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Article

Estimation of Sulfentrazone Leaching in Isolated Application and in Mixture with Glyphosate

Estimativa de Lixiviação do Sulfentrazone em Aplicação Isolada e em Mistura com Glyphosate

LANGARO, A.C.[1]*
BARROS, J.P.A.[1]
SOUZA, M.F.[1]
SILVA, E.M.G.[1]
SILVA, A.A.[1]

ABSTRACT - As an alternative to control of resistant biotypes, farmers have resorted to the use of herbicides that have residual soil activity and good pre-emergence action. One of the most used mixtures in Brazil is that of sulfentrazone + glyphosate. When a herbicide has a pre-emergence action, small leaching of its molecules is necessary for its superficial incorporation into the soil, where most of the weed seeds are found. However, if excessive leaching occurs, the herbicide may have reduced efficiency in controlling weed, increasing the risks of groundwater contamination. In this study, sulfentrazone leaching was evaluated in columns with soil, in isolated applications and in mixture with formulations of glyphosate (Roundup Ready®, Roundup Ultra® and Zapp Qi®). For this, the columns were filled with samples of Red-Yellow Latosol, collected in the 0-20 cm depth layer. After moistening the columns to near field capacity, the herbicides were applied to the top and, 24 hours later, a 60 mm rainfall was simulated. After 72 hours, the columns were sectioned in 5 cm segments, collecting the soil from each segment. In these samples, indicator plants (*Sorghum bicolor*) were cultivated for the presence of the herbicide. It is concluded that, in general, glyphosate does not alter the leaching potential of sulfentrazone in the Red-Yellow Latosol. However, mixtures with Roundup Ready® and Zapp Qi® reduce the sorptive forces of sulfentrazone to soil colloids. Therefore, in some types of soil, herbicide desorption may occur with greater intensity, making its availability in the soil solution more difficult. This may result in better control of plants and/or intoxication of sensitive crops.

Keywords: herbicide, environmental impact, soil, bioassay.

RESUMO - Como alternativa de controle de biótipos resistentes, os agricultores têm recorrido ao uso de herbicidas que possuem atividade residual no solo e têm boa ação em pré-emergência. Uma das misturas mais utilizadas no Brasil é a de sulfentrazone + glyphosate. Quanto um herbicida tem ação em pré-emergência, pequena lixiviação de suas moléculas é necessária para sua incorporação superficial no solo, onde se encontram a maioria das sementes das plantas daninhas. No entanto, ocorrendo lixiviação excessiva, o herbicida pode ter sua eficiência no controle das plantas daninhas reduzida, aumentando os riscos de contaminação do lençol freático. Neste trabalho foi avaliada a lixiviação do sulfentrazone, em colunas com solo, em aplicações de forma isolada e em mistura com formulações de glyphosate (Roundup Ready®, Roundup Ultra® e Zapp Qi®). Para isso, as colunas foram preenchidas com amostras de Latossolo Vermelho-Amarelo, coletadas na camada de 0-20 cm de profundidade. Após umedecimento das colunas, até próximo à capacidade de campo, aplicaram-se os herbicidas no topo e, 24 horas depois, foi simulada chuva de 60 mm. Após 72 horas, as colunas foram seccionadas em

*** Corresponding author:**
<langaro.ac@gmail.com>

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[1] Universidade Federal de Viçosa, Viçosa-MG, Brasil.

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*segmentos de 5 cm, coletando-se o solo de cada segmento. Nessas amostras foram cultivadas plantas indicadoras (**Sorghum bicolor**) da presença do herbicida. Conclui-se que, em geral, o glyphosate não altera o potencial de lixiviação do sulfentrazone no Latossolo Vermelho-Amarelo. No entanto, as misturas com Roundup Ready® e Zapp Qi® reduzem as forças sortivas do sulfentrazone aos coloides do solo. Em razão disso, em alguns tipos de solo poderá ocorrer com maior intensidade a dessorção do herbicida, tornando maior a sua disponibilidade na solução do solo. Isso poderá resultar em melhor controle das plantas e/ou intoxicação das culturas sensíveis.*

Palavras-chave: herbicida, impacto ambiental, solo, bioensaio.

INTRODUCTION

The use of herbicides is essential to avoid the losses caused by the competition of weeds in crops in large areas. However, the misuse and indiscriminate use of these pesticides has caused environmental problems, such as contamination of the soil (making crop rotation unviable) and ground or surface water bodies (Tanabe et al., 2001; Santos et al., 2015).

In Brazil, sulfentrazone is widely used in pineapple, coffee, sugar cane, citrus, tobacco and soybean crops. It has excellent activity when applied in pre-emergence to control eudicotyledonous weeds and several monocotyledonous species (Agrofit, 2017). In addition, in recent years it has been used as one of the alternatives in the control of glyphosate resistant biotypes in soybean crop (Knezevic et al., 2009; Minozzi et al., 2014). In this case, sulfentrazone is often used in admixture with glyphosate for a broader spectrum of action, as well as economic advantages.

When a pesticide is applied in the field, large proportion reaches the soil where several factors can influence its final destination. Some factors depend on the intrinsic properties of the pesticide (octanol-water partition coefficient, solubility and acid dissociation constant). Other factors depend on the physical-chemical and biological properties of the soil (e.g. organic matter, moisture, pH, mineralogy) (Holland, 2004). Soil properties are also influenced by climatic factors, such as precipitation and temperature, as well as by cultivation practices and soil management (Okada et al., 2016).

The knowledge of the processes that interfere in the behavior of herbicides is fundamental for the understanding of their destiny in the environment, especially in relation to the leaching process in the soil (Andrade et al., 2010). This process is characterized as the main form of transport of the herbicides, by the downward movement of the products in the soil matrix in mass flow, together with the soil water (Silva et al., 2013).

The leaching of a chemical compound in the soil is influenced by its physicochemical characteristics, whether ionic (basic or acidic) or non-ionic (neutral) (Inoue et al., 2003), by soil attributes such as pH and organic matter (Silva et al., 2013), and also by the edaphoclimatic conditions of the place of cultivation (Andrade et al., 2011). Soils with high adsorption capacity make herbicides less mobile, resulting in longer residues of these compounds in the superficial layers of the soil (Queiroz et al., 2011).

The leaching process is the main form of transport in the soil of non-volatile and water soluble molecules. These molecules move in the soil profile, following the flow of water, which is governed by the difference in water potential between two points (Prata et al., 2003). Leaching is fundamental in the surface incorporation of most herbicides, reaching seeds or plants in germination but, when excessive, it can transport them to deeper layers of the soil, limiting their action and may even promote contamination of the water table (Monquero et al., 2010).

Biological tests using plant species that show high sensitivity to the herbicide have been widely used in several studies to monitor herbicides in the soil (Pessala et al., 2004; Guerra et al., 2016; Nunes and Vidal, 2016, 2017; Refatti el al., 2017). Herbicides are toxic agents often found in very low concentrations in the soil, which makes it difficult to quantify them by chemical methods. Thus, the bioassay presents itself as an alternative to the chemical method, allowing the detection of herbicides even when in very low concentrations through the use of plants sensitive to these xenobiotics.

Several studies have shown that the dynamics of sulfentrazone depends largely on the physical and chemical characteristics of the soil, especially organic matter content, pH and mineralogy (Passos et al., 2013; Freitas et al., 2014). Some authors have classified sulfentrazone as a herbicide with high leaching potential (Ohmes and Mueller, 2007; Melo et al., 2010; Monquero et al., 2010); however, little is known about the effect of blending sulfentrazone with other herbicides, such as glyphosate, for example, on soil sulfentrazone dynamics.

In previous studies, using sorghum (*Sorghum bicolor*) as an indicator plant in the evaluation of sulfentrazone leaching in two soil types, submitted to different rainfall regimes, it was observed that under precipitation of 90 mm of rain the herbicide was detected up to 12.5 cm deep in the column containing Red Nitosol (Rossi et al., 2005). However, it is believed that when applied in admixture with glyphosate, the leaching potential of sulfentrazone can be altered. One of the hypotheses is that different formulations of glyphosate may influence in different ways the retention forces of herbicides on soil colloids. In this research, considering the biological method using sorghum as an indicator plant, it was estimated the leaching potential of sulfentrazone in isolated application and in mixture with glyphosate in different formulations.

MATERIAL AND METHODS

Place and soil samples

The work was carried out in a greenhouse using samples of a Red-Yellow Latosol, with no history of herbicide use, collected in the superficial layer (0-20 cm) of depth. These samples were dismantled, dried in the shade, sieved in 2 mm mesh and later characterized chemically and physically (Table 1).

To study the leaching of the herbicides in the soil profile, PVC columns of 10 cm in diameter and 50 cm long were used, filled with soil samples. Before filling the columns with the soil samples, paraffin was applied to the inside of the columns to avoid lateral water runoff. After being filled with the soil samples, they were saturated with water to remove the air bubbles trapped in the pores for a period of 48 hours. Subsequently, the columns were left in the upright position for 72 hours to drain the excess water, leaving the soil moisture close to the field capacity.

The design was completely randomized with four replicates, and the treatments were arranged in subdivided plots. The plots corresponded to the herbicides (Sulfentrazone, Sulfentrazone+Roundup Ready®, Sulfentrazone+Roundup Ultra® and Sulfentrazone+Zapp Qi®), and the subplots corresponded to the depths of sample collection in the soil columns (0-5, 5-10, 10-15, 15-20, 20-25, 25-30, 35-40, 40-45 e 45-50 cm).

Herbicide application and rain simulation

Herbicides were applied to the top of the columns, with high precision pressurized CO_2 (TTI 110 02 tips, calibrated to apply 150 L ha^{-1}). Sulfentrazone was applied at a dose of 1,500 g ha^{-1}, while glyphosate, in the different formulations, was applied at a dose of 1,080 g ha^{-1}. In order to

Table 1 - Results of the physical and chemical analysis of the samples of the Red-Yellow Latosol used in the experiment[1]

Sand (%)			Silt (%)				Clay (%)		
76			7				17		
pH	P	K	Ca^{2+}	Mg^{2+}	Al^{3+}	H+Al	SB	CTC(t)	MO
(H_2O)	(cmolc dm^{-3})								(dag kg^{-1})
4.7	2.33	41	2.2	0.7	0.2	5.61	3	3.2	2.52

[1] Analyzes carried out in the Laboratório de Análises de Solo Viçosa (Soil Analysis Laboratory of Viçosa), according to the methodology of the Empresa Brasileira de Pesquisa Agropecuária – Embrapa (Brazilian Agricultural Research Company – 1997); SB - base sum; CTC - cation exchange capacity; MO - organic matter.

avoid drift, the applications were carried out at 25 °C room temperature and 70% relative humidity. Twenty-four hours after the application of the herbicide, 60 mm of rain were simulated for two hours, with the columns standing upright. During the simulated rainfall applications, the volume of water reaching each column was monitored with rain gauges. The columns were then held for 72 hours to drain excess remaining water. Thereafter, the columns were cut into 5 cm segments representing the depths. The soil samples contained in each segment were homogenized and transferred to pots with a volumetric capacity of 0.3 L, where the indicator species were cultivated.

Cultivation of the indicator plant

Sorghum was cultivated in soil samples collected in each segment of the column at different depths, spaced 5 cm apart, for 21 days. During this period, the soil moisture in the pots was maintained close to the field capacity, replenishing evapotranspiration water daily. Visual assessments were performed at 7, 14 and 21 days after sowing (DAS). At that time, the symptoms of intoxication of sorghum plants were evaluated visually, assigning scores from 0 (absence of intoxication symptoms) to 100 (plant death) (Frans et al., 1986). At 21 DAS, the aerial part of the plants was collected to determine the dry matter. For this, the collected plants were dried in greenhouse (70 ± 2 °C) until reaching a constant mass, and then the dry matter of the aerial part was determined using a precision balance of 0.001 g.

Analysis of Results

In order to evaluate the intoxication of the sulfentrazone indicator plant as a function of depth, a bar chart with standard deviation was used (the depth on the Y axis and the intoxication on the X axis).

For the representation of the dry matter of the aerial part of the sulfentrazone indicator plant as a function of depth, a bar chart with standard deviation was used (the depth on the Y axis and the dry matter on the X axis).

The graphical analyzes were done using the program SigmaPlot 12.0 (Exact Graphs and Data Analysis) for Windows.

RESULTS AND DISCUSSION

Higher intoxication was observed in plants of the 0-5 cm layer, regardless of the applied herbicide treatment. In all cases, at 7 DAS, the herbicide was detected up to 15 cm deep, with no symptoms of intoxication in the sorghum plants at the greatest depths. Intoxication symptoms observed in plants treated with sulfentrazone are characterized by necrosis of epicotyl, leaves and stems, which results in the death of plants (Silva et al., 2014). The damage to the tissue of the sensitive plant occurs by contact with the sulfentrazone at the moment the seedling emerges, the light being necessary for the herbicidal action.

Intoxication due to herbicides sulfentrazone+Roundup Ready® and sulfentrazone+Zapp Qi® was higher than the other treatments, indicating a higher availability of the herbicide in the soil solution (Figure 1), which may result in higher leaching potential. However, only the increased availability of the herbicide in the soil solution is not sufficient to predict the potential for leaching. In addition to the amount of herbicide available, the physical-chemical characteristics of the herbicide, the soil attributes and the soil-climatic conditions also influence the leaching process. Thus, rains that provide water depths greater than 60 mm can carry the herbicide to deeper layers, and this risk is greater when using Roundup Ready® or Zapp Qi® in the mixture, since more herbicide is available in the soil solution. In the present study, rainfall may have been insufficient to reduce sulfentrazone concentration in the upper layers, as higher rainfall intensities may distribute the herbicide in the column (Mueller et al., 2014; Wlodarczyk, 2014).

In Figures 2 and 3, the intoxication values of sorghum plants at 14 and 21 DAS, respectively, are presented as a function of the application of sulfentrazone alone and in mixture with the

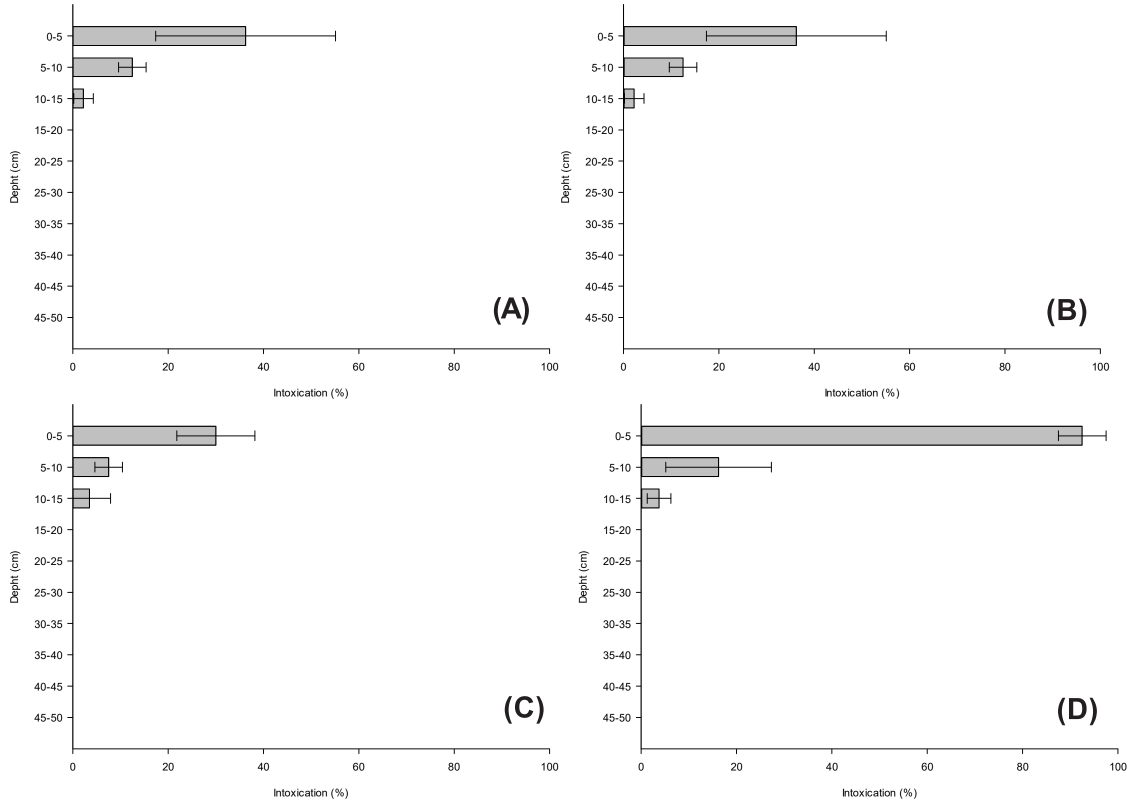

Figure 1 - Percentage of intoxication of sorghum plants at seven days after sowing in soil samples, collected at different depths, treated with sulfentrazone alone (A) and mixed with glyphosate (Roundup Ready (B), Roundup Ultra (C) and Zapp Qi (D)).

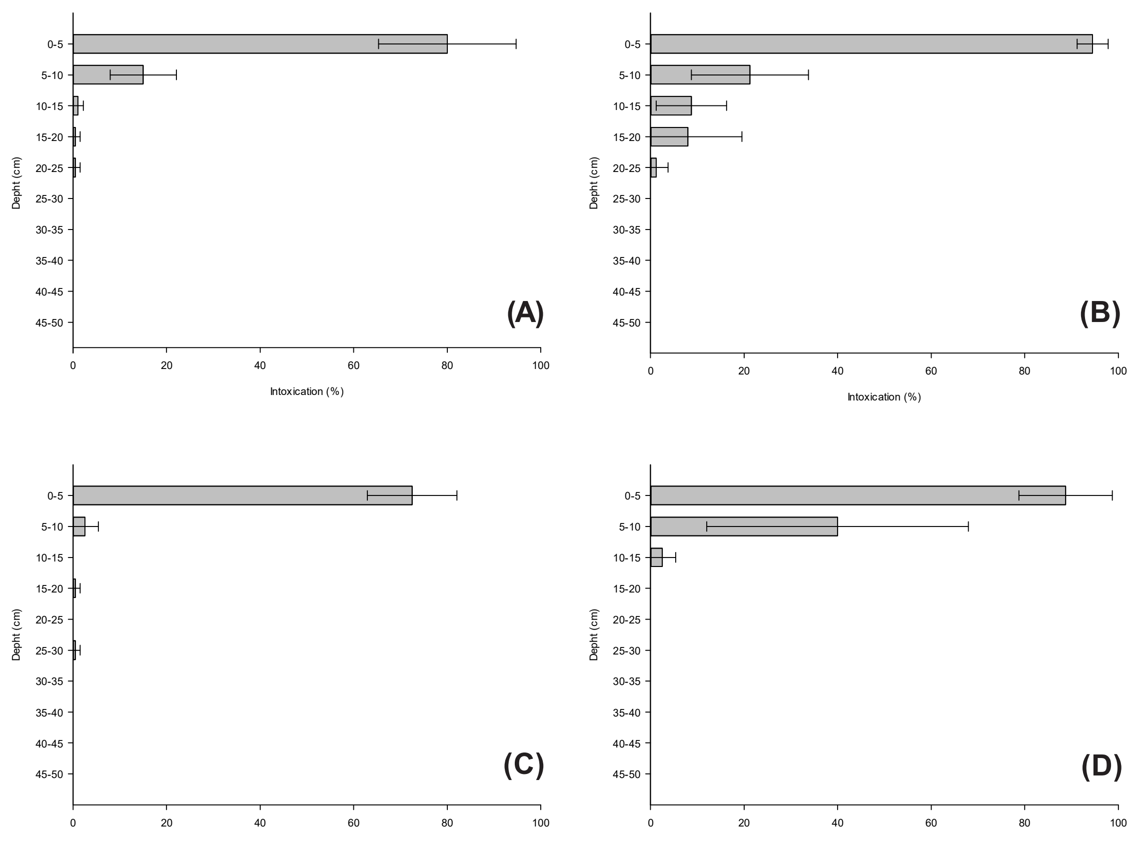

Figure 2 - Percentage of intoxication of sorghum plants at 14 days after sowing in soil samples, collected at different depths, treated with sulfentrazone alone (A) and mixed with glyphosate (Roundup Ready (B), Roundup Ultra (C) and Zapp Qi (D)).

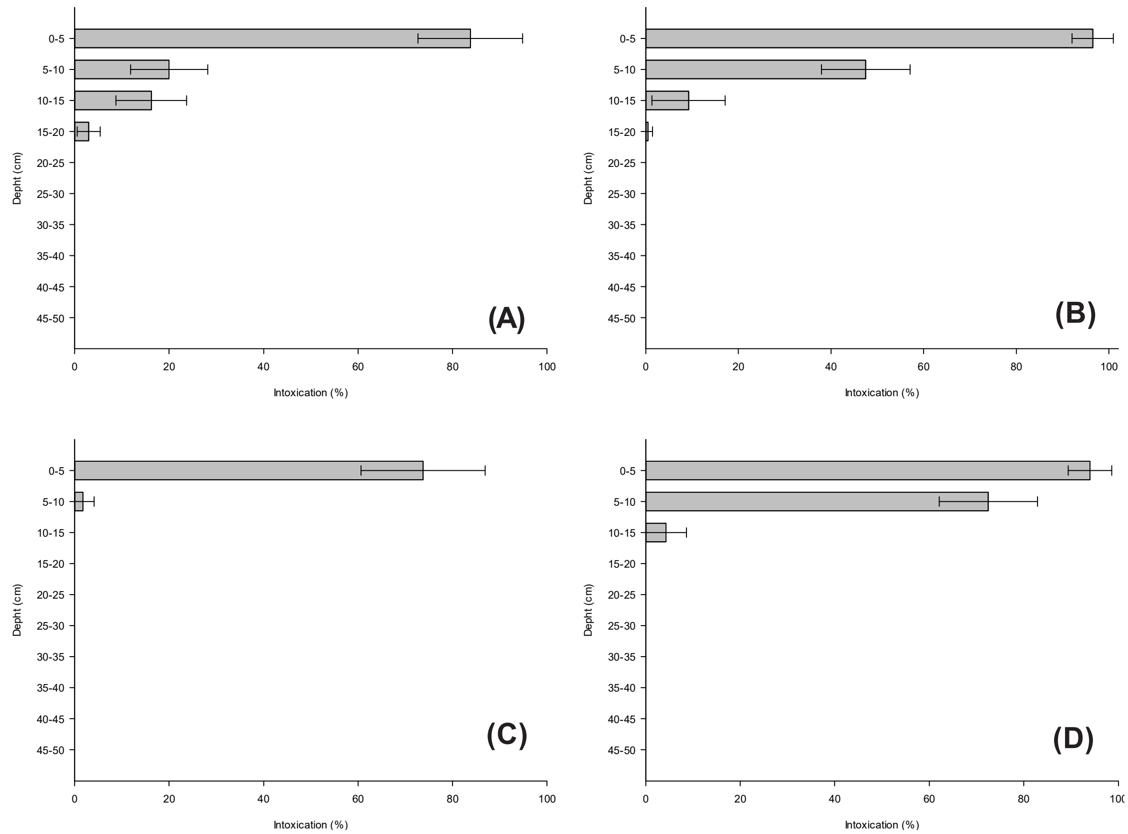

Figure 3 - Percentage of intoxication of sorghum plants at 21 days after sowing in soil samples, collected at different depths, treated with sulfentrazone alone (A) and mixed with glyphosate (Roundup Ready (B), Roundup Ultra (C) and Zapp Qi (D)).

glyphosate formulations. It was verified that the results obtained at 14 and 21 DAS were similar to those observed at 7 DAS, but with symptoms evolving at 0-5 cm layers, even when sulfentrazone was used alone or in combination with Roundup Ultra®. Similar results were observed by Freitas et al. (2014), where sulfentrazone significantly reduced the dry matter of *S. bicolor* to the depth of 10 cm but, with the addition of the adjuvant, this effect was limited to the superficial 5 cm. Also, Rossi et al. (2005) evaluated the sulfentrazone leaching in PVC columns at different rainfall rates (30, 60 and 90 mm) under greenhouse conditions using a Red Latosol and a Chernosol. These authors report that in the Chernosol there was uniformity of the distribution of the herbicide along the tube, proportional to the precipitation and, in the Red Latosol, the sulfentrazone was not very mobile, remaining in the superficial layer, independently of the volume of rainfall.

The mixture of sulfentrazone with Roundup Ultra® provided less intoxication in the plants, evidenced by the absence of symptoms in the layers of less than 10 cm, corroborating the results found when sulfentrazone was applied in isolation. It is suggested that the presence of Roundup Ultra® did not alter the sorption of sulfentrazone and, therefore, the symptoms of intoxication were less evident. In addition, in the evaluation of intoxication at 14 DAS, the presence of the herbicide up to 35 cm depth was observed when sulfentrazone was used in combination with Roundup Ultra®; at 7 DAS, it had only been detected in layers below 15 cm.

Although sulfentrazone was detected in the 35 cm layer, there was generally less intoxication of the plants when it was used in a mixture with Roundup Ultra®, regardless of the evaluation period. It is believed that, under these conditions, sulfentrazone sorption has not been altered when compared to sulfentrazone in isolated application, resulting in less intoxication and also reducing leaching potential. In the other treatments, the application of Roundup Ready® and Zapp Qi® altered the sulfentrazone sorting forces, making it more readily available in the soil solution and potentially altering the sulfentrazone leaching potential.

Glyphosate is a non-selective, post-emergent herbicide that has four ionization constants (pKa = 2.0; 2.6; 5.8 and 10.8) (Sprankle et al., 1975) and high solubility in water (11.6 g L^{-1} at

25 ºC) (Montgomery, 1993), which may increase the risk of being transported in the aqueous phase. On the other hand, it has a high tendency to adsorb to the soil particles, contributing to the accumulation of glyphosate in the soil. However, this adsorption is influenced by several factors, such as the clay content and the cation exchange capacity (Hiera da Cruz et al., 2007). The soil used in the present study had relatively low clay content (17%), but it was believed that it was sufficient for the glyphosate to be sorbed to the soil colloids and, therefore, exerted little or no influence on the sulfentrazone sorption.

Another factor that may have contributed to the high sorption of sulfentrazone is soil pH (pH = 4.7). Sorption and mobility of sulfentrazone depend on soil type, pH and soil management (Passos et al., 2013). Sulfentrazone behaves as a weak acid and presents pKa of 6.56. As the pH of the soil was lower than the pKa of the herbicide, the tendency of the sulfentrazone is to remain mostly in the molecular form, and thus the sorption is favored. Thus, being sorbed to the ground, possible interactions with glyphosate are impaired.

When the dry matter of the aerial part of the sorghum plants was evaluated as a function of the application of sulfentrazone alone and in mixture with glyphosate, a reduction of the dry matter was observed only in the upper layers, confirming that the herbicide was not leached to the lower layers of the soil (Figure 4). If the layer of 45-50 cm is considered as a control, only the 0-5 cm layer showed difference when applied sulfentrazone mixture with Roundup Ready® and Roundup Ultra®. Yet, when sulfentrazone was applied alone or in combination with Zapp Qi®, differences were observed in the 0-5 and 5-10 cm layers (Figure 4).

Similar results were observed in tests performed in Red-Yellow Argisol (Vivian et al., 2006). In this work, the authors evaluated the sulfentrazone leaching potential in isolated application. They verified an evident reduction of aerial part and height dry matter, and increased phytotoxicity

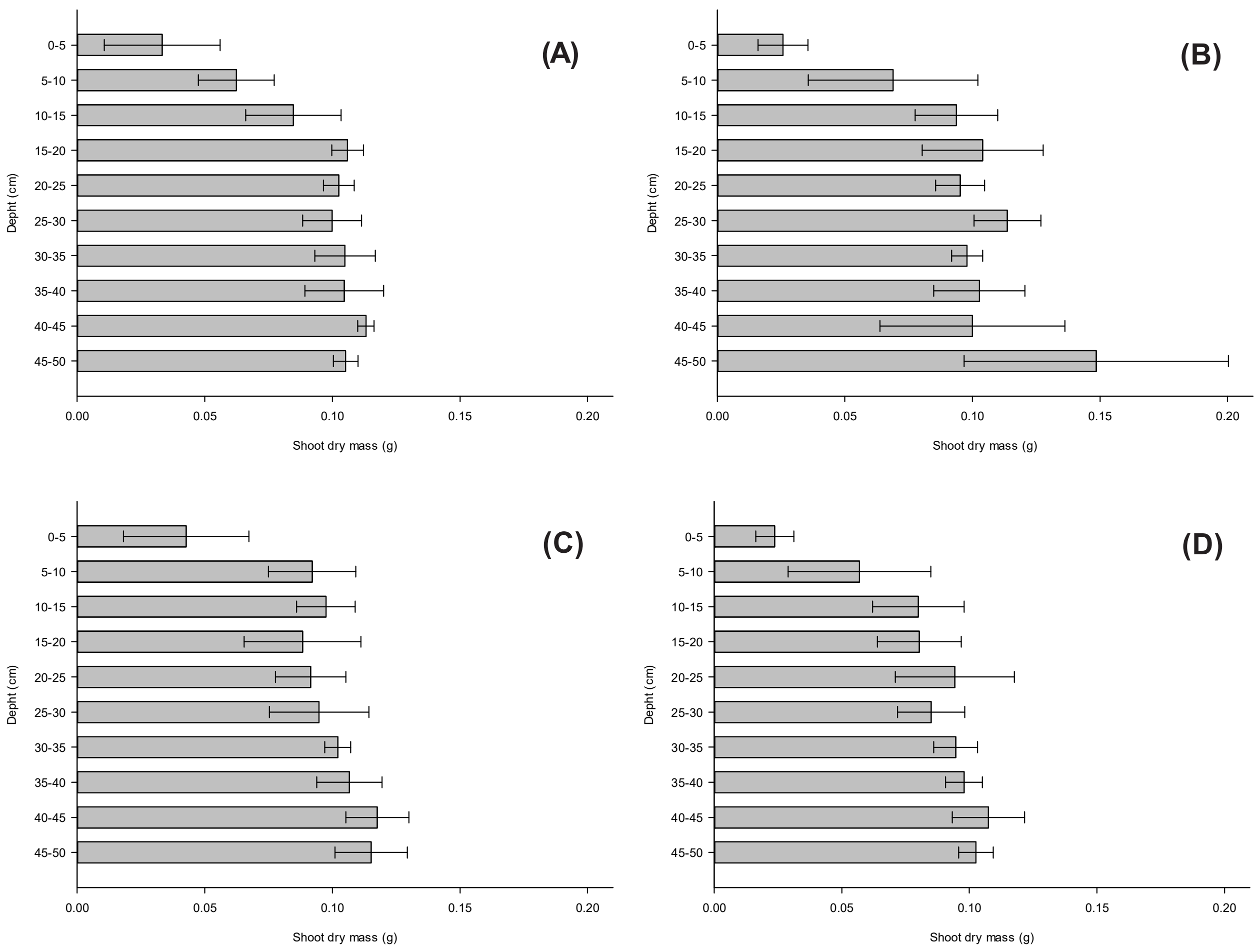

Figure 4 - Dry matter of sorghum aerial part at 21 days after sowing in soil, collected at different depths, treated with sulfentrazone alone (A) and mixed with glyphosate (Roundup Ready (B), Roundup Ultra (C) and Zapp Qi (D)).

indexes in plants grown in soil samples collected at depths of 0 to 10 cm from the soil profile (Vivian et al., 2006).

It is concluded that, in general, glyphosate does not alter the leaching potential of sulfentrazone. However, mixtures with Roundup Ready® and Zapp Qi® can reduce sulfentrazone sorption forces to soil colloids, allowing their desorption to occur more easily, making their availability in the soil solution greater, which may result in better control of the plants and/or intoxication of sensitive crops.

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