Determination of Fluroxypyr-MHE in Clay Soil during Dry and Wet Season

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ABSTRACT

Leaching of fluroxypyr-MHE in an oil palm agroecosystem was conducted for two seasons; the dry and wet season. The oil palm estate is situated in central Selangor, Peninsular Malaysia at an altitude of 70 m to 100 m above sea level. The study plot covered six ha, and consisted of nine subplots of 0.66 ha each. The nine subplots received three treatments namely; the manufacturer's recommended dosage (T1), double the manufacturer's recommended dosage (T2), and the control (T3) with no herbicide treatment. Each treatment was done in triplicate each subplot was separated by a two meter buffer zone. The herbicide fluroxypyr-MHE (Starane® 200 EC; Dow Elanco Ltd.) was applied as an aqueous spray using a knapsack sprayer at a spraying volume of 250 L/ha. Soil samples collected at different depths (*viz*. 0-10, 10-20, 20-30, 30-40 and 40-50 cm) and at the following intervals -1 (before treatment), 1, 3, 7, 14, 30 and 60 days after treatment. Fluroxypyr-MHE was observed at all depths (0-50 cm) for the wet season when applied at the recommended and doubles the recommended dosages. Meanwhile for the dry season, fluroxypyr-MHE was detected in the soil up to 1 and 5 days after treatment when applied at the recommended and double the recommended dosages, respectively.

Keywords: Double recommended dosage, fluroxypyr, leaching, oil palm plantation, recommended dosage

ABSTRAK

Proses larut lesap fluroxypyr-MHE di dalam agroekosistem kelapa sawit telah dilaksana dan dikaji selama dua musim; musim kering dan musim hujan. Ladang kelapa sawit berkenaan terletak di kawasan tengah Selangor, Semenanjung Malaysia pada altitud 70 meter hingga 100 meter di atas aras laut. Plot kajian meliputi kawasan seluas enam hektar, dan terdiri daripada sembilan subplot yang berukuran 0.66 hektar setiap satu. Kesemua sembilan plot ini disembur dengan tiga jenis semburan iaitu pada dos yang disyorkan oleh pengeluar (T1), dos dua kali ganda yang disyorkan oleh pengeluar (T2), dan kawalan iaitu tiada semburan dilakukan (T3). Setiap perlakuan atau semburan dibuat secara triplikat, dan setiap subplot dipisahkan oleh zon tampan seluas dua meter. Herbisid fluroxypyr-MHE (Starane® 200 EC; Dow Elanco Ltd.) telah disembur sebagai semburan akueus dengan menggunakan penyembur galas pada

isipadu penyemburan 250 L/ha. Sampel tanah kemudian disampel pada kedalaman yang berbeza (0-10, 10-20, 20-30, 30-40, 40-50 cm) pada -1(sebelum perlakuan), 1, 3, 7, 14, 30 dan 60 hari selepas perlakuan. Fluroxypyr-MHE telah dikesan pada semua kedalaman (0-50 cm) pada musim hujan apabila disembur pada dos yang disyurkan pengeluar dan dos dua kali ganda yang disyurkan oleh pengeluar. Manakala pada musim kering pula, fluroxypyr-MHE dikesan di dalam tanah pada 1 hingga 5 hari selepas perlakuan; apabila disembur pada dos yang disyurkan pengeluar dan dos dua kali ganda yang disyurkan oleh pengeluar, secara berturutan.

Kata kunci: Dos dua kali ganda yang disyurkan, fluroxypyr, larut lesap, ladang kelapa sawit, dos yang disyorkan

INTRODUCTION

The most widely used herbicides in Malaysia are glyphosate, paraquat, 2, 4-D, fluroxypyr, metsulfuron methyl, triclopyr and glufosinate ammonium (Chung *et al.,* 2000; MADI, 2003/2004). These herbicides are among the most common herbicides currently used in most plantations in Malaysia, including oil palm plantations.

Fluroxypyr-MHE is a common name for 4-amino-3, 5-dichloro-6-fluoro-2-pyridyloxy acetic acid. In the formulated product, fluroxypyr is present as an ester. The ester and the acid forms display herbicidal activity.

The movement of the pesticide from the application site may affect its bioefficacy, availability, degradation, microbial toxicity, phytotoxicity and leaching pattern. Some pesticide metabolites have been associated with insecticidal activity (Chapman and Harris, 1980), enhanced microbial degradation of the parent compound, phytotoxicity (Bergstrom *et al.,* 1990) and groundwater contamination (Cohen *et al.,* 1984). Although the mobility of herbicides such as metsulfuron-methyl and its metabolites in the soil has been studied in detail, little is known about other groups of chemicals such as fluroxypyr. Environmental factors such as soil water content, soil temperature, rainfall and soil texture may also affect the mobility of pesticides in soil profiles (Ismail and Teoh, 1994).

Heavy usage of pesticides for agricultural activities may cause adverse effects to the environment and consequently human health. The leaching of pesticides into groundwater is a major environmental concern because it affects the quality of underground water (Lehmann *et al.,* 1993). The number of different pesticides in ground and surface water has been found to be increasing steadily (Lehmann and Miller, 1989). Therefore, an understanding of adsorption, desorption, and mobility of pesticides in soil need immediate attention. It was reported that when fluroxypyr was used for weed control in oil palm plantations, no residue was detected in crude palm oil and crude palm kernel oil irrespective of the sampling interval and the dosage applied at the recommended or double the manufacturer's recommended dosage (Halimah *et al*., 2008). Fluroxypyr in soil was easily leached into the lower profile of the soil, probably because of the high amount of rainfall; however the fluroxypyr has a short persistence span in soil (Halimah *et al.,* 2005). Therefore, this paper is aimed at investigating the fate of the primary compound of fluroxypyr-MHE in the oil palm agroecosystem during the wet and dry season.

MATERIALS AND METHODS

Experimental Design

This study was conducted at the Kuala Lumpur International Airport (KLIA) Oil Palm Plantation, owned by the Malaysian Agricultural Horticultural Sdn. Bhd. (MAAH). This oil palm estate is situated in the central portion of the state of Selangor in Peninsular Malaysia; at an altitude between 70 m to 100 m above sea level. The study plot covered 6 ha, and consisted of 9 subplots of 0.66 ha each. The 9 subplots received three treatments, namely; the manufacturer's recommended dosage (T1), double the manufacturer's recommended dosage (T2) and the control (T3) with no herbicide treatment. Each treatment was done in triplicate and each subplot was separated by a 2 m buffer zone. The palm trees used for this study were two and a half years old.

The study plot was situated on hilly land of slope 45 ^{o}C with soil predominantly clayey in texture. The physico-chemical properties of the soil were determined. The soil had a high content of clay (52.2%), a CEC (cation exchange capacity) of 6.9%, 15% coarse sand, 27% fine sand and 5.8% silt. The soil was classified as clay soil. All soil data reported are on a dry weight basis.

Fluroxypyr-MHE (Starane® 200 EC; Dow Elanco Ltd.) was applied as an aqueous spray, by using a knapsack sprayer (nozel 5/64) at a spraying volume of 250 L/ha. The herbicide was applied at 72.05 g *a.i.*/ha (T1) and 144.10 g *a.i.*/ha (T2), which represented the manufacturer's recommended and double the manufacturer's recommended dosage, respectively. The control plot (T3) was not treated with herbicide.

Determination of Fluroxypyr-MHE in Soil

Approximately 5 ± 0.002 g of fluroxypyr-MHE soil samples were placed in 250 mL conical flasks. The soil was spiked with fluroxypyr-MHE standard solution at 0.2 μg/mL to 2.5 μg/mL, equivalent to 0.02 to 0.25 μg/g soil, and the contents were mixed on a vortex mixer. The mixture in each conical flask was allowed to stand for 15 minutes in order to obtain a homogenous distribution. Then, 20 mL of acetone: water (95:5) was added to the conical flask and it was again mixed thoroughly for 30 s on the vortex mixer. The conical flask was placed in an ultrasonic bath for 20 min after which the contents were transferred to a test tube and centrifuged at high speed (3500 rpm) at 26 °C for 10 min. Ten mL of the top layer (acetone + water) were then transferred to a graduated micro vial using a Pasteur pipette. The solution was evaporated to dryness using the N-evaporator and the residue was re-dissolved with 1 mL acetonitrile and then mixed using an ultrasonic bath for 3 s prior to injection into the HPLC–DAD, set at a wavelength of 210 μm, for quantification. The extraction of fluroxypyr-MHE from the soil spiked with the standard solution was carried out with each concentration, injected thrice.

RESULTS AND DISCUSSION

Weather Conditions for Fluroxypyr-MHE Field Trial

Figure 1 shows the average daily rainfall at the oil palm plantation during the wet season. During the study period, the highest precipitation at the experimental site was 169 mm recorded from 1st to 27th November 2001. The area received higher rainfall in November 2001 than in January and February 2002, where rainfall amounted to 31.7 and 44.2 mm, respectively. The number of rainy days in November and December 2001 were 18 and 14 days, while the rainy days in January and February

2002 were 8 and 2 days, respectively. However, the precipitation showed an uneven distribution. The maximum evaporation was about 9 mm/day. The air temperature ranged from 36 °C during the day to 25 °C at night. Figures 2 and 3 show the rate of evaporation and maximum and minimum air temperature (ºC) at the KLIA estate during the study period.

The daily rainfall, rate of evaporation and the maximum/minimum air temperature (ºC) for the dry season are shown in Figures 4, 5 and 6, respectively. The dry season for this study was from 15th June 2002 to 30th September 2002. During this study period, the amount of rainfall received from the first day of spraying to day 21 (after spraying), was 10.4 mm with an average of 0.49 mm per day. The amount of rainfall during the dry season was 93% lower compared to the wet season. The rates of evaporation and maximum/minimum air temperatures were almost similar for both seasons.

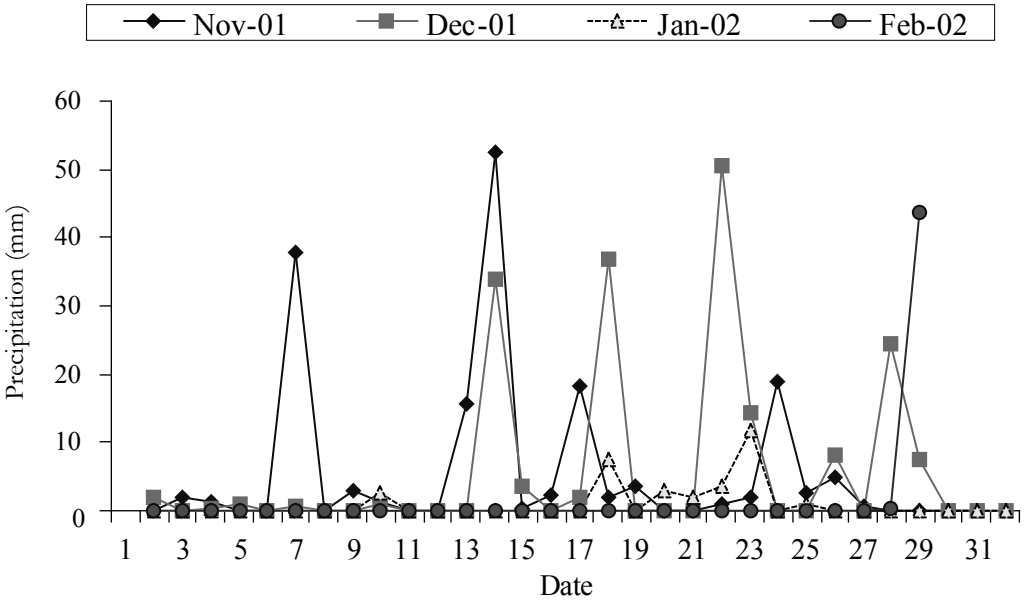

Fig. 1. Daily precipitation at KLIA, Sepang, Selangor during the wet season.

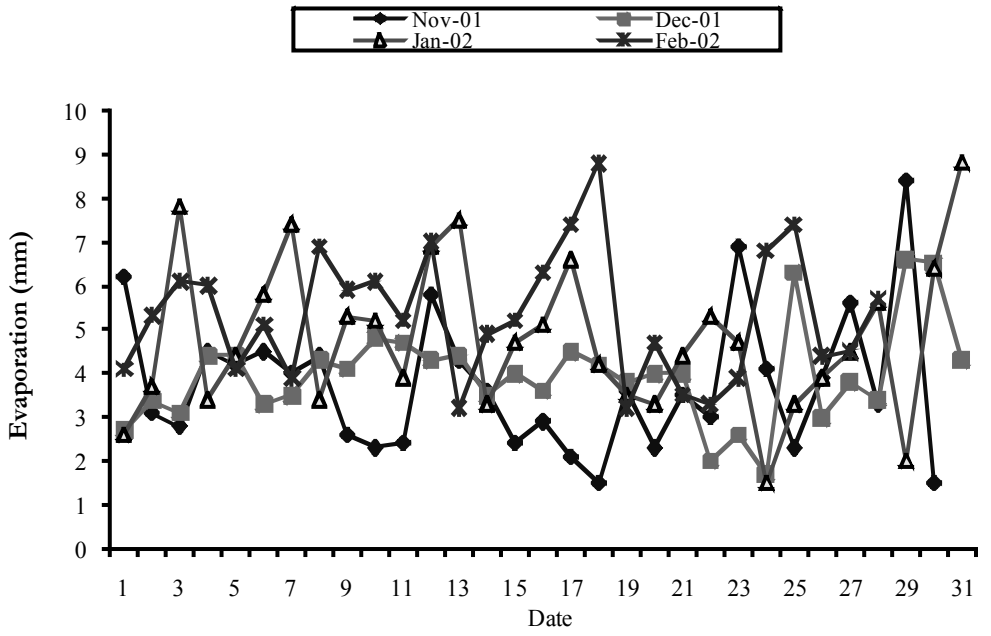

Fig. 2. Water evaporation rate at KLIA, Sepang, Selangor during the wet season.

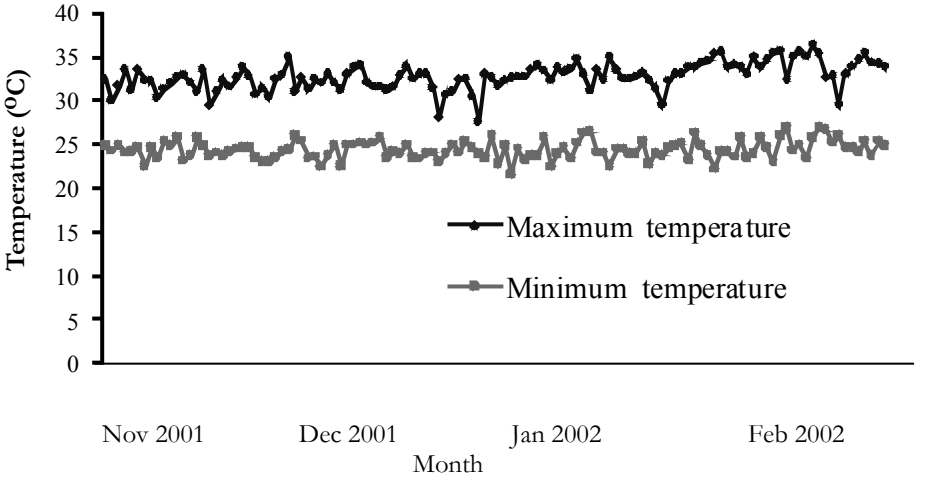

Fig. 3. Variation of the maximum and minimum air temperature (°C) at KLIA, Sepang, Selangor during the wet season.

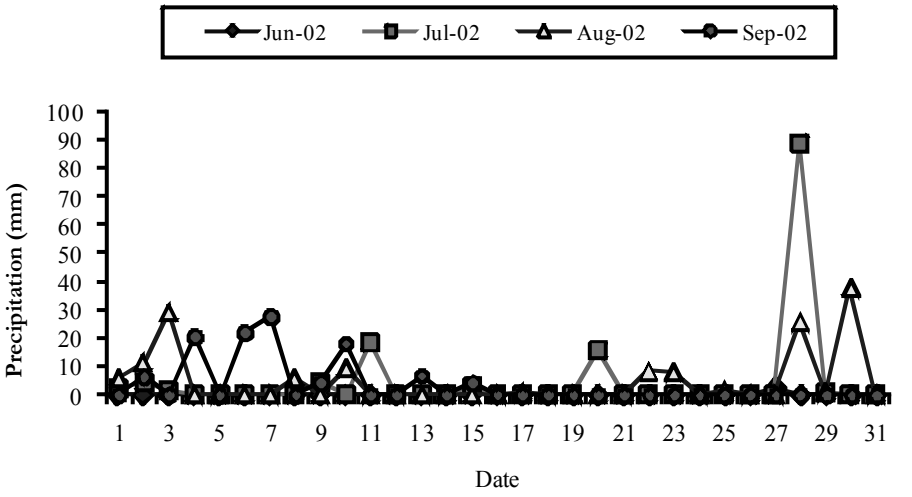

Fig. 4. Daily rainfall at KLIA, Sepang, Selangor during the dry season in 2002.

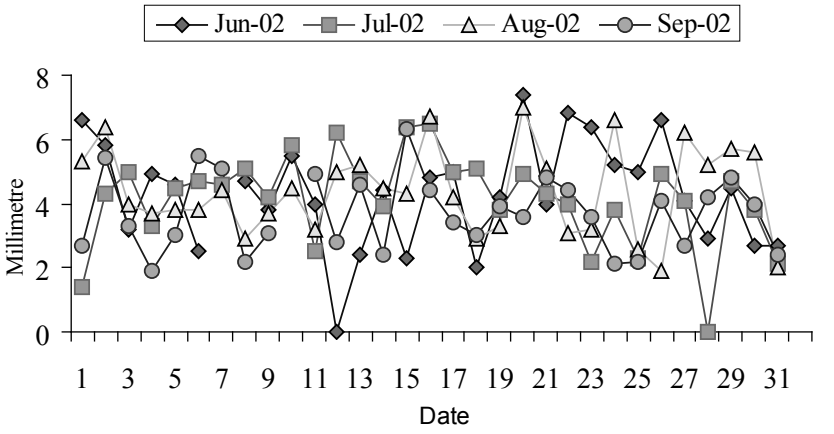

Fig. 5. Daily water evaporation at KLIA, Sepang, Selangor during the dry season in 2002.

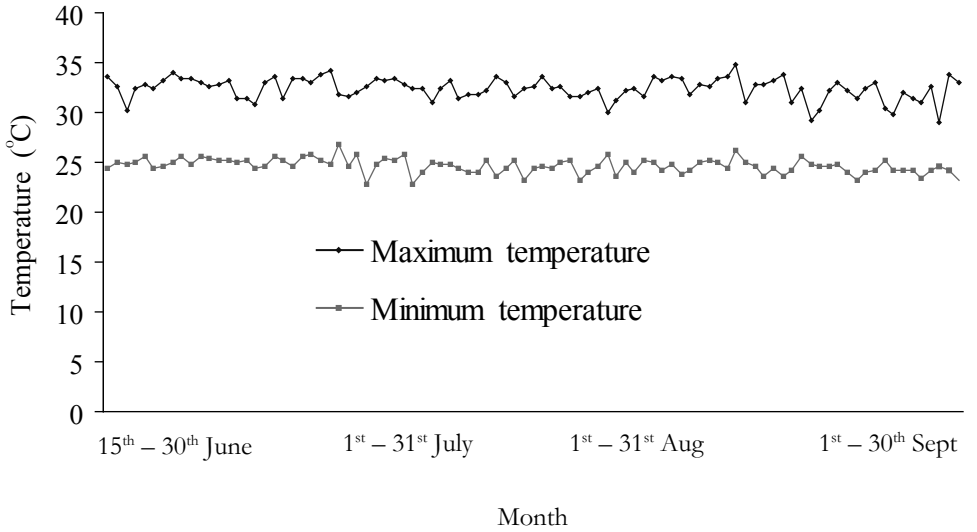

Fig. 6. Variation in the maximum and minimum air temperature (°C) during the dry season in 2002.

Fate of Fluroxypyr-MHE in the Soil (Wet Season)

Table 1 shows the amount of fluroxypyr-MHE residue in the soil (μg/kg dry soil) when sprayed at the recommended dosage during the wet season. At the recommended dosage, the total amount of fluroxypyr-MHE found on 1 DAT in the soil profile (0-50 cm) was 200 μg/kg with the highest amount deposited in the top layer of 0-10 cm, followed by the next highest deposit in the second layer (10-20 cm). The total amount of fluroxypyr-MHE was not detected at day 5 after treatment. At double the recommended dosage, fluroxypyr-MHE was detected at all depths (0-50 cm) on day 1, when the field was sprayed with herbicide as indicated in Table 1. Most fluroxypyr-MHE was deposited at the depth of 0-10 cm (210 μg/kg) and the concentration decreased with increasing depth. The amount of fluroxypyr deposited in the soil ranged from 10 to 210 μg/kg, with the standard deviations of 1.3 to 4.2, respectively. The total amount of fluroxypyr in the soil at the depth of 0-50 cm was 430 μg/kg. Eventually on day 5, fluroxypyr was not detected at all depths.

Table 1. Fluroxypyr–MHE residue in the soil at different soil depths when applied at the manufacturer's recommended and double the manufacturer's recommended dosage during the wet season.

[a]DAT	Depth (cm)	Concentration of fluroxypyr -MHE (μg/kg)	
		Manufacturer' s recommended dosage	Double the manufacturer's recommended dosage
1	0-10	90 ± 2.5	210 ± 4.2
	10-20	60 ± 1.0	140 ± 3.4
	20-30	20 ± 1.9	60 ± 2.9
	30-40	10 ± 1.2	10 ± 1.3
	40-50	20 ± 1.9	10 ± 1.5
5	0-10	ND	ND
	10-20	ND	ND
	20-30	ND	ND
	30-40	ND	ND
	40-50	ND	ND

[a]DAT: Day after treatment ND: Not Detected = < 1 μg/kg

Fate of Fluroxypyr-MHE in the Soil (Dry Season)

Table 2 shows fluroxypyr–MHE residue in the soil at the recommended and double the recommended dosage during the dry season. Fluroxypyr-MHE residue in the soil was found at all soil depths (0-50 cm) for both dosages. Results showed that most of the fluroxypyr-MHE was deposited in the uppermost layer followed by the second layer, and it appeared that the applied herbicide moved downwards through the soil profile from 0-50 cm depth.

On 1 DAT, the amount of fluroxypyr–MHE ranged from 140 to 440 μg/kg at all soil depths, when the plot was sprayed at the recommended dosage (Table 2). It was also found that on the first day after treatment, fluroxypyr-MHE was deposited mostly at the depths of 0-10 cm and it decreased gradually with increasing depth. However, this compound did not persist long in the environment and it was not detected on day 5 after treatment.

When the plot was sprayed at double the manufacturer's recommended dosage (1 DAT), the amount of fluroxypyr-MHE residue in the soil ranged from 260 to 870 μg/kg. However, fluroxypyr-MHE concentrations in the soil was reduced to very low levels on day 5 after treatment.

Eventually, on day 7 after treatment, fluroxypyr-MHE was reduced to non-detectable levels. It was observed that a higher concentration of fluroxypyr-MHE was found in the soil when the herbicide spraying was carried out during the dry season as compared to the wet season. The reason is attributed to fluroxypyr-MHE surface run-off and loss to the rivers or streams around the trial plot, because of higher rainfall during the wet season. Fluroxypyr-MHE was not detected on the fifth and seventh day after treatment, when the herbicide was sprayed at the recommended dosage. Results showed that at the recommended dosage, the total amount of fluroxypyr-MHE deposited in the soil was about half of that deposited at double the recommended dosage.

Table 2. Concentration of fluroxypyr-MHE residue at different soil depths when applied at the manufacturer's recommended and double the manufacturer's recommended dosage during the dry season.

[a]DAT	Depth (cm)	Concentration of fluroxypyr -MHE (μg/kg)	
		Manufacturer's recommended dosage	Double the manufacturer's recommended dosage
1	0-10	440 ± 10	870 ± 12.0
	10-20	410 ± 9.9	810 ± 13.0
	20-30	210 ± 7.0	370 ± 8.7
	30-40	140 ± 6.0	320 ± 4.5
	40-50	150 ± 5.0	260 ± 6.5
5	0-10	ND	35 ± 4.2
	10-20	ND	35 ± 4.3
	20-30	ND	30 ± 3.5
	30-40	ND	39 ± 7.8
	40-50	ND	74 ± 10.3
7	0-10	ND	ND
	10-20	ND	ND
	20-30	ND	ND
	30-40	ND	ND
	40-50	ND	ND

[a]DAT: Day after treatment ND: Not Detected = < 1 μg/kg

CONCLUSION

The field study indicated that fluroxypyr-MHE leached down to 50 cm depth when the plots were treated with the recommended and double the recommended dosages. During the dry season at the recommended dosage, Fluroxypyr-MHE was found in the soil profile on 1 DAT, meanwhile at double the recommended dosage fluroxypyr-MHE was found on 1 and 5 DAT. However, during the dry season fluroxypyr-MHE was detected in the soil as residue on 1 DAT and 5 DAT at the recommended and double the recommended dosage, respectively. The leaching of up to 50 cm in soil depth is considered as liable to occur as sub-surface water contamination, but fluroxypyr-MHE persists for a short period only.

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