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J. Environ. Biol.
33, 573-577 (2012)
ISSN: 0254-8704
CODEN: JEBIDP

Adsorption, desorption and mobility of metsulfuron-methyl in soils of the oil palm agroecosystem in Malaysia

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Publication Data

Paper received:
26 September 2010

Revised received:
12 July 2011

Accepted:
06 August 2011

Abstract

Laboratory experiments were conducted to evaluate adsorption, desorption and mobility of metsulfuron-methyl in soils of the oil palm agroecosystem consisting of the Bernam, Selangor, Rengam and Bongor soil series. The lowest adsorption of metsulfuron-methyl occurred in the Bongor soil (0.366 ml g^{-1}), and the highest in the Bernam soil (2.837 ml g^{-1}). The K_{fads} (Freundlich) values of metsulfuron-methyl were 0.366, 0.560, 1.570 and 2.837 ml g^{-1} in Bongor, Rengam, Selangor and Bernam soil, respectively. The highest K_{fdes} value of metsulfuron-methyl, observed in the Bernam soil, was 2.563 indicating low desorption 0.280 (relatively strong retention). In contrast, the lowest K_{fdes} value of 0.564 was observed for the Bongor soil, which had the lowest organic matter (1.43%) and clay content (13.2%). Soil organic matter and clay content were the main factors affecting the adsorption of metsulfuron-methyl. The results of the soil column leaching studies suggested that metsulfuron-methyl has a moderate potential for mobility in the Bernam and Bongor soil series with 19.3% and 39%, respectively for rainfall at 200 mm. However, since metsulfuron-methyl is applied at a very low rate (the maximum field application rate used was 30 g ha^{-1}) and is susceptible to biodegradation, the potential for ground water contamination is low.

Key words

Adsorption, Desorption, Metsulfuron-methyl, Mobility, Leaching

Introduction

Metsulfuron-methyl is a sulfonylurea herbicide, widely used at low application rates for post emergence broadleaf weed control in oil palm plantations. It has good selectivity to oil palm and is very effective against a wide range of broadleaf weeds at application rates of 15 to 30 g ha^{-1} (Khairuddin and Teoh, 1992; Chung, 1997). Metsulfuron-methyl has become popular because of it low application rates, low mammalian toxicity and minimal impact on the environment (Ye *et al.,* 2003; Li *et al.,* 2005).

Adsorption of pesticides to soils is an important process that influences their migratory behavior in various compartments of the environment. Sarmah *et al.* (1998) reported that sorption of metsulfuron-methyl is pH-dependent, as it is strongly negatively correlated with pH. In acidic soils, however, sorption of metsulfuron-methyl is greatly influenced by soil temperature, clay content, and, particularly, organic matter content. Being acidic in nature with a pKa of 3.3 (Beyer *et al.,* 1988), the metsulfuron-methyl molecule is found predominantly in the ionized form and can move to a considerable depth in the soil profile by leaching. Movement of sulfonylureas in the soil is largely influenced by organic matter content and soil pH (Abdullah *et al.,* 2001a,b; Mohd Tahir *et al.,* 2005, Mehdi *et al.*, 2011). The potential for leaching of chlorsulfuron in coarse-textured soils (neutral to slightly acidic) with low organic matter content was illustrated in a laboratory study, using intact soil cores (Beckie and McKercher, 1990). The soil type used had a pH range of 5.5-7.0 and organic matter content of 1.8-5.1 and 1.0-2.1% in the 0-10 and 10-30 cm depths, respectively.

Information on the behaviour of metsulfuron-methyl in Malaysian agricultural soils is limited, especially for the oil palm agroecosystem. Only a few studies on persistence and bioactivity of metsulfuron-methyl in agricultural soils have been reported (Mersie and Foy, 1986; Ismail and Kalithasan, 1997; Rahman *et al.,* 1997). Data generated from research on these aspects is of major importance in determining the environmental fate of this herbicide and the potential of groundwater contamination in the Malaysian agro-environments.

The objective of the present study was to determine the adsorption, desorption and mobility of metsulfuron-methyl in four major types of oil palm soils, using chemical analyses.

Materials and Methods

Samples of Bernam and Selangor series soil types were collected from an oil palm estate at Kuala Selangor, Selangor and these two soil types were selected to represent coastal soils. The soil samples representing inland soils were collected from Labu, Negeri Sembilan and they belonged to the Rengam and Bongor series. The soil samples were collected from 0-10 cm depth, air dried (at room temperature 27°C for 72 hr) and passed through a 2 mm sieve prior to use. The physico-chemical properties of the soils were analyzed (Miller and Donahue, 1990).

The commercial formulation of metsulfuron-methyl (Ally, 20% *a.i.*) used in the study was obtained from the manufacturer. (E.I. Du Pont de Nemours and Company). The radiolabelled form [*triazine*-2-^{14}C] of metsulfuron-methyl with a specific activity of 1.85 MBq mg^{-1} (49.87 Ci mg^{-1}) was synthesized at DuPont New England Nuclear Research Products (Boston, MA, USA) and was used to spike the commercial product.

Adsorption-desorption studies: Two grams of soil were weighed separately into 50 ml conical centrifuge bottles, into which was added 10 ml of 0.01 M $CaCl_2$, and aqueous solutions of ^{14}C labelled metsulfuron-methyl (0.05654 mCi) at concentrations of 0.1, 0.5, 1.0 and 5.0 mg l^{-1}. The samples were agitated at 160 rpm (25°C) using an orbital shaker for 24 hr, a period that preliminary studies showed was sufficient to attain pseudo-equilibrium. Subsequently, the samples were centrifuged at 3000 rpm for 15 min. A 1 ml aliquot was removed from the supernatant solution in each tube, placed in 10 ml of the scintillation fluid (NBCS104, Amersham), and measured using a liquid scintillation counter (LSC). The resulting radioactivity in the solution was compared with a 1 ml aliquot of the appropriate concentration standard, which represented the amount of added radioactivity. Differences between the amounts of ^{14}C found in the standard solutions and the supernatant of the samples were considered to be the amounts adsorbed. The herbicide sorption isotherm (by log transformation) was plotted and the Freundlich equation (Ismail *et al.,* 2009) was used to calculate the adsorption coefficient (K_f).

Desorption was determined (Ismail *et al.,* 2009) on the same samples used for adsorption. After the supernatant was removed, 10 ml of fresh 0.01 M $CaCl_2$ was added into the same centrifuge bottle. The mixture was then agitated and centrifuged as described above. A 1ml aliquot was removed and measured at the end of a 24 h equilibration period. The process was repeated four times. After the 4th desorption step, the soil (150 g total soil from the 3 replicates) was air-dried (at 25°C, 48 hr) and then homogenized manually using a mortar and pestle. An aliquot (0.3 g) was combusted using the biological oxidizer and the carbon dioxide released was trapped and radioassayed using the LSC.

Column leaching studies: Three glass columns (15 cm length and 25 cm diameter) were assembled end to end using clamps to produce a system with a combined length of 45 cm (Mehdi *et al.,* 2009). Each of two such columns was packed manually with the Bongor and Bernam soil using light pressure to ensure uniform density. A flask was placed at the bottom of each column to collect the leachate. The columns were shielded by wrapping with black polyethylene sheets and arranged in the greenhouse under natural conditions.

The radiolabelled metsulfuron-methyl (18.5 kBq) was supplemented with non-radiolabelled material to obtain the rate of 30 g ha^{-1} (0.0176 ml total volume) by application of the herbicide dropwise in a grid pattern to the soil surface of the column, using a pipette. The columns were pre-moistened (24 hr) prior to the application, in order to compact and stabilize the soils. After application, distilled water was immediately and continuously passed through the columns to simulate natural rainfall. The experiment was conducted at room temperature of 27°C. Rain events of 100 and 200 mm (equivalent to 49.05 and 98.1 ml, respectively) were simulated at the rate of 0.05 and 0.1 ml min^{-1}. The intensity of the simulated rainfall was adjusted to fall over a period of 48 hr. The column experiment was carried out in triplicate. Soil samples were collected from 0-10, 10-20, 20-30 and 30-40 cm segments, air-dried, homogenized and three aliquots (0.3 g) were radioassayed using the biological oxidizer as previously described.

In additional to the direct leaching studies, leaching studies with "aged" soils were also carried out following the method described by Ismail *et al.* (2009). Twenty grams [equivalent to (5 × 25 cm) diameter soil section] of Bongor and Bernam soil were each treated with radiolabelled metsulfuron-methyl solution (18.5 kBq), supplemented with non-labelled material to achieve the application rate of 30 g ha^{-1} (0.0176 ml total volume). The samples were placed in the greenhouse for 30 days and watered when necessary to maintain 75% of the maximum moisture holding capacity (46.5% at 0.1 bar moisture content).

After 30 days, the aged and the treated soils (20 g each) were transferred to the top of a corresponding pre-moistened soil column to give the final depth of 45 cm. The same rain events were simulated. Samples were collected from 0-10, 10-20, 20-30 and 30-40 cm segments, air-dried, homogenized and three aliquots (0.3 g) were radioassayed using the biological oxidizer as previously described.

Data were subjected to analyses of variance and means were compared using the LSD test at the 5% level of significance (Halimah *et al.,* 2011).

Results and Discussion

Table 1 shows the physico-chemical properties of the four soils types studied. Two soil types namely the Bernam and Selangor series were classified as a loamy clay soils while the Rengam and Bongor series were classified as sandy loam soils. The Bernam soil series contained the highest organic matter content (4.03%), followed by Selangor (2.89%), and Rengam series (1.66%) soils. The Bongor series contained the lowest organic matter content (1.43%) compared to the other three soils types. A similar pattern was observed for Bongor which contained less clay and silt. In contast, the Bongor soil contained higher percentage sand and bulk density in the hierarchial order of Bongor>Rengam> Selangor>Bernam. In general, the pH was not very different for the four soil types.

Adsorption and desorption studies: The highest adsorption of metsulfuron-methyl was observed in the Bernam soil followed by the Selangor, Rengam and the Bongor soil, respectively. In general the adsorption percentage was lower at the higher concentrations of metsulfuron-methyl (Table 2) and this is reflected in the 1/n values, which are less than 1.0 (Table 3), illustrating that adsorption is non-linear with respect to concentration (Rhodes *et al.,* 1970). This may be due to the possibility that the herbicide molecules occupy most of the adsorption sites at the higher concentrations. Similar observations have also been reported by other researchers (Grover, 1973; Ismail and Chong, 2003).

The K_f values of metsulfuron-methyl was positively correlated to the cation exchange capacity (CEC), organic matter, clay and silt content but negatively correlated to the fine and coarse sand content (Table 4). The highest correlation coefficients were obtained with organic matter (OM) and clay, with R^2 values of 0.994 and 0.983, respectively. The correlation coefficients of K_{fdes} for the various physical properties of the soil were lower than the corresponding K_{fads} values, but followed the same trend. Even though the soils used in the study had a narrow range of soil pH (4.02-4.71), a negative correlation ($R^2 = 0.661$) between soil pH and adsorption of metsulfuron-methyl was observed. This study suggested that organic matter is more important than any other soil fraction for adsorption of metsulfuron-methyl. Walker *et al*. (1989) also reported that adsorption of metsulfuron-methyl was significantly correlated only with soil organic matter. Perhaps, the pH range in these soils is low enough so that the effective pH at the clay surface approached the pK_a value for metsulfuron methyl, which could result in the production of higher number of neutrally charged molecules and consequently lead to higher adsorption.

The K_{fads} values observed for the coastal soils (Bernam and Selangor soil) suggested that the higher adsorption of metsulfuron-methyl to soil could be attributed to the high OM and

Table - 1: Physico-chemical properties of four Malaysian agricultural soils

Soil (Series)	Soil type	C.E.C (meq 100 g^{-1})	Mechanical analysis (%)				OM (%)	OC (%)	pH	Bulk density (g cm^{-3})
			Clay	Coarse sand	Fine sand	Silt				
Bernam	Loamy clay	26.89	40.5	9.8	18.2	31.5	4.03	2.34	4.13	0.92
Selangor	Loamy clay	21.58	30.0	16.3	23.2	30.5	2.89	1.68	4.02	0.97
Rengam	Sandy loam	6.14	16.3	28.7	43.5	11.5	1.66	0.96	4.55	1.06
Bongor	Sandy loam	5.80	13.2	32.2	45.3	9.3	1.43	0.83	4.71	1.23

OM = Organic matter, OC = organic carbon - calculated as OM (%) × 0.58, CEC = Cation exchange capacity

Table - 2: Percentage of metsulfuron-methyl adsorbed onto four Malaysian agricultural soils

Concentration of metsulfuron-methyl (mgl^{-1})	Adsorption (% of initial concentration)			
	Bernam soil	Selangor soil	Rengam soil	Bongor soil
0.1	76 ± 0.4	63 ± 0.8	38 ± 0.5	29 ± 0.7
0.5	74 0.4	59 ± 0.5	36 ± 0.5	26 ± 0.4
1.0	63 ± 0.3	51 ± 0.5	28 ± 0.5	20 ± 0.5
5.0	57 ± 0.2	31 ± 0.8	21 ± 0.4	16 ± 0.3

Values are mean triplicate ± SD

Table - 3: Freundlich adsorption and desorption coefficient of metsulfuron-methyl in four Malaysian agricultural soils

Parameters	Bernam soil	Selangor soil	Rengam soil	Bongor soil
K_{fads}	2.837	1.570	0.560	0.366
$1/n_{ads}$	0.873	0.906	0.873	0.873
K_{fdes}	2.563	1.411	1.069	0.564
$1/n_{des}$	0.280	0.437	0.332	0.521
K_{fads}/ K_{fdes}	1.107	1.113	0.524	0.702

clay content of the soils. Consequently, the lower K_{fads} observed for the inland soils (Rengam and Bongor Series soils) could be attributed to the low OM and clay content (Table 3). Sarmah *et al.* (1998) reported that in acid soils, sorption of metsulfuron-methyl was strongly influenced by soil temperature, clay content, and, particularly, organic matter content. The coastal soils have lower pH compared to the inland soils and the sorption coefficient values generally decrease as the soil pH increases (Cranmer *et al.,* 1999).

Table 5 shows the percentage desorption of metsulfuron-methyl in the four soils types studied. The initially adsorbed concentration that desorbed from the coastal soils was slightly less compared to that observed for the inland soils. The results show that metsulfuron-methyl adsorbed to the soils and was not very easily desorbed since the highest desorption only occurred in the fourth and last step (with the exception of the Bongor soil). The higher K_{fdes} value for the Bernam soil indicated low desorption. In contrast, the lowest K_{fdes} value was observed for the Bongor soil. K_{fdes} was positively correlated to the CEC, organic matter and clay content, but negatively correlated to pH plus fine and coarse sand content (Table 4). The $1/n_{des}$ values were less than 1 for all the soils tested (Table 3), indicating that desorption percentages were positively correlated to the total amount adsorbed. The K_{fads}/K_{fdes} ratios were 1.107, 1.113, 0.524 and 0.702 in the Bernam, Selangor, Rengam and Bongor soil, respectively. The K_{fads}/K_{fdes} ratio for metsulfuron-methyl in the coastal soils demonstrated that they showed a stronger tendency for adsorption and low potential for leaching. The higher K_{ads}/K_{des} ratio for metsulfuron-methyl shown in the peat soil is further evidence of its stronger tendency for adsorption in soils with higher organic matter content, and low potential for leaching (Ismail and Chong, 2003). This implies that metsulfuron-methyl has the low potential to leach in soils with high organic matter content.

Table - 4: Correlation coefficients (R^2) of Freundlich adsorption coefficients for soil properties

Soil properties	Adsorption-desorption coefficient			
	K_{fads}	$1/n_{ads}$	K_{fdes}	$1/n_{des}$
CEC	0.925	0.161	0.800	-0.236
Clay	0.983	0.069	0.916	-0.377
Coarse sand	-0.950	-0.120	-0.876	0.357
Fine sand	-0.897	-0.202	-0.776	0.236
Silt	0.819	0.300	0.690	-0.191
OM	0.994	0.046	0.931	-0.386
pH	-0.661	-0.451	-0.556	0.172

Negative and positive R^2 values indicate negative and positive orelations, respectively

Column leaching studies: Table 6 shows the mobility of metsulfuron-methyl on freshly treated and aged Bernam and Bongor soils under simulated rainfall of 100 and 200 mm. The metsulfuron-methyl residue was leached down the column in both the Bernam and Bongor soil columns. The residue could be detected in the 30-40 cm soil profiles after 48 hr of simulation except in the aged Bernam soil column. In general, the amount of the residue leached was not much affected by the volume of the simulated rainfall in the Bernam soil column, but the difference was observed in the Bongor soil columns especially those with fresh soil. The residues of metsulfuron-methyl in freshly treated Bongor soil leached about

Table - 5: Percentage of metsulfuron-methyl desorbed from four Malaysian agricultural soils

Soil series	Desorption (% of initial concentration)				
	1st desorption	2nd desorption	3rd desorption	4th desorption	Total
Bernam	10.829 ± 0.003	15.489 ± 0.023	16.318 ± 0.033	24.432 ± 0.043	67.068 ± 0.926
Selangor	8.578 ± 0.762	17.087 ± 1.054	18.316 ± 0.043	25.564 ± 0.730	69.545 ± 1.158
Rengam	6.888 ± 0.065	9.581 ± 0.076	24.924 ± 0.045	38.315 ± 0.304	79.708 ± 1.213
Bongor	16.178 ± 0.871	11.818 ± 0.452	27.628 ± 0.378	25.877 ± 0.982	81.501 ± 1.062

Values are mean of triplicate ± SD

Table - 6: Distribution of metsulfuron-methyl in fresh and aged Bongor and Bernam soil columns at different level of rainfall simulation

Treatment	Simulated rainfall (mm)	Applied radioactivity (%)											
		Bernam soil						Bongor soil					
		0-10 cm	10-20 cm	20-30 cm	30-40 cm	Total retained	Total leached	0-10 cm	10-20 cm	20-30 cm	30-40 cm	Total retained	Total leached
Fresh	100	33.67 (±7.54)	27.37 (±5.32)	25.31 (±2.57)	3.01 (±0.67)	89.36 (±15.67)	10.64	18.34 (±3.45)	22.73 (±6.31)	21.56 (±4.56)	12.72 (±3.21)	75.35 (±12.54)	24.65
	200	28.35 (±6.12)	21.67 (±5.54)	19.98 (±.38)	10.45 (±2.87)	80.63 (±13.32)	19.37	10.34 (±1.26)	16.54 (±2.59	17.78 (±3.81)	15.46 (±4.13)	60.12 (±9.38)	39.88
Aged	100	38.40 (±6.51)	28.60 (±4.55)	17.29 (±2.46)	0.00 (±0.00)	84.24 (±10.35)	15.76	23.32 (±3.32)	21.21 (±3.56)	20.27 (±4.02)	15.51 (±4.15)	80.31 (±8.76)	19.69
	200	32.20 (±5.89)	25.30 (±5.23)	24.78 (±3.31)	3.56 (±1.18)	85.74 (±5.43)	14.26	13.45 (±2.06)	24.68 (±4.50)	23.67 (±4.41)	16.02 (±2.56)	77.82 (±6.34)	22.18

24.65 and 39.88% of the applied radioactive pesticide for rainfall simulations of 100 and 200 mm, respectively. However in the column, with fresh Bernam soil the total amount of the residue found in the leachate was about 10.64 and 19.37% with 100 and 200 mm of simulated rainfall. The amount of the residues retained in the upper layer (0-10 cm profile) was higher in the Bernam soil compared to Bongor soil. This may due to higher organic content in the Bernam soil than in Bongor soil series. The organic matter has been reported to adsorb more pesticides residues (Mehdi *et al.,* 2009; Mehdi *et al.,* 2011). The residues of metsulfuron-methyl in the aged soils were generally higher than those in the freshly treated soils (except in the case of the Bernam soil) for 100 mm of simulated rainfall. It was evident from the study that metsulfuron-methyl residue levels at various depths generally depended on the amount of simulated rainfall and the soil type. The treatment on the aged soil would likely have resulted in degradation of a significant portion of the metsulfuron methyl, leaving degradation products with different adsorption characteristics from the parent molecule. This may have contributed to the higher amount of leaching of the applied radioactive pesticide from the aged versus freshly treated Bernam soil (15.76 vs. 10.64%, respectively). Residues of metsulfuron-methyl in freshly treated Bernam soil for both levels of simulated rainfall were consistently higher in the top 10 cm. This showed that the Bernam soil had better ability to retain the residues of metsulfuron-methyl compared to the Bongor soil and reduce the amount from leaching deeper into the soil. Although residues leached to the deeper segments in the Bernam soil, most of the residues of metsulfuron-methyl were retained at the 10-20 cm and 20-30 cm level in the Bongor soil. The increased volume of the simulated rainfall was sufficient to cause additional movement of metsulfuron-methyl residues to the lower depths except in the treatment with aged Bernam soil. The increase in adsorption of mesulfuron methyl due to the higher organic matter and clay content in the Bernam soil may have been responsible for the increased retention of metsulfuron-methyl in that particular soil. Studies by Ismail and Chong (2003) clearly indicated that there was a reduction in extent of leaching of metsulfuron-methyl with increasing Organic Matter content. In general, the residues of metsulfuron-methyl retained in the aged soils at all soil depths were higher that those in the freshly treated soil samples.

In conclusion, the soil type and amount of rainfall plays an important role in the leaching process of metsulfuron methyl in the soil ecosystem. This reflects the adsorption and desorption process whereby the molecule of metsulfuron-methyl gets strongly adsorbed onto soil particles with high organic matter and thus causes a slow down in the desorption process. At lower concentrations of metsulfuron-methyl, the adsorption was much stronger as the molecules get attached to the binding site of the soil particles. In soils with high organic matter content (Bernam series) a higher amount of metsulfuron-methyl residue is retained compared to soils with low organic matter content (Bongor series).

Acknowledgments

The authors wish to thank DuPont, Malaysia for permission to present and publish this paper. Thanks are also extended to Dr. Harry J. Strek, DuPont Crop Protection, USA for reviewing the paper.

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