UNIVERSITI PUTRA MALAYSIA

PERFORMANCE AND PHYSICAL PROPERTY EVALUATION OF PALM OIL-BASED ADJUVANT FORMULATED IN GLYPHOSATE ISOPROPYLAMINE AND GLYPHOSATE MONOAMMONIUM FOR WEED CONTROL

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By

MEOR BADLI SHAH BIN AHMAD RAFIE

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

June 2014

Dedicated to:
My supportive father, Ahmad Rafie B. Yeop Abd Aziz and my forever-loving mother, Siti Maimunah Bt. Syed Jaffar,
My beloved wife, Mimizufa Bt. Waheed and my daughters, Zalia Akhmaliza Bt. Meor Badli Shah and Zalina Azwaliza Bt. Meor Badli Shah,
whose never ending support, believe, love and patience made all this work possible.
Alhamdulillah, Syukur PadaMu Allah S.W.T.

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirements for the degree of Master of Science

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In all glyphosate herbicide formulations, an adjuvant is usually added or incorporated to enhance the wetting, spreading and penetration of the active ingredient on the leaf surface. However, conventional adjuvants have side effects such as aquatic toxicity and irritation to skin and eyes. Palm oil-based adjuvants being renewable, environmental friendly, less flammable (due to higher flash points) and cause fewer medical problems and allergies as to end-users or spraying operators provides the alternative solution to the problems. The general objective of the experiment is to evaluate the performance and physical properties of palm oil-based adjuvant or blended adjuvant formulated in the glyphosate isopropylamine and glyphosate monoammonium herbicides from the number of adjuvants developed by the Advanced Oleochemical Technology Division of Malaysian Palm Oil Board (AOTD, MPOB). Four experiments were conducted which includes bioefficacy evaluation of seven type of palm oil based adjuvants formulated in glyphosate isopropylamine and glyphosate monoammonium herbicides, physical properties evaluation of the selected formulations, influences of different adjuvant concentrations on the performance of the selected glyphosate formulations and rainfastness evaluation. The results showed that MAGIPAS3 and MAGIPAS4 with 10% adjuvant concentration were the most effective glyphosate isopropylamine formulation as it has given visual weed mortality of 95.00% and 95.55% on *Paspalum conjugatum,* 93.90 % and 96.00% on *Asystasia gangetica* and 95.10% and 98.07% on *Ottochloa nodosa* respectively at 14 Days After Treatment (DAT) which is significantly higher than other formulation and comparable with the standard product Roundup and Asset. For glyphosate monoammonium formulations, MAGMAS6 and MAGMAS7 with 10% adjuvant concentrations were the most effective formulation with the visual weed mortality of 85.00% and 95.24% on *Paspalum conjugatum,* 85.00 % and 95.00% *on Asystasia gangetica,* 95.02% and 98.00% on *Ottochloa nodosa* respectively. In the physical properties evaluation, all the glyphosate isopropylamine and glyphosate

monoammonium samples formulated with palm oil based adjuvant showed surface tension below 40 mN/m for all the three adjuvant concentrations of 7.5%, 10% and 12.5%. Surface tension for MAGIPAS4 with 7.5% (35.25 mN/m), 10% (34.86 mN/m) and 12.5% (34.98 mN/m) was significantly lower than the standard product Asset (45.03 mN/m) but no significant different with Roundup (40.05 mN/m). As for glyphosate monoammonium, MAGMAS6 and MAGMAS7 at all adjuvant concentrations showed significantly lower surface tension (39.55 mN/m and 34.20 mN/m respectively) than the standard product of Ammo Supre (50.57 mN/m) and water (control) (72.24 mN/m). MAGIPAS4 at 10% concentration showed lower mean contact angle at 32.6° on *Paspalum conjugatum* and 27.7° on *Asystasia gangetica* indicating good wetting properties on the leaf surface. MAGMAS7 at 10% adjuvant concentration recorded lower mean contact angle on all weed leaf surface (74.6° on *Paspalum conjugatum,* 52.7° on *Asystasia gangetica,* 58.2° on *Ottochloa nodosa* and 37.5° on *Clidemia hirta)* compared to MAGMAS6. MAGIPAS4 and MAGMAS7 at 10% adjuvant concentration is the optimum formulation that consistently provide the largest droplet spread area and higher droplet spread coefficient on leaf surfaces. In the influence of adjuvant concentration study, MAGIPAS4 has edged over MAGIPAS3 significantly in term of visual weed mortality for adjuvant concentrations of 10.0 and 12.5% on *P. conjugatum* and *A. gangetica* but were equal to the standard product of Roundup and Asset. While MAGMAS7 at all concentration has shown superiority over MAGMAS6 in term of visual weed mortality, fresh and dry weight reduction and chlorophyll content degradation for *P. conjugatum* and *A. gangetica* resulting from better droplet spread area and spread coefficient. In the rainfastness experiment, none of the glyphosate isoropylamine or glyphosate monoammonium formulations with palm oil based adjuvant formulated samples were capable of providing rainfastness effect 30 minutes post-spraying on *P. conjugatum* and *A. gangetica.* However, MAGIPAS4 with 10% concentration proved to be a rainfast formulation on *P. conjugatum* 90 minutes post-spraying. For glyphosate monoammonium, none of the formulations were rainfast 90 minutes post-spraying. The increase of adjuvant concentrations did not increase the level of rainfastness of the glyphosate formulations.

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PENENTUAN PRESTASI DAN CIRI-CIRI FIZIKAL ADJUVAN BERASASKAN MINYAK SAWIT YANG DIFORMULASIKAN DALAM GLIFOSAT ISOPROPILAMINE DAN GLIFOSAT MONAMMONIUM UNTUK KAWALAN RUMPAI

Oleh

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Adjuvan selalunya ditambah atau dicampurkan dalam formulasi racun rumpai glifosat untuk meningkatkan kebolehbasahan, penyerakan dan penembusan bahan aktif di atas permukaan daun. Walaubagaimanapun, adjuvan konvensional boleh menyebabkan kesan sampingan seperti toksisiti kepada hidupan air dan kerengsaan kepada kulit dan mata. Adjuvan berasaskan minyak sawit pula adalah antara alternatif penyelesaian masalah ini kerana ianya boleh diperbaharui, mesra alam, kurang mudah terbakar (disebabkan oleh takat kilat yang tinggi) dan kurang menyebabkan masalah perubatan dan alergi kepada pengguna atau operator penyemburan. Objektif am eksperimen ini adalah untuk mengkaji prestasi dan ciri-ciri fizikal adjuvan atau campuran adjuvan berasaskan minyak sawit yang diformulasikan di dalam racun rumpai glifosat isopropilamine and glifosat monoammonium daripada beberapa jenis adjuvan yang telah dicipta oleh Advanced Oleochemical Technology Division Malaysian Palm Oil Board (AOTD, MPOB). Empat jenis eksperimen telah dilakukan termasuk kajian keberkesanan-bio tujuh jenis adjuvant yang diformulasikan didalam racun rumpai glifosat isopropilamine dan glifosat monoammonium, kajian ciri-ciri fizikal formulasi yang terpilih, pengaruh konsentrasi adjuvan terhadap keberkesanan formulasi glifosat yang terpilih dan kajian kebolehtahanan hujan. Keputusan kajian menunjukkan bahawa formulasi MAGIPAS3 dan MAGIPAS4 pada konsentrasi adjuvant 10% adalah formulasi glifosat isopropilamine yang paling berkesan oleh kerana ia telah memberi kesan peratus kematian visual sebanyak of 95.00% dan 95.55% ke atas *Paspalum conjugatum*, 93.90% dan 96.00% ke atas *Asystasia gangetica* dan 95.10% serta 98.07% ke atas *Ottochloa nodosa* masing-masing pada 14 Hari Selepas Rawatan (HSR) di mana ia adalah lebih tinggi secara signifikan berbanding formulasi sampel lain dan adalah setara dengan produk piawai Roundup dan Asset. Bagi formulasi glifosat monoammonium pula, sampel MAGMA10% and MAGMAS7 pada konsentrasi 10% adalah formulasi yang paling efektif dengan peratus kematian visual masing-masing sebanyak 85.00% dan 95.24% ke atas *Paspalum conjugatum*, 85.00%

dan 95.00% ke atas *Asystasia gangetica*, dan 95.02% serta 98.00% ke atas *Ottochloa nodosa*. Dalam kajian ciri-ciri fizikal, kesemua sampel formulasi glifosat isopropilamine dan glifosat monoammonium menggunakan adjuvan berasaskan minyak sawit menunjukkan bacaan ketegangan permukaan di bawah 40 mN/m untuk tiga jenis konsentrasi adjuvan iaitu 7.5%, 10% dan 12.5%. MAGIPAS4 pada konsentrasi adjuvan 7.5% (35.25 mN/m), 10% (34.86 mN/m) dan 12.5% (34.98 mN/m) menunjukkan bacaan ketegangan permukaan yang lebih rendah secara signifikan berbanding produk piawai Asset (45.03 mN/m) tetapi tiada perbezaan signifikan dengan Roundup (40.05 mN/m). Untuk formulasi glifosat monoammonium, kedua-dua MAGMAS6 dan MAGMAS7 pada kesemua konsentrasi adjuvan menunjukkan bacaan ketegangan permukaan secara signifikan (39.55 mN/m and 34.20 mN/m masing-masing) lebih rendah berbanding produk piawai Ammo Supre (50.57 mN/m) dan air (kawalan) (72.24 mN/m). MAGIPAS4 pada konsentrasi 10% menunjukkan sudut sentuhan yang lebih rendah pada 32.6° di atas permukaan daun *Paspalum conjugatum* dan 27.7° di atas permukaan daun *Asystasia gangetica* menunjukkan tahap kebolehbasahan titisan yang baik di atas permukaan daun. MAGMAS7 pada konsentrasi adjuvan 10% secara konsisten menunjukkan sudut sentuhan yang lebih rendah di atas permukaan daun kesemua jenis rumpai yang dikaji (74.6° pada *Paspalum conjugatum*, 52.7° pada *Asystasia gangetica*, 58.2° pada *Ottochloa nodosa* dan 37.5° pada *Clidemia hirta*) berbanding dengan formulasi MAGMAS6. Formulasi MAGIPAS4 dan MAGMAS7 pada konsentrasi adjuvan 10% telah memberi luas sebaran titisan dan koefisi sebaran titisan yang tertinggi secara konsisten di atas permukaan daun. Dalam eksperimen pengaruh perbezaan konsentrasi adjuvan, MAGIPAS4 telah menunjukkan kelebihan berbanding MAGIPAS3 secara signifikan dari segi peratus kematian visual untuk konsentrasi adjuvan 10 dan 12.5% untuk *P.conjugatum* dan *A. gangetica* tetapi adalah setara dengan produk piawai Roundup dan Asset. Manakala, MAGMAS7 pada kesemua konsentrasi adjuvan menunjukkan kelebihan yang nyata berbanding MAGMAS6 dari segi kematian visual, penurunan berat basah dan berat kering dan pengurangan kandungan klorofil untuk *P. conjugatum* dan *A. gangetica* berikutan daripada luas sebaran dan koefisi sebaran titisan yang lebih baik. Dalam kajian kebolehtahanan hujan, tiada satu pun formulasi glifosat isopropilamine atau glifosat monoammonium bersama adjuvan minyak sawit yang mampu memberikan kesan kebolehtahanan hujan 30 minit selepas semburan ke atas *P. conjugatum* dan *A. gangetica*. Walaubagaimana pun MAGIPAS4 pada konsentrasi adjuvan 10% terbukti adalah formulasi yang boleh tahan hujan untuk *P. conjugatum* 90 minit selepas semburan. Untuk glifosat monoammonium, tiada satu pun formulasi yang tahan hujan selepas simulasi hujan turun 90 minit selepas semburan. Keputusan kajian ini memberi kesimpulan bahawa peningkatan konsentrasi adjuvan tidak meningkatkan tahap kebolehtahanan hujan formulasi glifosat.

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I certify that a Thesis Examination Committee has met on 16 June 2014 to conduct the final examination of Meor Badli Shah Bin Ahmad Rafie on his thesis entitled "Performance and Physical Property Evaluation of Palm Oil-Based Adjuvant Formulated in Glyphosate Isopropylamine and Glyphosate Monoammonium for Weed Control" 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 Master of Science.

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LIST OF ABBREVIATIONS

A. gangetica	*Asystasia gangetica*
B. latifolia	*Borreria latifolia*
C. dactylon	*Cynodon dactylon*
C. hirta	*Clidemia hirta*
C. rotundus	*Cyperus rotundus*
D. ocimifolia	*Diodia ocimifolia*
E. indica	*Eleusine indica*
I. cylindrica	*Imperata cylindrical*
M. micrantha	*Mikania micrantha*
P. conjugatum	*Paspalum conjugatum*
ANOVA	Analysis of Variance
DAT	Day After Treatment
HLB	Hydrophilic Lipophilic Balance
GLM	General Linear Module
SAS	Statistical Analysis System
AOTD, MPOB	Advance Oleochemical Technology Division, Malaysian Palm Oil Board
SYABAS	Syarikat Bekalan Air Selangor
UPM	Universiti Putra Malaysia
NPK	Nitrogen Phosporus Kalium
CRD	Complete Randomized Design
RCBD	Randomized Complete Block Design
a.i.	active ingredient
a.e.	acid equivalent
mN/m	milliNewton per metre
w/v	weight/volume
w/w	weight/weight
®	Registered Trademark

CHAPTER 1

INTRODUCTION

1.0 Introduction

Glyphosate herbicide is one of the major weed control products that are being used in the oil palm plantation and other major crops in Malaysia. It is estimated that the total Glyphosate market in Malaysia is about 15 Million litres which is valued around RM250 Million (Ismail *et al.*, 2010). Global economic value of glyphosate is valued around USD20 Billion in 2010 (Frabotta, 2012). In all glyphosate formulations, an adjuvant is usually being added or incorporated to enhance the effectiveness of the product. The economic value of the global adjuvant market is estimated to be approximately USD1.5 Billion (Underwood, 2007).

Adjuvant system is important in herbicide compositions as promoters of wetting, spreading and penetration of the active agent. The adjuvant can enhance the performance of the herbicide, but it can also have side effects such as aquatic toxicity and irritation to skin and eyes. The commercial glyphosate herbicide product, Roundup is formulated with the POEA (polyethoxylated tallow amine) or also known as TAE (tallow amine ethoxylate) adjuvant. This formulation however was found be highly toxic to animals and humans (Hedberg *et al.,* 2010; Alteri, 2009; Benachour and Seralini*,* 2009; Hedberg and Wallin, 2010 and Zeliger, 2008). Recent studies by Mesnage *et al.,* (2012) has reported that the polyethoxylated tallowamine (POEA) or tallow amine ethoxylate (TAE), clearly appears to be the most toxic principle against human cell. Other studies by Benachour and Seralini (2009) reported that POEA adjuvants in Roundup formulation change human cell permeability and amplify toxicity induced already by glyphosate, through apoptosis and necrosis. Hence, there is a real safety problem of using POEA or TAE adjuvants in glyphosate formulations.

Palm oil based adjuvant which is derived from the palm oil itself is an environmentally friendly compound. Since it is natural oil it is renewable, biodegradable, non- flammable, harmless to the environment and less toxic to end-users (Ismail, 2007). It is considered safer than other conventional adjuvant since it causes fewer medical problems or allergies to the operators. Palm oil is the largest produced and consumed vegetable oil in the world accounting for 33% of world production and consumption of oil and fats. Interestingly, 14% of the world vegetable oil production comes from Malaysia with the major portion of it is palm oil (Source: USDA as cited in Malaysian Agribusiness Directory (2011-2012)). Total hectarage of oil palm area in Malaysia has reached 5.038 million hectare in 2012 and the production output has reached 18.8 million tonnes in 2012 (MPOB, 2012). Therefore due to the abundance of raw materials of palm oil, it is timely that more focus to develop and produce adjuvants from palm oil or in general term known as oleochemicals from palm oil. There are several groups of adjuvant derived from palm oil such as methyl ester, fatty acid, ester, fatty alcohol and alkyl polyglycoside (Henkel, 1995).

Recently, seven types of adjuvants or blended adjuvants originated from palm oil was developed by Advanced Oleochemical Technology Division, Malaysian Palm Oil Board (AOTD, MPOB) to be formulated with the glyphosate herbicides, of the isopropylamine and monoammonium salt. Research is needed to determine the bioefficacy and performance of these adjuvants in the glyphosate herbicide formulations.

1.1 Justification

Glyphosate herbicides formulated with palm oil based adjuvant is an environmentally friendly as the adjuvant is renewable, less flammable and safer than other glyphosate formulated with conventional adjuvants i.e. POEA or TAE adjuvants. Therefore the project is justified to encourage pesticide companies in Malaysia to produce glyphosate herbicides that is environmentally friendly and safer to human being, operators and workers.

1.2 Objectives of Experiment

The objective of the project was (1) to determine the most effective palm oil based adjuvant from the number of adjuvants or blended adjuvant developed by AOTD, MPOB (main objective) and (2) to evaluate the performance and physical properties of the palm oil based adjuvant formulated in the glyphosate isopropylamine and glyphosate monoammonium herbicides (specific objectives). The selected adjuvants will be commercialized by MPOB and its collaborators.

1.3 Hypothesis

Palm oil based adjuvant formulated in glyphosate herbicides influences the effectiveness of the glyphosate herbicides.

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