

THE EFFECT OF PALM-BASED SURFACTANTS AS SPRAY ADJUVANTS IN GLYPHOSATE ISOPROPYLAMINE (IPA)

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The utilization of some palm-based surfactants in glyphosate isopropylammonium (IPA) formulations were tested. Three groups of nonionic surfactants were used, i.e, fatty alcohol ethoxylate (FAE) with five moles of ethylene oxide (EO), 7 EO, 9 EO and 20 EO, fatty amine ethoxylate (FAmE) and alkylpolyglycosides (APG). *Cyperus rotundus*, a thin-leaf and tuber-producing weed was used to test the efficacy of glyphosate IPA formulations.

Twelve glyphosate IPA formulations were prepared and their stabilities tested at room temperature (RT) and 45°C. All the formulations were stable at RT, and nine appeared as one layer after keeping at 45°C for seven days.

Efficacy tests were carried out under full sunlight. The application rates were 2.0kg/ha, 1.5kg/ha and 1.0kg/ha of glyphosate IPA, the active ingredient (a.i) per hectare (ha), and the spraying volumes used were 200 litres (l) /ha and 400 l/ha. The surfactant concentrations in the spraying solutions varied from 0.1% to 0.35% (w / w). Visual observation and analysis of variance showed that six formulations gave very promising results for controlling *Cyperus rotundus*. The formulations were FAmE, FAE (20EO), FAmE IAPG (C8-C10), FAmE / APG (C8-C16), APG (C8-C10), and APG (C8-C16).

INTRODUCTION

Palm oil is the second most produced oil in the world after the soybean oil (Mielke, 1995). About 90% of it is used for food and the

remaining 10% for non-food products or oleochemicals (Salmiah, 1995). With the expected growth in palm oil production and encouragement for the industry to go downstream, oleochemicals are expected to become increasingly important because they are environmentally friendly. One of the potential uses of these products is to replace the less environmentally friendly hydrocarbons and their derivatives as inert ingredients in pesticide formulations. This paper discusses the effect of palm-based surfactants as adjuvants on the efficacy of glyphosate IPA spray.

Surfactants as Spray Adjuvant

Agrochemical adjuvants, or activity optimisers, are materials that enhance the activity of active ingredients, usually herbicides (Aumatell, 1996; Turner, 1985). The types of adjuvants are surfactants, emulsifiable oils, ammonium sulphate and related compounds. This paper will only highlight the utilization of palm-based surfactants as adjuvants in herbicide application.

Surfactants are widely used to improve the effectiveness of herbicide application on foliage (Aumatell, 1996; Caseley, 1996). Surfactants lower the surface tension of the spray droplets, increase their spread over the surface of the foliage, and so enhance the uptake and efficacy of the herbicide. This lowers the application rate required as well as broadens the activity spectrum of the herbicide.

Generally, adjuvants can be divided into two categories: spray modifiers and activators (Aumatell, 1996). Spray modifiers improve the wetting and spreading properties of a spray solution by lowering its surface tension. This type of adjuvant is probably more important for non-systemic chemicals that require a more complete coverage for effectiveness. Activator adjuvants enhance foliage absorption of systemic chemicals, thus increasing their biological activity.

There are four major classes of surfactants i.e., anionic, cationic, non-ionic and amphoteric, according to the chemical structure of the hydrophilic portion of their molecules (Mc

Whorter, 1985). Anionic and cationic surfactants ionize in water to form negatively and positively charged substances. Amphoteric surfactants in aqueous solution contain both positive and negative charges on the same molecule.

The most widely used surfactants are non-ionic. The term 'non-ionic surfactant' normally refers to the derivatives of polyoxyethylene and polyoxypropylene (Gaskin and Holloway, 1992; Towson and Price, 1987; Caseley, 1996). They are non-electrolytes and usually chemically inactive in the presence of salts, unaffected by hard water, and have low mammalian toxicity and low phytotoxicity.

The effects of surfactants are variable and unpredictable, but almost all surfactants enhance phytotoxicity. In general, ethoxylated amine surfactants are better than non-ionic ones, with their performance improving with an increased ethylene oxide content. Turner and Loader (1980) found that hydrophilic non-ionic or cationic surfactants are more effective when no other additive is used. However, in the presence of ammonium sulphate, the more lipophilic surfactants give better results. Normally, the concentrations of surfactant(s) in spray solutions range from 0.1% to 2.5% (w/w).

Use of Biodegradable Surfactants

For ecological reasons, there is currently an interest to produce surfactants from natural raw materials, such as carbohydrates (starch, sugar, etc.) and natural oils and fats. The alkyl chain of the natural oils and/or their derivatives is rendered with the carbohydrate instead of ethylene oxide derived from mineral oil. For example, an alkyl polyglycoside (APG) is a non-ionic surfactant that has been produced from glucose molecules and the alkyl chain (Baumann and Biermann, 1994; Garst, 1995). APG exhibits excellent wetting and surface/interfacial tension reduction properties, outstanding hydrotropics and dispersing properties as well as superior electrolyte and hard water tolerances compared to surfactants from petroleum-based materials (Henkel, 1995).

Glyphosate

Glyphosate or N - (phosphonomethyl) glycine is one of the most important organophosphorus herbicides (Hance and Holly, 1990). It was described by Baird et al. 1971 (in Grossbard and Atkinson, 1985) and initially marketed under the trade name Roundup by Monsanto Co. USA, as the isopropylamine salt ($C_6H_{17}N_2O_5P$).



Glyphosate (Grossbard and Atkinson, 1985)

Glyphosate is a systemic, non-volatile, relatively non-selective and non-residual post-emergent herbicide. It is very effective on deep rooted perennial, annual and biennial species of grasses and sedges, but not very effective on broad leaf weeds. The herbicide has a relatively low mammalian toxicity, and is categorized as Class III poison by the Malaysia Pesticide Board. It is effective when applied to green foliage and has no effect on browned tissues of stems and roots. Symptoms on the shoots include chlorosis and necrosis, and the leaves of regrowing perennials are malformed or striated shooting (Hance and Holly, 1990). Visible effects appear on annual plants in 2 to 4 days and 7 to 10 days for perennial species.

Glyphosate prevents the formation of aromatic amino acids, such as phenylalanine (Pha), Tyrosine (Tyr) and Tryptophan (Tryp) by inhibiting the Shikimate pathway (Cole, 1982). Consequently, the plant experiences a depletion of metabolic pools, resulting in the slowing or cessation of all processes dependent on amino acids. The effects are generally slower in mature and more rapid in meristematic tissues (Devine, 1993).

Besides acting on the Shikimate pathway, glyphosate also decreases the chlorophyll content of green tissue (Grossbard and Atkinson, 1985). Glyphosate is an effective inhibitor to the formation of chlorophyll, as it inhibits the synthesis of 5 - aminolevulinic acid (ALA), a

common precursor for all porphyrins, including chlorophyll.

The effects of the chemical on the growth and yield of oil palm have been investigated (Teng and Lim, 1987). They found that application of glyphosate and dicamba at twice the recommended rate in young and mature oil palm did not cause any deleterious effect on mesocarp and kernel oils, vegetative growth, nutritional status, oil yield, and bunch characters of the oil palm. Normal fruit development of palms in the glyphosate and dicamba treatments also suggest that spraying of this mixture for general weed control does not cause parthenocarpy.

MATERIALS AND METHODS

Chemicals

The active ingredient used in all experiments was a technical grade glyphosate isopropylamine (MON 20058) supplied by Monsanto(M) Sdn. Bhd. It contained 54% active ingredient (a.i) or 40.5% acid equivalent (a.e). A commercial formulation, Roundup, also from Monsanto was used as a control.

The surfactants used were supplied by Henkel Oleochemical (M) Sdn. Bhd. and Albright & Wilson (S) Sdn. Bhd.. The surfactants used were alkylpolyglycosides (APG) with C8-C10 and C8-C16; fatty alcohol ethoxiates (FAE) with 5, 7, 9 and 20 moles of ethylene oxide (EO); and fatty amine ethoxylates (FAmE). The concentrations used were 0.1% to 0.35% (w/w).

Test Plants

The planting material was the underground tubers of *Cyperus rotundus* L. collected from Universiti Putra Malaysia (UPM). The tubers were sorted, trimmed of roots, rhizomes and shoots. The trimmed tubers were then pregerminated for three days under layers of wet paper. Four tubers were planted 1-2cm deep in a plastic pot (diameter 16cm and height 13cm) with a soil mixture of 55% clay, 34% silt and 11% sand.

The pots were arranged in blocks, and each block was about 25cm apart and placed in a

glass house at UPM. Watering was done twice daily and Nitrophoska fertilizer (15:15:15 NPK) was applied four weeks after planting. Spraying with glyphosate was done about 2 to 4 weeks later.

Spraying Equipment

The sprayer used was a conventional lever operated knapsack sprayer (Crossmark Sdn. Bhd.) with spraying pressure maintained at one bar by a spray management valve (Hoechst (M) Sdn. Bhd.). The nozzle was low drift (SD-02-110, Lurmark Ltd. U.K) and spraying was done from 50cm above the foliage.

Solubility and Stability Tests

The solubility of surfactant was measured by adding 7.5% (w/w) concentration of surfactant in water at room temperature (RT). The mixture was stirred at 500rpm with a magnetic stirrer until a homogenous solution was produced. The appearance of the solutions was determined by visual observation.

The glyphosate IPA formulations containing 41% (w/w) and 21% (w/w) glyphosate IPA and 10% surfactant (s) were prepared in the following sequence: glyphosate + surfactant + distilled water. The mixture was also stirred at 500rpm at RT until a homogenous solution was produced. The formulations were tested for stability by storing at room temperature (RT) for two months and 45°C for seven days.

Efficacy Test 1

The experiment was done in the open condition. Six pots containing six week-old plants (8-10 shoots per pot) were arranged in two blocks per treatment. The application rates were 2.0kg and 1.5kg a.i./ha with 200l/ha spraying volume. The concentrations of surfactant(s) in the spray solutions ranged from 0.18% to 0.35% (w/w). The treatments were glyphosate + FAmE, glyphosate + FAE (20EO), glyphosate + FAE (7EO) and FAmE, glyphosate + APG (C8-C16), Roundup and glyphosate only as T1, T2, T3, T4, T5 and T6, and the untreated control.

Efficacy Test 2

The experiment was also carried out in open condition. Five pots containing six weeks old plants (10-15 shoots per pot) were arranged in two blocks per treatment. The application rate and spraying volumes were 1.0kg a.i./ha and 400l/ha respectively. The concentration of surfactant(s) in spraying solutions was 0.1% (w/w). The treatments were glyphosate + FAE (20EO), glyphosate + FAmE, glyphosate + APG (C8-C10), glyphosate + APG (C8-C16), blended FAmE and FAE (7EO), blended FAmE and APG (C8-C10), blended FAmE and APG (C8-C16), Roundup, glyphosate only as T1, T2, T3, T4, T5, T6, T7, T8 and T9, and the untreated control.

Data Collections

A qualitative scale suggested by Burrill et al. (1976) was adopted to assess the effectiveness of weed control. Data were collected at 3 days, 7 days, 10 days and 14 days after treatment. The data were analysed by analysis of variance (ANOVA) and the least significant differences (LSD) calculated.

RESULTS AND DISCUSSIONS

The solubility test of surfactants derived from palm and palm kernel oils was carried out in water at RT as shown in **Table 1**. Three groups of non-ionic surfactants were used, *i.e.*, fatty alcohol ethoxylates (FAE) with 5, 7, 9 and 20 moles of ethylene oxide (EO), fatty amine ethoxylate (FAmE) and alkylpolyglycosides (APG). The concentration of surfactants used was 7.5% (w/w) and the hydrophilic-lipophilic balance (HLB) ranged from 10.4 to 15.4. It was found that all the surfactants were completely soluble in water at RT except FAE (5EO). The soluble and dispersible surfactants in water were indicated as a clear or transparent solution and a cloudy or translucent solution respectively. The solubilities of the surfactants were proportional to their HLB values. Similar results were also shown by Henkel (1993).

TABLE 1. SOLUBILITY OF SURFACTANTS IN WATER

surfactant (7.5% w/w)	HLB Value	Solubility in water (at RT)	
		Appearance	Appearance*
FAE(C12-C18); 5E0	10.4	D	S
FAE(C12-C18); 7 E 0	12.0	S	S
FAE(C12-C18); 9EO	13.4	S	S
FAE(C12-C18); 20E0	15.4	S	S
APG (C8-C10)	13.5	S	S
APG (C8-C16)	12.8	S	S
FAmE	14.3	S	S

* = adopted from Henkel, 1993.

HLB = Hydrophilic-lipophylic balance.

D = Dispersible indicates as a cloudy or translucent solution.

S = Soluble indicates as a clear or transparent solution.

Moore and Bell (1965) indicated that the optimum HLB number of surfactant(s) as wetting or dispersing agents was 13.4. Turner and Loader (1980) indicated that the hydrophilic types of non-ionic and cationic surfactants with HLB values from 9 to 16 were more effective when no other additive was used. However, in the presence of ammonium sulphate, the more lipophilic wetters gave better results.

The results in Table 2 showed that all surfactants in glyphosate IPA formulations were stable and appeared in one layer after storing for two months at RT. All glyphosate IPA formulations were also stable and appeared in one layer after keeping for seven days at 45°C, except the FAE with 5, 7 and 9 moles of EO that were unstable and separated into two layers. These results indicated that the FAE with 9, 7 and 5 moles of EO have their cloud points at about 45°C or lower. Tadros (1995) stated that the stability of non-ionic surfactants in water decreases with temperature using a cloud point as a stability limit. A cloud point is defined as the critical point of the surfactant or blended surfactants at 1%

concentration in which the solution becomes turbid and separates into two liquid layers above its cloud point.

The effects of surfactant or blended surfactants on the efficacy of glyphosate IPA formulations were studied on *Cyperus rotundus* under open conditions. *Cyperus rotundus* was chosen because it is one of the most serious weeds found in the tropics. It can produce tubers rapidly, and the tubers can survive in extreme conditions such as heat, flooding or anaerobic conditions. Glyphosate, a systemic herbicide is very effective on deep rooted perennial, annual and biennial species of grasses and sedges such as *Cyperus rotundus*, *Sorghum halepense*, *Imperata cylindrica*, *Paspalum conjugatum*, etc. (Hance and Holly, 1990).

The concentrations of surfactant or blended surfactants in the spraying solutions were 0.18% to 0.35% depending on the purity of the surfactant. The tested plants received rain at 2, 3, 4, 7, 8 and 13 days after the treatment. The results were not observed for three days due to heavy rain on day 2, 3 and 4 after the treatment and so the development of chlorosis

TABLE 2. THE STABILITY OF SURFACTANT(S) IN GLYPHOSATE IPA FORMULATIONS AT ROOM TEMPERATURE (RT)

10% (w/w) of surfactant(s) in glyphosate IPA	Appearance			
	RT		45°C	
	0 day	60 days	0 day	7 days
APG (C8-C10) 1 layer			
APG (C8-C16) 1 layer			
FAmE 1 layer			
FAE (5EO) 1 layer 1 layer	1 layer	2 layers
FAE (7EO) 1 layer 1 layer	1 layer	2 layers
FAE (9EO) 1 layer 1 layer	1 layer	2 layers
FAE (20EO) 1 layer 1 layer	1 layer
FAmE/FAE (5EO) 1 layer 1 layer	1 layer
FAmE/FAE (7EO) 1 layer 1 layer	1 layer
FAmE/FAE (9EO) 1 layer 1 layer	1 layer
FAmE/APG (C8-C10) 1 layer 1 layer	1 layer
FAmE/APG (C8-C16) 1 layer 1 layer 1 layer

was not clearly observed.

Efficacy test 1 (*Figures 1a* and *1b*) showed that all glyphosate IPA formulations gave comparable results to Roundup, but were significantly superior to using glyphosate by itself. This indicated that surfactant or blended surfactants increased the uptake and translocation of glyphosate, and therefore, enhanced its efficacy to kill *Cyperus rotundus*. Surfactant or blended surfactants also reduced the time required for glyphosate to act. The analysis of variance (*Table 3*) indicated significant differences between the treatments, and treatments linked with the rates of application at 7, 10 and 14 days after treatment. However, the effect of rate of application was only significant at seven days.

Moosavinia and Dore (1979) showed that better control with glyphosate on *Imperata*

cylindrica and *Cyperus rotundus* was achieved with plants grown under shade conditions. However, under full sunlight, several factors such as wind speed at the spraying time; the presence of water on leaves resulting from dew, rain or gutation; light intensity; and heavy rainfall soon after application will affect the efficacy of most foliage-applied pesticides (Caseley, 1996).

Previous studies (Gaskin and Holloway, 1992; Knoche and Bukovac, 1993; Caseley, 1996) and, in our recent study (*Figure 1*) found that surfactant or blended surfactants as spray adjuvant can enhance the efficacy of foliage-applied pesticides such as glyphosate. Surfactants lower the surface tension of the spray droplets, increase their spread over the surface of the foliage, and so enhance the uptake and efficacy of glyphosate. Therefore,

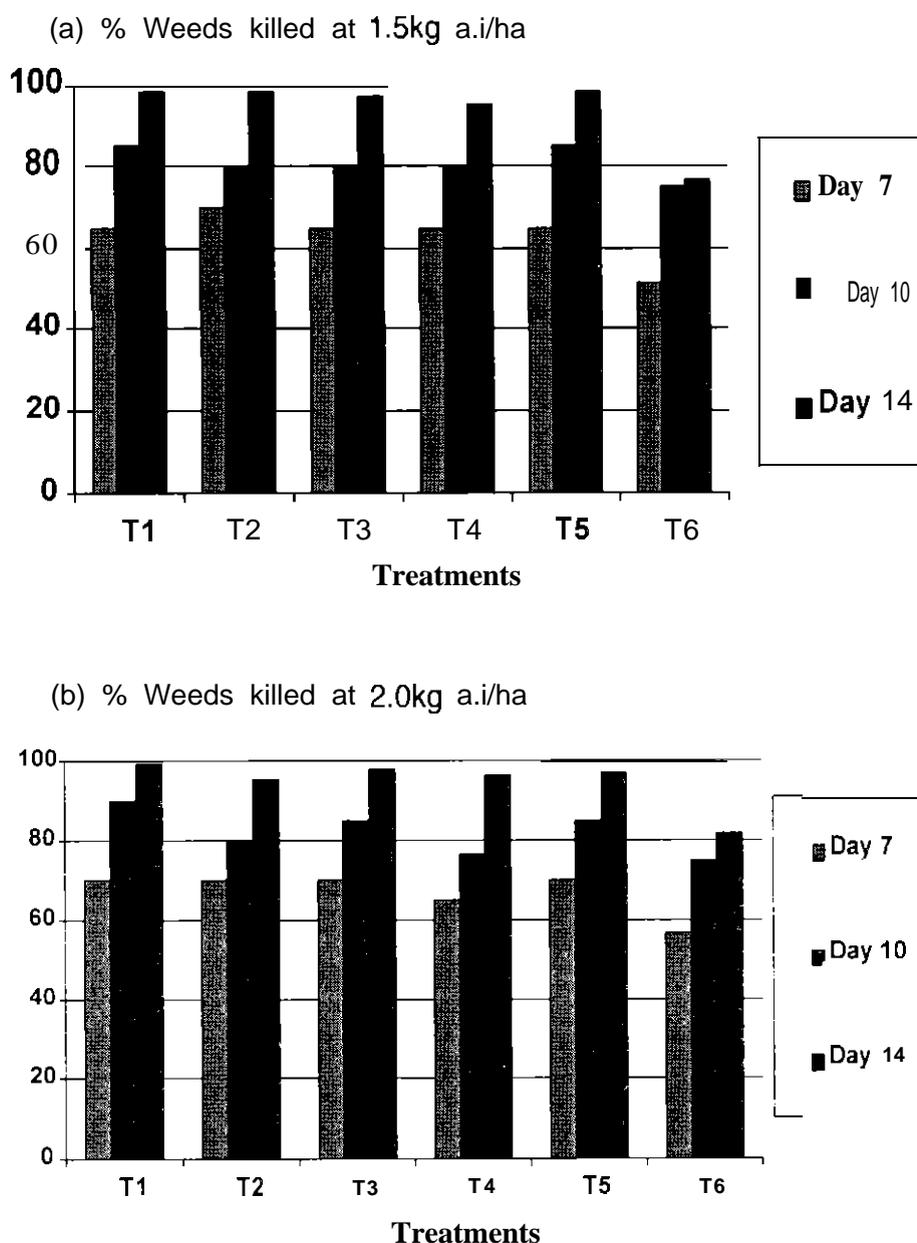


Figure 1. Bioefficacy 1: Effects of surfactants on the efficacy of glyphosate IPA at 1.5kg a.i/ha (Figure 1a) and 2.0kg a.i/ha (Figure 1b) on *Cyperus rotundus*

this can lower the application dosage per hectare as well as broaden the activity spectrum of the glyphosate.

Efficacy test 2 as shown in Figure 2 was carried out to determine the effect of 0.1% (w/w) surfactant or blended surfactants on the efficacy of glyphosate IPA at 1.0kg a.i/ha and 400l of spraying solution per hectare. The experiment was done under open conditions. The tested plants received rainfall at 2 to 14 days after the treatment.

In terms of efficacy to *Cyperus rotundus*, the results showed that all the glyphosate IPA formulations were as effective as Roundup. The effects were significantly better than that of glyphosate itself. Efficacy 2 indicated that glyphosate IPA at 1.0kg a.i/ha and 400l/ha of spraying volume containing 0.1% (w/w) surfactant(s) gave as good a control on the weed as the glyphosate IPA application in efficacy 1. The analysis of variance as shown in Table 4 indicated that

TABLE 3. ANALYSIS OF VARIANCES FOR EFFICACY 1

Rate (R) of Glyphosate (kg a.i/ha)	% Weeds killed at day 7					
	FAmE	FAE(20EO)	FAE(7EO)/ FAmE	APG (C8-C16)	Roundup	Glyphosate
1.5	65.00	70.00	65.00	65.00	65.00	51.66
2.0	70.00	70.00	70.00 (± 0.680, LSD = 1.986)	65.00	70.00	56.66
Mean	67.50	70.00	70.00 (± 0.481, LSD = 1.404)	65.00	67.50	54.16

F(T) = * * * * F(RxT) = * * * * F(R) = * * * *

Rate of Glyphosate (kg a.i/ha)	% Weeds killed at day 10					
	FAmE	FAE(20EO)	FAE(7EO)/ FAmE	APG (C8-C16)	Roundup	Glyphosate
1.5	85.00	80.00	80.00	80.00	85.00	75.00
2.0	90.00	80.00	85.00 (± 0.962, LSD = 2.808)	76.60	85.00	75.00
Mean	87.50	80.00	82.50 (± 0.680, LSD = 1.986)	78.33	85.00	75.00

F(T) = * * * * F(RxT) = * * F(R) = +

Rate of Glyphosate (kg a.i/ha)	% Weeds killed at day 14					
	FAmE	FAE(20EO)	FAE(7EO)/ FAmE	APG (C8-C16)	Roundup	Glyphosate
1.5	98.00	98.00	97.00	95.00	98.00	76.66
2.0	99.33	92.66	98.00 (± 1.049, LSD = 3.064)	96.33	97.00	81.66
Mean	98.66	95.33	97.50 (± 0.742, LSD = 2.166)	95.66	97.50	79.16

F(T) = * * * * F(RxT) = * * F(R) = not significant (NS)

* * *, * *, * and + indicate the significant levels at 0.1%, 1%, 5% and 10%

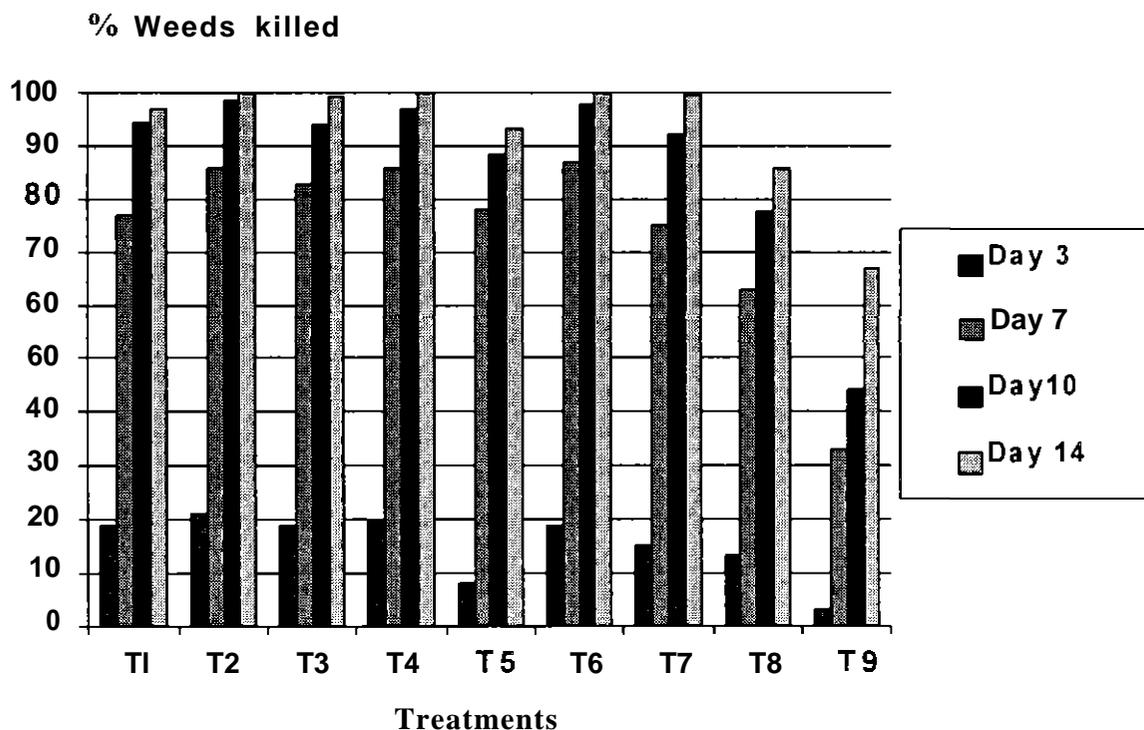


Figure 2. Bioefficacy 2: Effects of surfactants on the efficacy of glyphosate IPA on *Cyperus rotundus*

TABLE 4. ANALYSIS OF VARIANCES FOR EFFICACY 2

Treatment (R)	% Weeds killed		
	day 3	day 7	day 10
	(± 3.392 , LSD = 9.68)	(± 7.736 , LSD = 22.11)	(± 7.223 , LSD = 20.64)
FAE (20EO)	19.00	77.00	94.40
FAmE	21.03	86.03	98.40
APG(C8-C10)	19.00	83.00	94.20
APG(C8-C16)	12.00	86.00	97.00
FAE(7EO)/ FAmE	8.00	78.00	88.40
APG(C8-C10)/ FAmE	19.00	87.00	97.80
APG(C8-C16)/ FAmE	15.20	75.00	92.40
Roundup	13.40	63.00	77.60
Glyphosate	3.20	33.00	44.00
Mean	14.98	73.30	86.52
F (T) value	* *	* * *	* * *

they were significant differences between treatments at 3, 7 and 10 days after the treatment.

Grossbard and Atkinson (1985), Caseley (1996) and this study indicated that the surfactant concentrations of around 0.1% (w/w) per litre of spraying solution optimized spray drop retention and spread. However, the maximum herbicide performance is often achieved at higher concentrations. For example, 0.4% of a cationic tallow amine ethoxylate maximized the activity of glyphosate IPA salt against *Elymus repens*.

CONCLUSION

In recent years, there is a shift from the less environmentally friendly hydrocarbons and their derivatives to the more environmentally friendly oleochemical products as inert ingredients like wetting agents, adjuvants, dispersing agents, emulsifiers, solvents, carrier or diluents for pesticide formulations. These inert ingredients are more advantages over their petroleum-based counter parts in that they are renewable, biodegradable, less flammable and cause fewer medical problems and allergies to the end users.

In general, a herbicide formulation includes two main components, that is, the active ingredient(a.i) and inert ingredients. In this study, glyphosate IPA (a.i) is used as the active ingredient for killing weeds. Meanwhile, surfactants which have no herbicidal action are used as inert ingredients. Surfactant(s) lower the surface tension of the spray droplets, increase their spread over the surface of the foliage, and so enhance the uptake, translocation and efficacy of glyphosate.

Glyphosate IPA, a systemic herbicide needs two types of surfactants, i.e., a spray modifier to improve formulation wetting and spreading properties, and an activator to enhance its foliage adsorption of systemic to increase its activity.

Stability and bioefficacy tests were carried out to determine the effect of palm-based surfactants on the efficacy of glyphosate IPA controlling *Cyperus rotundus*. It was found that nine glyphosate IPA formulations containing

palm-based surfactants were stable after keeping for two months at RT and seven days at 45°C. Six of the stable formulations gave comparable results to Roundup, and were very significantly superior to glyphosate itself. The application rates were 2.0kg, 1.5kg and 1.0kg a.i/ha with 200 l and 400 l spraying volumes in the efficacy 1 and efficacy 2 respectively. The surfactant(s) concentrations in the spraying solutions were 0.18% - 0.35% (w/w) for efficacy 1 and 0.1% (w/w) for efficacy 2.

Though the results were obtained with Roundup and formulated glyphosate IPA with surfactants from palm and palm kernel oils on *Cyperus rotundus*, more research is required to test more systemic and non systemic herbicides. This will pave way for the manufacturers to overcome the constraints to get the herbicides into weeds.

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