. This offer an opportunity for its crude extract as a natural herbicide in weed management *(Tanveer et al.*, 2015). Despite having various bioactive compounds, plant crude extract was always associated with lack of good physicochemical properties and short shelf-life (Zorzi *et al.*, 2015). The properties have precluded natural product for further product development. Combination of the natural active component and effective delivery system would serve as a safe and efficient herbicide formulation in the agriculture industry.

Nanotechnology in agriculture industry has received greater attention with various applications however, nanoemulsion for herbicide formulation remain less explored. A good delivery system not only contributing for its stability but also increase the bioavailability of the plant extract. A formulation containing a surfactant and having the properties of small in particle size might enhance the penetration of plant extract into plant cell. A well-design delivery system such as nanoemulsion could help in development an eco-friendly herbicide formulation containing *P. hysterophorus* crude extract (PHCE).

1.3 Significant of study

Above all problem arises, achievement on 100 % control of weeds is an unrealistic objective in a balanced ecosystem. However, an effort could be done as an alternative to reduce total reliance on synthetic herbicide which has affected the environment and economy due to toxicity and resistance of weeds issues. The use of plant extract in herbicide formulation could give advantages as it combines environmental friendly element and a new mode of action (MOA) into the formulation for a safer and efficient product.

Therefore, the ultimate goal of this study is to develop a stable nanoemulsion containing PHCE for an efficient pre-emergence herbicide formulation against one of the noxious weed in Malaysian Palm oil plantation, *Diodia ocimifolia*. To the best of our knowledge, this is the first study to report the encapsulation of PHCE in nanoemulsion for pre-emergence herbicide application. The study may serve as a based and reference for the development of nanoemulsion containing crude plant extract for herbicide formulation. The study will also benefit in increasing the value of unwanted plant such as *P. hysterophorus* by utilization of its bioactive compounds.

1.4 Objectives

The aim of the study was to develop nanoemulsion containing *P. hysterophorus* crude extract (PHCE) for pre-emergence herbicide formulation. This study was focused on qualitative and quantitative research as an approach for testing several objectives listed below:

1. To identify *P. hysterophorus* species by using genetic identification and determine the major allelochemical parthenin in PHCE
2. To determine the phase behaviour of PHCE in oil-in-water emulsion system
3. To formulate and optimize the nanoemulsion containing PHCE by Mixture Experimental Design (MED)

4. To determine the physicochemical characteristic of the *P. hysterophorus* nanoemulsion (PHNE)
5. To investigate the efficacy of PHNE against *Diodia ocimifolia*

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LIST OF PUBLICATIONS

Noorul Jannah Zainuddin, Norazlinaliza Salim, Siti Efliza Ashari, Norhayu Asib, Dzolkhifli Omar, and Gwendoline Ee Cheng Lian. Herbicidal activity of Parthenium weed (*Parthenium hysterophorus L.*) against slender button weed (*Diodia ocimifolia*). (Submitted- Archives of Phytopathology and Plant Protection)

Noorul Jannah Zainuddin, Norazlinaliza Salim, Siti Efliza Ashari, Norhayu Asib, Dzolkhifli Omar, and Gwendoline Ee Cheng Lian. 2019. Optimization and Characterization of Palm Oil-based Nanoemulsion Loaded with *Parthenium hysterophorus* Crude Extract for Natural Herbicide Formulation. *Journal of Oleo Science*, 68(8), 1-11.

Noorul Jannah Zainuddin, Norazlinaliza Salim, Siti Efliza Ashari, Norhayu Asib, Dzolkhifli Omar, and Gwendoline Ee Cheng Lian. Phase behaviour of *Parthenium hysterophorus* crude extract in oil-in-water emulsion system. (In process for submission)

Conferences

Zainuddin, N.J., Basri, M., Omar, D., Ee, G.C.L. and Salim, N. *Parthenium hysterophorus L.* extract as potential bioactive compound in bioherbicide formulation. 2nd Joint Symposium of Universiti Putra Malaysia and University of Hyogo, 22-23 October 2015, Hyogo, Japan. (Oral and poster presenter)

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