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The leaching of trifloxysulfuron-sodium and pyrithiobac-sodium in soil columns as a function of soil liming

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ABSTRACT. Scarce research has been published concerning the effect of soil pH on the leaching potential of herbicides in tropical soils. Thus, we designed this study to evaluate the influence of soil liming on the leaching of trifloxysulfuron-sodium and pyrithiobac-sodium after simulated rainfall depths in soil columns. In the study, two trials were conducted simultaneously; the first experiment evaluated trifloxysulfuron-sodium (7.5 g ha^{-1}), while the second experiment evaluated pyrithiobac-sodium (70 g ha^{-1}). Both experiments were conducted in a randomized block design with a 2 x 4 x 5 factorial scheme and four replications. The design's factors corresponded to 2 soil liming conditions (with or without liming), 4 simulated rainfall depths (0, 15, 30, and 45 mm) and 5 depths in the soil column (0-5, 5-10, 10-15, 15-20, and 20-25 cm). The trials were repeated, and only the source for the soil neutralization was changed, i.e., dolomitic limestone in Experiment 1 and calcium oxide in Experiment 2. Compared to trifloxysulfuron-sodium, the herbicide pyrithiobac-sodium indicated a greater potential for leaching. With more acidic soils, the leaching potential in limed soils was greater for both herbicides. Only the liming that used calcium oxide provided a significant leaching of trifloxysulfuron-sodium for depths greater than 20 cm. Simulated rainfall $\geq$ 15 mm provided leaching of pyrithiobac-sodium to a depth of 25 cm at near-neutral soil pH values.

Keywords: ALS inhibitors, mobility, soil pH, tropical soils.

Lixiviação de trifloxysulfuron-sodium e pyrithiobac-sodium em função da calagem do solo

RESUMO. Em razão da escassez de informações a respeito de como o pH do solo interfere no potencial de lixiviação de herbicidas em solos tropicais, foram realizados ensaios com o objetivo de avaliar a influência da calagem do solo na lixiviação dos herbicidas trifloxysulfuron-sodium e pyrithiobac-sodium, após simulação de diferentes lâminas de precipitações. Para isso, foram realizados dois ensaios simultaneamente, um para o trifloxysulfuron-sodium (7,5 g ha^{-1}) e outro para o pyrithiobac-sodium (70 g ha^{-1}), em esquema fatorial 2 x 4 x 5, sendo os fatores duas condições de calagem do solo (com e sem calagem), 4 lâminas de irrigação (0, 15, 30 e 45 mm) e 5 profundidades na coluna (0-5, 5-10, 10-15, 15-20 e 20-25 cm). Os experimentos foram repetidos no tempo, mudando-se apenas a fonte utilizada para neutralização do solo (calcário dolomítico e óxido de cálcio, respectivamente). O herbicida pyrithiobac-sodium apresentou maior potencial de lixiviação que o trifloxysulfuron-sodium. Em solos submetidos à calagem, há maior movimentação de ambos herbicidas. Somente a calagem do solo com óxido de cálcio proporcionou lixiviação expressiva de trifloxysulfuron-sodium para profundidades maiores do que 20 cm. Lâminas a partir de 15 mm proporcionaram acentuada lixiviação de pyrithiobac-sodium em solos com pH próximo da neutralidade, sendo este herbicida detectado até 25 cm de profundidade.

Palavras-chave: inibidores de ALS, mobilidade, pH do solo, solos tropicais.

Introduction

An herbicide's fate is affected by the processes related to its retention, transformation and transport in the soil. The mobility of a particular herbicide has a major influence on the herbicide's effectiveness in weed control and its dissipation into the environment.

Leaching is the major transport pathway for non-volatile, water-soluble molecules. After the movement of water, these molecules shift in the soil profile, which is indicated by the difference of hydric potential between two points (PRATA et al., 2003). Leaching is a fundamental process in most herbicides' incorporation into the soil. This incorporation is necessary if the herbicides are to reach either the germinating seeds or emerging seedlings. However, over leaching might also drive these particles into deeper layers of the soil, thereby limiting the herbicide's activity and increasing its potential to become a groundwater contaminant as well.

The dynamics and fate of herbicides in the environment are influenced primarily by sorption with soil particles, which determines the availability of the molecule in the soil solution. An inverse relationship between sorption and leaching potential is usually observed for these compounds (OLIVEIRA et al., 2005).

A number of factors influence the downward movement of herbicides in soil, such as the content and composition of organic matter, texture, pH level, soil density, pore size and distribution, solubility of the herbicide molecules and site rainfall (INOUE et al., 2009, 2010; OLIVEIRA JUNIOR et al., 2001; PRATA et al., 2003; SPADOTTO; HORNSBY, 2003; SPADOTTO et al., 2005). Intense rainfall can promote leaching of the herbicides and the contamination of groundwater. Little is known about the influence of soil liming on the retention and movement of herbicides. Consequently, there is little information concerning soil liming's effects on the efficiency and leaching potential of herbicides, particularly in tropical soils (INOUE et al., 2002).

The results obtained by Matocha and Hossner (1999) indicate that pyrithiobac mobility is influenced by both the soil type and the preferential flow processes when it is leached through intact soil columns. Under field conditions, no residue of trifloxysulfuron was found in the sampled soil depths after the application of trifloxysulfuron by bioassays and High Performance Liquid Chromatography (HPLC) methods (VIVIAN et al., 2007).

Studies concerning the fate of trifloxysulfuron-sodium and pyrithiobac-sodium in tropical soils are scarce, but such investigations are needed to understand the dynamics of these herbicides in the environment. The aim of this study was to evaluate the leaching potential of the herbicides trifloxysulfuron-sodium and pyrithiobac-sodium in limed soil columns.

Material and methods

Under greenhouse conditions, two trials were simultaneously conducted. The first trial was conducted for trifloxysulfuron-sodium, while the second trial was conducted for pyrithiobac-sodium. Each trial was arranged in a completely randomized design with a 2 x 4 x 5 factorial scheme. The factors were assigned as follows: First factor - Soil liming (with or without); Second factor – Irrigation depths (0, 15, 30 and 45 mm); and the Third factor - Depth ranges in soil columns (0-5, 5-10, 10-15, 15-20 and 20-25 cm).

The trials were performed twice at distinct periods of time. The first experiment (Experiment 1) was conducted from May to June 2010, and the second experiment (Experiment 2) was conducted from October to November 2010.

In all of the experiments and trials, soil samples were collected from 0-20 cm depth of a soil classified as Rhodic Ferralsol (Typic Haplorthox) (EMBRAPA, 2006). The samples were then air-dried, sieved (2 mm) and stowed in plastic bags. The main physicochemical properties were pH (H_2O) 4.2, 4.06 $cmol_c\ dm^{-3}$ $H^+ + Al^{3+}$, 0.45 $cmol_c\ dm^{-3}$ Ca^{+2}, 0.21 $cmol_c\ dm^{-3}$ Mg^{+2}, 0.07 $cmol_c\ dm^{-3}$ K^+, 1.4 mg dm^{-3} P and 9.21 g dm^{-3} OC. The soil texture was classified as sandy-clay-loam (79% sand, 20% clay and 1% silt). The humid climatic conditions of tropical Brazil have resulted in the formation of extremely old and deeply weathered soils. The properties of these soils contain the depletion of major elements, such as Si, Ca, Mg, K and Na, and the relative accumulation of Fe and Al oxides and hydroxides. The cation exchange capacity and pH values are generally low, as is the content of the soil organic matter. Ferralsols are typical soils that are formed because of ferrallitic processes. These soils have a substantial, homogeneous B horizon that merges with the C horizon in great depths.

In experiment 1, the soil acidity was neutralized by the application of dolomitic limestone at 4500 kg ha^{-1} (RPTN - Relative Power of Total Neutralization 87%). After mixing the lime thoroughly in the soil, the samples were incubated for 90 days with sufficient moisture to enable the reaction between the lime and soil. After the incubation period, the pH (H_2O) in the soil was 5.5. In experiment 2, the soil acidity was neutralized by the application of calcium oxide (CaO) at 3500 kg ha^{-1} (RPTN 125%). The incubation period was 30 days, and the final soil pH (H_2O) was 7.2. All calculations for soil neutralization were performed per hectare and took into account the soil volume at a depth of 0.20 m.

The soil samples were packed in PVC columns (0.01 m diameter, 0.30 m height). Each column was previously sectioned longitudinally according to the procedure proposed by Inoue et al. (2010). A polyethylene piece of fabric (mesh = 1 mm) attached by a flexible piece of rubber was placed at the bottom of each column to prevent soil losses. To keep the two halves together, the columns were taped and tied with smooth wire. Each column was filled with 3 kg of soil. After packing, the columns were wetted by capillarity for 24 h, at which point the soil was saturated to the top of the column. The soil columns were subsequently kept on greenhouse benches for 24 h to drain any excess water.

The herbicides trifloxysulfuron-sodium (7.5 g ha^{-1}) and pyrithiobac-sodium (70 g ha^{-1}) were applied to the top of the soil columns on 5/16/2010 (experiment 1) and 10/7/2010 (experiment 2). In both experiments, the applications were made using a backpack sprayer that was pressurized by CO_2 with a flat fan nozzle XR110.02 at 200 KPa, which caused the application volume to reach 200 L ha^{-1}. The air temperature, relative air humidity and wind speed for the first and second applications were 22ºC and 18ºC, 78 and 85%, and 1.0 and 1.4 km h^{-1}, respectively.

Twenty-four hours after the application of the herbicides, rainfall was simulated in the top of the columns at depths of 0, 15, 30 and 45 mm. One day after the rainfall simulation, the columns were separated lengthwise. From the surface where the herbicides had been applied, each half was divided into five sections of 5 cm (depths ranging from 0-5, 5-10, 10-15, 15-20 and 20-25 cm). The soil from each of these depths was transferred to 250 cm^3 polyethylene pots. Based on a previous bioindicator selection, three seeds of cucumber (*Cucummis sativus*) were then sown per pot (GUERRA et al., 2011). A microsprinkler irrigation, which supplied the pots with adequate water, was performed twice a day. In the figures depicting the results of both experiments, each depth range of 5 cm is numerically represented by the largest absolute value.

Twenty-one days after the bioindicator sowing, the number of live plants per pot was recorded, and the shoots were harvested. The biomass was weighed on a precision scale to obtain the fresh weight of the shoots. Using these data, the percentage inhibition in relation other treatments for each soil liming condition was calculated.

Because the trials were performed twice, a combined analysis was run, which led to the conclusion that each experiment should be analyzed separately.

The data were subjected to an ANOVA and F test ($p < 0.05$) and to a regression analysis. Based on their biological significance and determination coefficients, the regression models were fitted to the data. An F test ($p < 0.05$) was also performed for the unfolding of the liming effects in each soil depth range and the depths of simulated rainfall.

Results and discussion

Related to the inhibition of shoot fresh biomass, there was a significant interaction between soil liming, simulated rainfall and the depth range of herbicide leaching for both herbicides and experiments. The leaching of trifloxysulfuron-sodium and pyrithiobac-sodium along the soil profile was observed in both experiments.

For the simulated rainfall depths of 30 and 45 mm, the mobility of trifloxysulfuron-sodium (Experiment 1 - Figure 1A) in unlimed soil was observed along the top 15 cm of the soil columns. The rainfall depths of 0 and 15 mm did not cause the displacement of this herbicide in depths exceeding 10 cm.

In Experiment 2 (Figure 1B), the leaching of trifloxysulfuron-sodium with no rainfall simulation was limited to 0 to 10 cm soil depth. However, by simulating any of the simulated rainfall depths, herbicide leaching was found at a depth of 20 cm with no important differences among the rainfall depths.

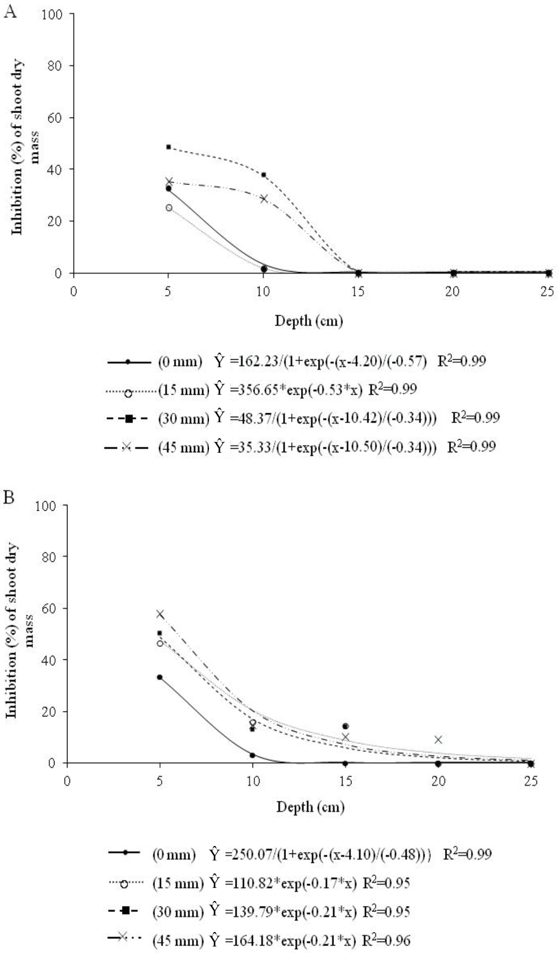

Figure 1. Trifloxysulfuron-sodium's inhibition of cucumber shoot biomass as a function of soil depth (% of check) in unlimed soil (pH 4.2) in Experiment 1 (A) and Experiment 2 (B).

When the soil was limed (pH 5.5) (Figure 2A), a simulated rainfall of 45 mm was the only level of rainfall that changed the magnitude and depth of herbicide leaching. For the simulated rainfalls of 0, 15 and 30 mm, there was virtually no biomass inhibition below 15 cm. However, a simulated rainfall of 45 mm inhibited at least 43% of biomass at that depth range.

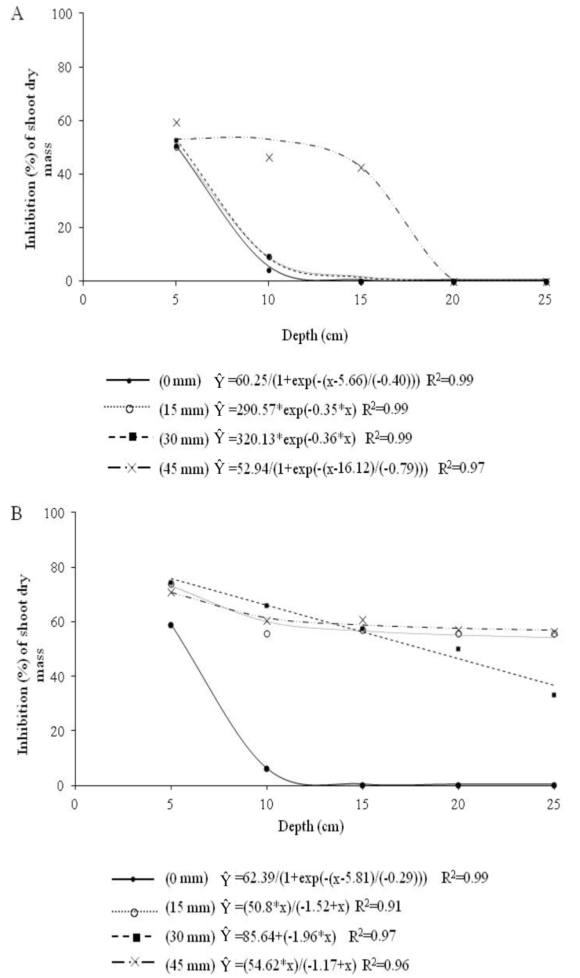

Figure 2. Trifloxysulfuron-sodium's inhibition of the shoot fresh biomass of the cucumber as a function of soil depth (% in relation to check) in plants grown in limed soils in experiment 1- pH 5.5(A) and experiment 2 - pH 7.2 (B).

In the absence of a rainfall simulation, when trifloxysulfuron-sodium was applied to soil limed with calcium oxide (pH 7.2), a perceptible downward movement was observed within 12 cm of depth (Figure 2B). Herbicidal activity was detected at 25 cm of depth when simulated rainfall depths of 15, 30 and 45 mm were applied, thereby providing evidence that in a soil with a pH close to neutrality, the occurrence of precipitation might lead to herbicide leaching in deeper soil layers.

These results are similar to those observed by Vivian et al. (2007), who found that soils with high contents of calcium and low contents of iron and aluminum oxides and hydroxides (similar to the limed soils in this study) usually present poor sorption of trifloxysulfuron-sodium. This poor sorption allowed for a greater movement of the herbicide in the soil profile and consequently reduced the herbicidal activity in topsoil layers under intensive rainfall.

The comparisons of the shoot fresh biomass inhibition in both experiments and soil liming conditions are indicated in Table 1. Generally, the percentages of shoot biomass inhibition were similar to or higher than those percentages found for unlimed soil, regardless of the experiment. Despite the differences found in the results for both experiments, increasing the depth of simulated rainfall generally resulted in an increased leaching of herbicide in the column profile. According to Goetz et al. (1986), sulfonylureas are relatively mobile in soil, and their mobility in soil is increased by increased increments in the soil pH levels.

Table 1. Trifloxysulfuron-sodium's inhibition of the shoot fresh biomass of cucumber plants (% in relation to check) in limed and unlimed soils in progressive depth ranges of soil.

		Trifloxysulfuron-sodium							
Rainfall depth (mm)	Column depth (cm)	Experiment 1				Experiment 2			
		Unlimed		Limed		Unlimed		Limed	
0	5	32.50	b	50.75	a	33.48	b	58.77	a
	10	3.00	a	0.00	a	2.55	a	0.00	a
	15	0.00	a	0.00	a	0.00	a	0.00	a
	20	0.00	a	0.00	a	0.00	a	0.00	a
	25	0.00	a	0.00	a	0.00	a	0.00	a
15	5	25.00	b	50.50	a	46.78	b	73.95	a
	10	2.00	a	9.25	a	15.90	b	55.80	a
	15	0.00	a	0.00	a	14.67	b	57.18	a
	20	0.00	a	0.00	a	0.00	b	55.90	a
	25	0.00	a	0.00	a	0.00	b	55.80	a
30	5	48.50	b	52.50	a	50.60	b	74.48	a
	10	9.00	b	37.50	a	13.28	b	65.78	a
	15	0.00	a	0.00	a	14.12	b	57.47	a
	20	0.00	a	0.00	a	0.00	b	50.03	a
	25	0.00	a	0.00	a	0.00	b	33.23	a
45	5	35.25	b	59.25	a	58.02	a	71.05	a
	10	28.50	b	46.25	a	15.00	b	60.50	a
	15	0.00	b	42.75	a	10.48	b	60.95	a
	20	0.00	a	0.00	a	9.25	b	57.25	a
	25	0.00	a	0.00	a	0.00	b	56.40	a
CV (%)	-	35.53							

In each experiment, the means followed by the same letter in the lines do not differ (F test $p < 0.05$).

Regardless of the depth of simulated rainfall, when pyrithiobac-sodium was applied to unlimed soil (pH 4.2) (Experiment 1 - Figure 3A), the movement of the herbicide was found to the 25-cm column depth. When irrigation depths of 0, 15 and 30 mm were simulated, inhibitions of cucumber fresh shoot biomass between 6 and 21% were found. These inhibitions increased to 45% under simulated rainfalls of 45 mm.

In Experiment 2, which was conducted in acidic (unlimed) soil (pH 4.2) (Figure 3B), the activity of pyrithiobac-sodium was detectable only in the top 11 cm of soil columns when there was no rainfall simulated. The other rainfall depths shifted the herbicide down to the 25 cm depth. For rainfall depths of 15 and 30 mm, there was practically no difference in the inhibition, which ranged from 9 to

13%. At 25 cm depth, the inhibition was 5%. With a rainfall simulation of 45 mm, the inhibition for this column depth was 32%.

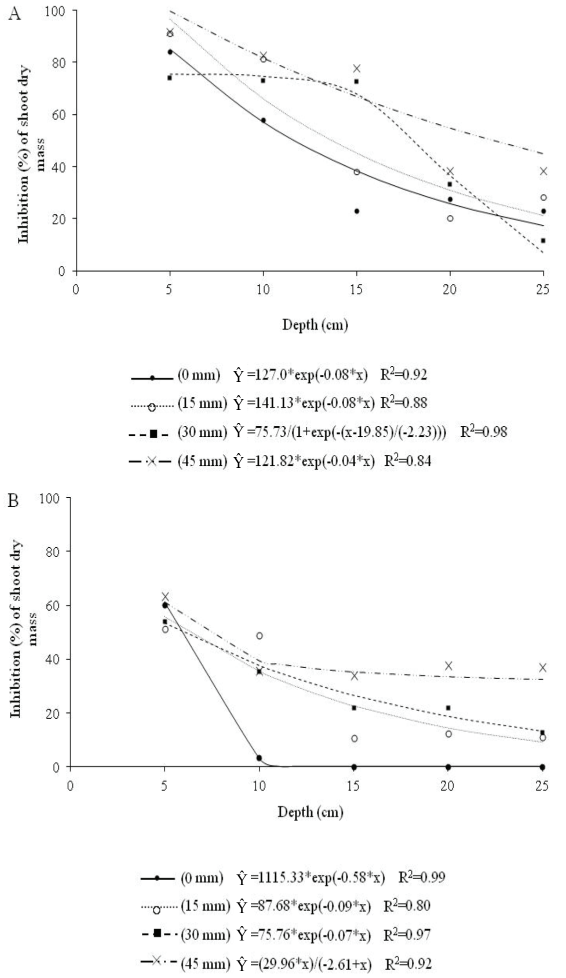

Figure 3. Pyrithiobac-sodium's inhibition of cucumber shoot biomass as a function of soil depth (% of check) in unlimed soil (pH 4.2) in Experiment 1 (A) and Experiment 2 (B).

Regardless of the chemical source used to neutralize the soil, when pyrithiobac-sodium was applied to the limed soil, a similar result was found (Figures 4A and B). With no rainfall simulation, the herbicide concentrated within the initial section of the soil column caused the highest percentages of bioindicator growth inhibition. However, even at 25 cm of soil column depth, herbicide activity was detected. This activity led to an inhibition of biomass accumulation of 46 and 52% for Experiment 1 (pH 5.5) and Experiment 2 (pH 7.2), respectively.

For the simulated rainfall depths of 15, 30 and 45 mm, there were no differences found in the inhibition percentage of the cucumber shoot biomass along the soil column. Therefore, no models were adjusted for any of the former experiments with soil liming. In Experiment 1 (pH 5.5), the simulated rainfall depths of 15, 30 and 45 led to the inhibitions along the soil columns of 55, 59 and 57%, respectively. In Experiment 2 (pH 7.2), the inhibitions were 67, 71 and 83%. In Experiment 2 (pH 7.2), the inhibitions of fresh shoot biomass were higher than the values observed in Experiment 1 (pH 5.5) for every depth of simulated rainfall.

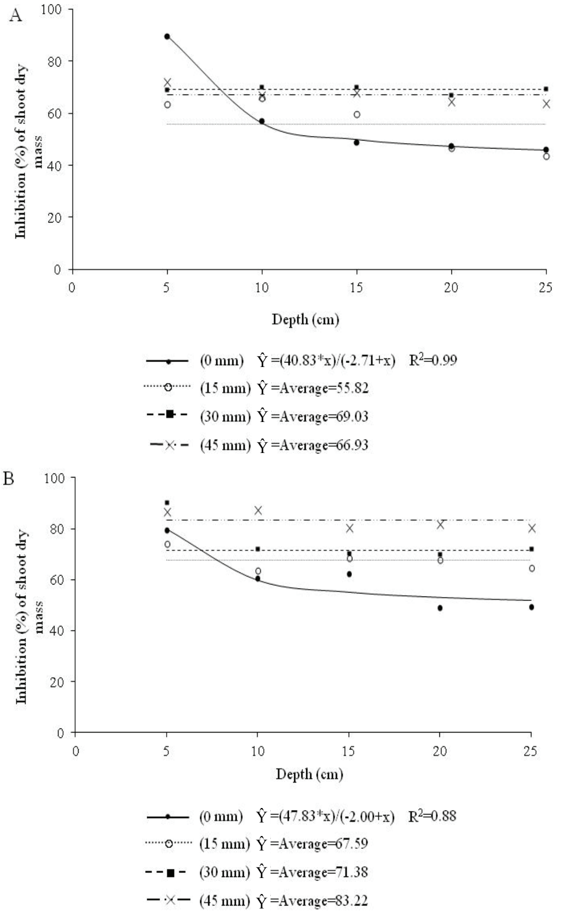

Figure 4. Pyrithiobac-sodium's inhibition of the shoot fresh biomass of the cucumber as a function of soil depth (% in relation to check) in plants grown in limed soils in Experiment 1- pH 5.5(A) and Experiment 2 - pH 7.2 (B).

Table 2 contains the comparisons of the bioindicator shoot fresh biomass inhibition in both the limed and unlimed soils that received an application of pyrithiobac-sodium. In the limed soils (utilizing either dolomitic limestone or calcium oxide), the inhibition of biomass was similar to or higher than the inhibition observed in unlimed soils for all depths of the soil columns.

When comparing the two herbicides in this study, pyrithiobac-sodium indicates greater mobility, regardless of the chemical source used to neutralize the soil and the depth of simulated rainfall. The only condition at which the leaching of trifloxysulfuron-sodium was found was at 25 cm when the soil was limed with calcium oxide and rainfall depths from 15 to 45 mm were simulated. However, even under those circumstances, the inhibition of the bioindicator shoot biomass remained lower than the levels observed with the application of pyrithiobac-sodium.

Table 2. Pyrithiobac-sodium's inhibition of the shoot fresh biomass of the cucumber plants (% in relation to check) in limed and unlimed soils in progressive depth ranges of the soil columns.

		Pyrithiobac-sodium			
Rainfall depth (mm)	Column depth (cm)	Experiment 1		Experiment 2	
		Unlimed	Limed	Unlimed	Limed
0	5	70.68 a	89.50 a	59.92 a	79.25 b
	10	30.80 a	56.92 b	3.25 a	60.50 b
	15	11.65 a	48.77 b	0.00 a	62.13 b
	20	13.90 a	47.36 b	0.00 a	48.98 b
	25	11.54 a	46.00 b	0.00 a	49.00 b
15	5	63.25 a	71.21 a	51.08 a	73.90 b
	10	65.15 a	65.92 a	48.92 a	63.32 a
	15	24.40 a	59.57 b	10.65 a	68.35 b
	20	16.38 a	46.62 b	12.32 a	67.65 b
	25	19.82 a	43.55 b	11.02 a	64.65 b
30	5	64.01 a	69.10 a	53.85 a	73.22 b
	10	54.26 a	69.82 a	35.50 a	71.95 b
	15	47.35 a	70.03 b	21.85 a	70.23 b
	20	27.47 a	66.75 b	21.70 a	69.65 b
	25	12.24 a	69.18 b	12.70 a	71.95 b
45	5	72.08 a	77.76 a	63.45 a	86.65 b
	10	59.36 a	66.67 a	35.80 a	87.37 b
	15	56.08 a	68.03 a	33.98 a	80.35 b
	20	38.16 a	64.30 b	37.95 a	81.57 b
	25	37.70 a	63.62 b	36.90 a	80.17 b
CV (%)		22.70			

The means followed by the same letter in the lines for each experiment do not differ (F test, $p < 0.05$).

Based on the results reported here, both herbicides are mobile in soil. However, because it was easily detected in depths down to 25 cm in the soil columns, pyrithiobac-sodium indicates a higher leaching potential in the soil profile. These results suggest similarities with the results obtained by Veletza et al. (2005), who observed the movement of pyrithiobac-sodium down to 30 cm depth in soil of different textures under a simulated rainfall of 35 mm.

Ionizable herbicides, such as trifloxysulfuron-sodium (pKa = 4.76) and pyrithiobac-sodium (pKa = 2.34), have two distinct forms as related to their chemical net charges. In pH levels lower than pKa, the main chemical species is the molecular form, which has no net charge. When the soil pH approaches neutrality, the anionic species is predominant, thereby decreasing the chemical attraction between the herbicide molecules and soil net charges (INOUE et al., 2002). This finding implies that a lower herbicide sorption to the soil increases leaching in such soils.

Pyrithiobac, which is a weak acidic herbicide with a pK_a of 2.34, aqueous solubility of 705 g L^{-1} (2 M) at pH 7 and 264 g L^{-1} (0.77 M) at pH 5, and a molecular mass of 326.4 g mol^{-1}, will behave as a singly charged anion at typical soil solution pH values (pH > 5). According to previous findings (MATOCHA; HOSSNER, 1998), which suggests that pyrithiobac is potentially mobile, the movement of pyrithiobac in soils should not be mitigated by sorption.

Carter (2000) has suggested that under normal circumstances, the amount of herbicide lost by leaching in the soil profile is usually less than 1% of the total herbicide applied. However, for weak acid herbicides such as imazaquin, more intense leaching occurs in soils with pH values that are almost neutral. This leaching can affect the herbicide's weed-controlling ability and the herbicide's persistence in the environment (INOUE et al., 2002). The elevation of soil pH levels also provides increased mobility for other weak acid imidazolinone herbicides, such as imazaquin, imazethapyr and imazapic (INOUE et al., 2009; STOUGAARD; MARTIN, 1990), or for similar soil-applied insecticides, such as imidacloprid (OLIVEIRA JUNIOR et al., 2000).

Water solubility is another chemical property that might facilitate herbicide leaching. In turn, the herbicide's water solubility is controlled by medium pH for sulfonylurea herbicides. The water solubility of trifloxysulfuron-sodium is 63 mg L^{-1} in pH 5 and 5016 mg L^{-1} in pH 7, i.e., it increases almost 100 x with the increase of two units of pH (MATOCHA; SENSEMAN, 2007). For pyrithiobac-sodium, the water solubility values are 264 g L^{-1} (pH 5.0) and 705 g L^{-1} (pH 7.0) (MATOCHA; HOSSNER, 1998). Bentazon, which is another weak acid herbicide (pKa = 3.2), proved to be highly mobile in neutral soils (ABERNATHY; WAX, 1973). Such behavior was attributed to the combined effects of relatively high water solubility and weak acid properties.

Conclusion

Pyrithiobac-sodium indicates higher leaching potential than trifloxysulfuron-sodium. Compared to more acidic soils, there is increased leaching of both herbicides in limed soils. Only the liming that used calcium oxide provided a significant leaching of trifloxysulfuron-sodium for depths greater than 20 cm. Pyrithiobac-sodium is notably mobile in soil, and regardless of the amount of simulated rainfall after the herbicide application, its herbicidal activity was observed to a depth of 25 cm. Simulated rainfalls depths ≥ 15 mm provided extensive leaching of pyrithiobac-sodium in soils with pH values that were almost neutral.

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