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Article

INFLUENCE OF SOIL DEPTH ON SORPTION AND DESORPTION PROCESSES OF HEXAZINONE

Influência da Profundidade do Solo nos Processos de Sorção e Dessorção do Hexazinone

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ABSTRACT - Herbicides with a high leaching capacity, such as hexazinone, tend to reach deeper soils more easily, where retention of the product affects its availability in the soil solution. Therefore, it is important to understand the behavior of hexazinone at a variety of soil depths. The objective of this research was to evaluate the sorption and desorption of hexazinone throughout the soil profile. The sorption and desorption processes of ^{14}C-hexazinone [triazine-6-^{14}C] at three soil depths (0-10, 10-20, and 20-30 cm) were evaluated by the equilibrium batch method, and the radioactivity of the herbicide was quantified by liquid scintillation spectrometry. Five different concentrations of hexazinone (0.12, 0.19, 0.25, 0.31, and 0.38 μg mL^{-1}) in a methanol solvent were used. The concentrations of the herbicide in equilibrium and sorbed in the soil were adjusted by using isotherms according to the Linear, Freundlich, and Langmuir models. Hexazinone sorption at soil depths of 0-10, 10-20, and 20-30 cm showed decreasing Freundlich coefficients (K_f) with values of 0.18, 0.11, and 0.08 g$^{(1-1/n)}$ L$^{1/n}$ kg^{-1} (50.49, 47.58, and 46.37% sorbed), and in the desorption the K_f were 7.96, 7.93, and 9.82 g$^{(1-1/n)}$ L$^{1/n}$ kg^{-1} (26.17, 26.58, and 28.68% desorbed), respectively. The small difference in organic carbon content between soil depths was sufficient to affect hexazinone retention, interfering with the bioavailability of this product in the soil solution for weed control.

Keywords: retention isotherms, mobile herbicide bioavailability.

RESUMO - Herbicidas com grande capacidade de lixiviação, como o hexazinone, tendem a atingir com mais facilidade as camadas mais profundas do solo, afetando a disponibilidade do produto na solução do solo pela retenção. Portanto, é importante compreender o comportamento do hexazinone em uma variedade de profundidades do solo. O objetivo desta pesquisa foi avaliar os processos de sorção e dessorção do hexazinone ao longo do perfil do solo. Os processos de sorção e dessorção do ^{14}C-hexazinone [triazine-6-^{14}C] em três profundidades do solo (0-10, 10-20 e 20-30 cm) foram avaliados pelo método de equilíbrio de batelada em lotes, analisando-se a radioatividade do herbicida foi quantificada por espectrometria de cintilação líquida. Foram utilizadas cinco diferentes concentrações do hexazinone (0,12, 0,19, 0,25; 0,31 e 0,38 μg mL^{-1}) em solvente metanol. As concentrações do herbicida em equilíbrio e sorvidas no solo foram ajustadas usando isotermas de acordo com os modelos Linear, Freundlich e Langmuir. A sorção do hexazinone nas profundidades do solo de 0-0, 10-20 e 20-30 cm apresentou coeficientes de Freundlich (K_f) decrescentes com valores de 0,18, 0,11 e 0,08 g$^{(1-1/n)}$ L$^{1/n}$ kg^{-1} (50,49, 47,58 e 46,37% sorvido); e na dessorção os K_f foram de 7,96, 7,93 e 9,82 mmol$^{(1-1/n)}$ L$^{1/n}$ kg^{-1} (26,17, 26,58 e 28,68% dessorvido),

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respectivamente. A pequena diferença no teor de carbono orgânico entre as profundidades do solo foi suficiente para afetar a retenção do hexazinone, interferindo na biodisponibilidade deste produto na solução do solo para o controle de plantas daninhas.

Palavras-chave: isotermas de retenção, herbicida móvel, biodisponibilidade.

INTRODUCTION

Herbicides applied in agriculture for weed control always reach the soil, regardless of the mode of application. As soon as they come into contact with soil particles, herbicides are subjected to several biotic and abiotic factors, which influence their final destination and their behavior in the environment (Mancuso et al., 2011).

One of the destinations of herbicides is binding to soil particles. This phenomenon, called sorption, is more related to soil organic carbon (OC) than to any other soil property, as is the case for hexazinone (Koskinen et al., 1996). Therefore, understanding the processes of herbicide sorption and desorption in the soil is extremely important, since these processes determine herbicide bioavailability, mobility, and degradation, as well as the possibility of contamination of other environmental compartments. The determinant factors in the sorption and desorption of herbicides in the soil, in general, are the physical, chemical, and biological properties of the soil, the environmental conditions, and the physicochemical properties of the herbicide (Bonfleur et al., 2015; Cha et al., 2016).

Leaching of herbicides in soils with a smaller amount of OC may leave the product exposed to the changes brought about by soil depth, such as changes in pH, microbial activity, and the quantity of organic matter in the soil, especially in the sorption and desorption of products in deeper soils, which depends on attributes such as clay and organic matter content (Taylor et al., 2004). From an environmental point of view, improving the amount of OC below the soil surface is a great option to control the target of herbicides, because in most soils the OC content decreases with increasing soil depth (Gaudinski et al., 2000; Wynn et al., 2005). Corroborating this, the weed seed bank is concentrated in the subsurface layer of the soil and is essential to the study of the product's ability to control weeds (Amim et al., 2016).

Hexazinone [3-cyclohexyl-6-(dimethylamino)-1-(dimethylamino)-1-methyl-1, 3,5-triazine-2-4 (1*H*, 3*H*) diona] belongs to the triazinone group. This herbicide has a residual effect on the soil and is used to control a wide variety of weeds, whether annual, biennial, or perennial. Hexazinone is registered in Brazil for sugar cane crops (Rodrigues and Almeida, 2018) and has a low coefficient of sorption relative to OC (K_{oc} = 54 L kg^{-1}), high persistence in the soil, represented by a high half-life (DT50 = 105 days), and high leaching potential throughout the soil profile, possessing a Groundwater Ubiquity Score (GUS) Index of 4.43 (PPDB, 2019). A study conducted by Koskinen et al. (1996) showed the relationship of soil properties in the sorption and desorption of hexazinone, where the increase in soil depth from 0-5 to 15-20 cm decreased the OC content from 7.63% to 4.27%. Concurrently, the same authors reported that the reduction of OC content directly influenced the values of the sorption coefficient (K_d), which ranged from 1.10 to 0.0632 L kg^{-1} and from K_{oc}, from 25.7 to 8.28 L kg^{-1}, as the soil depth increased, demonstrating that the variation in OC content with depth affected the retention of this product.

To adjust for the process of sorption and desorption of herbicides in the soil, researchers frequently use isotherms that adjust for the concentration of the product in equilibrium in the soil solution and sorbed onto it, in particular, the Linear, Freundlich, and Langmuir isotherms (Green and Karickhoff, 1990).

In view of the above, the objective of this research was to evaluate the sorption and desorption processes of hexazinone throughout the soil profile. The results of this study will help to elucidate the behavior of this mobile herbicide at different soil depths, as comprehension of its bioavailability is necessary for weed control.

MATERIAL AND METHODS

Soil

The soil used was collected in Piracicaba, SP, Brazil (S 22°42’52", W 47°37’10"), classified as Ultisol - Typic Hapludalf, in layers 0-10, 10-20, and 20-30 cm deep, by means of a probe-type auger (model SCTS-40). After drying at room temperature, the samples were sifted through 2 mm mesh and stored at room temperature until analysis. The physicochemical properties of soil samples taken at each depth are shown in Table 1.

Table 1 **-** Physicochemical properties of the different soil depths used in this study

Soil depth (cm)	K	Ca^{2+}	Mg^{2+}	H + Al	BS	CEC
	($mmol_c\ kg^{-1}$)					
0-10	2.7	19	8	11	29.7	40.7
10-20	1.7	18	8	11	27.7	38.7
20-30	2.0	19	7	11	28.0	39.0

	pH ($CaCl_2$)	P ($mg\ dm^{-3}$)	V (%)	OC (%)	Clay	Silt	Sand	Textural class
					($g\ kg^{-1}$)			
0-10	4.8	22	73	0.69	176	39	785	Sandy loam
10-20	5.0	10	72	0.46	203	22	775	
20-30	5.2	7	72	0.40	177	60	764	

K = potassium; Ca = calcium; MG = Magnesium; H + Al: acidity potential; BS = base saturation; CEC = cation exchange capacity; pH = hydrogen potential; P = phosphorus; V = saturation level per base; OC = organic carbon. Source: Department of Soil Science-ESALQ/USP, Piracicaba, SP, Brazil.

Hexazinone herbicide

The ^{14}C-Hexazinone [triazine-6-^{14}C] (radiolabeled), supplied by the company Izotop (Budapest, Hungary), had a radiochemical purity of 99.7% and a specific activity of 3.14 MBq mg^{-1}. An analytical standard (non-radiolabeled) with a chemical purity of 99.9% was acquired from Sigma Aldrich (Saint Louis, MO, USA). A solution with the analytical pattern (radiolabeled and non-radiolabeled) in methanol was prepared carefully using different concentrations of the herbicide (0.12, 0.19, 0.25, 0.31, and 0.38 µg mL^{-1}). The initial radioactivity (~1528 Bq per vial) of the herbicide was verified by placing 1 mL of a sample at each concentration prepared in liquid scintillation vials containing 10 mL of the scintillation solution. The radioactivity of samples in each vial were analyzed in a Liquid Scintillation Counter (LSC) (Tri-Carb 2910 TR LSA, LSA PerkinElmer, Waltham, MA, USA) for 5 minutes.

Sorption and desorption studies

Sorption studies were performed by the batch balancing method, according to the guidelines of the “Organisation for Economic Co-operation and Development – 106” (OECD, 2000). Teflon tubes with 50-mL screw caps were labelled and weighed. Each tube received 5 g of soil and 5 mL of aqueous solution of $CaCl_2$ 0.01 mol L^{-1} containing the herbicide, in a proportion of 1:1 (w v^{-1}). The tubes were prepared in duplicate and subjected to agitation in a horizontal pendular table (Tecnal Equipamentos Scientific, model TE-240, Piracicaba, SP, Brazil) at 200 rpm in a dark environment at 20 °C for 24 hours to equilibrate (Koskinen et al., 1996).

After the equilibration period, the tubes were centrifuged at 3000 rpm for 15 minutes (Hitachi CF16RXII, Hitachi Koki Co., Ltd., Indaiatuba, SP, Brazil) to separate the solution from the soil. After centrifugation, 1-mL aliquots of the supernatant from each tube were collected in duplicate and placed in scintillation flasks containing 10 mL of scintillation solution, and each sample was analyzed by LSC for 5 minutes. The concentration of the herbicide sorbed in the soil was calculated as the difference between the initial concentration of the product and the concentration after the equilibration period.

The desorption study was performed shortly after the sorption study. In the same Teflon tubes, 5 mL of a new aqueous solution of $CaCl_2$ 0.01 mol L^{-1} (non-radioactive) was added, and the old solution was discarded as radioactive waste. The tubes were again equilibrated for 24 hours with agitation on a shaking platform under the same conditions as in the sorption study. The tubes were centrifuged again, and 1 mL aliquots in duplicate supernatants were transferred to scintillation flasks containing 10 mL of scintillation solution for the analysis of sample radioactivity with the herbicide, performed by LSC for 5 minutes per sample. The amount of herbicide desorbed was calculated as the difference in the concentration of the solution before and after desorption.

Sorption and desorption model

The linear sorption coefficients were calculated (K_d, L kg^{-1}) according to the following equation:

$$K_d = \frac{C_s}{C_e}$$

in which C_s is the concentration of herbicide sorbed in the soil (mg kg^{-1}), and C_e is the concentration of the herbicide in equilibrium in the soil solution (mg L^{-1}).

The coefficients of sorption K_f and 1/n were determined by the Freundlich isotherm model:

$$C_s = K_f C_e^{1/n}$$

in which C_s is the amount of herbicide sorbed in the soil (mg kg^{-1}); K_f is the balance constant of Freundlich ($mg^{(1-1/n)}$ $L^{1/n}$ kg^{-1}); C_e is the concentration of the herbicide in equilibrium in the soil solution (mg L^{-1}); and 1/n is the degree of linearity of the isotherm.

The expression of the Langmuir isotherm was represented by the following equation:

$$C_s = \frac{q_{max} K_l C_e}{1 + q_{max} C_e}$$

in which C_s is the concentration of herbicide sorbed in the soil (mg kg^{-1}); q_{max} is the maximum herbicide concentration when the site surface is saturated (mg g^{-1}); K_l is the Langmuir equilibrium constant (L mg^{-1}); and C_e is the concentration of the herbicide in equilibrium in the soil solution (mg L^{-1}).

The sorption coefficients were normalized to the soil OC content (K_{oc}, L kg^{-1}), and the coefficients K_{foc} and K_{loc} were similarly determined as follows:

$$K_{oc} = \frac{K_d}{\%OC} x100$$

The values of K_f and 1/n, K_l and q_{max}, and K_d of desorption were determined in a similar manner to the sorption coefficients, in which the remaining amount of herbicide sorbed in the soil returns to the equilibrium solution, being desorbed. The hysteresis coefficient (H) was calculated by using the parameters that originated from the Freundlich isotherms for sorption ($1/n_{sorption}$) and desorption ($1/n_{desorption}$), with the following formula (Barriuso et al., 1994):

$$H = \frac{1/n_{desorption}}{1/n_{sorption}}$$

Statistical analysis of data

The data obtained in the sorption and desorption studies of hexazinone at different soil depths were expressed as means, followed by the standard deviation of each mean value obtained (*n* = 2). The hexazinone sorption and desorption isotherms of the three models were plotted using Sigma Plot® (version 10.0 for Windows, Systat Software Inc., Point Richmond, CA, USA).

RESULTS AND DISCUSSION

Hexazinone sorption and soil depth

The values of the coefficient of determination (R^2) are close to 1 in all three models of hexazinone sorption isotherms, and this indicates that the estimated curves were able to explain the observed points (Table 2).

Table 2 - Parameters of the sorption isotherms of the Linear, Freundlich and Langmuir model for hexazinone applied at different soil depths

Soil depth (cm)	Linear[1]			Freundlich				Langmuir				Sorption (%)
	K_d	K_{oc}	R^2	K_f	K_{foc}	1/n	R^2	K_l	K_{loc}	q_{max}	R^2	
	(L kg^{-1})			($g^{(1-1/n)}$ $L^{1/n}$ kg^{-1})				(L kg^{-1})		(g kg^{-1})		
0-10	0.20±0.01	28.57	0.99	0.18±0.01	25.71	0.81±0.02	0.99	0.92±0.03	131.420	0.28±0.02	0.99	50.49
10-20	0.13±0.02	28.26	0.99	0.11±0.02	23.91	0.79±0.03	0.99	0.81±0.05	176.90	0.16±0.03	0.99	47.58
20-30	0.08±0.01	19.51	0.99	0.08±0.02	19.51	0.83±0.04	0.99	1.20±0.16	292.90	0.15±0.02	0.98	46.37

[1] Mean value of each parameter, ± standard deviation of the mean ($n = 2$).

The sorption coefficients of the Freundlich (K_f), Linear (K_d), and Langmuir (K_l) models decreased proportionally to the sorption of hexazinone according to the increase in soil depth and with the variation in the amount of OC in it, suggesting that the decrease of OC in the soil reflected a lower retention of hexazinone (Table 1). The three sorption coefficients used obtained values ranging from 0.18 to 0.08 $mg^{(1-1/n)}$ $L^{1/n}$ kg^{-1} for K_f; 0.20 to 0.08 L kg^{-1} for K_d; and 0.92 to 1.20 L kg^{-1} for K_l at soil depths of 0-10, 10-20, and 20-30 cm (Table 2).

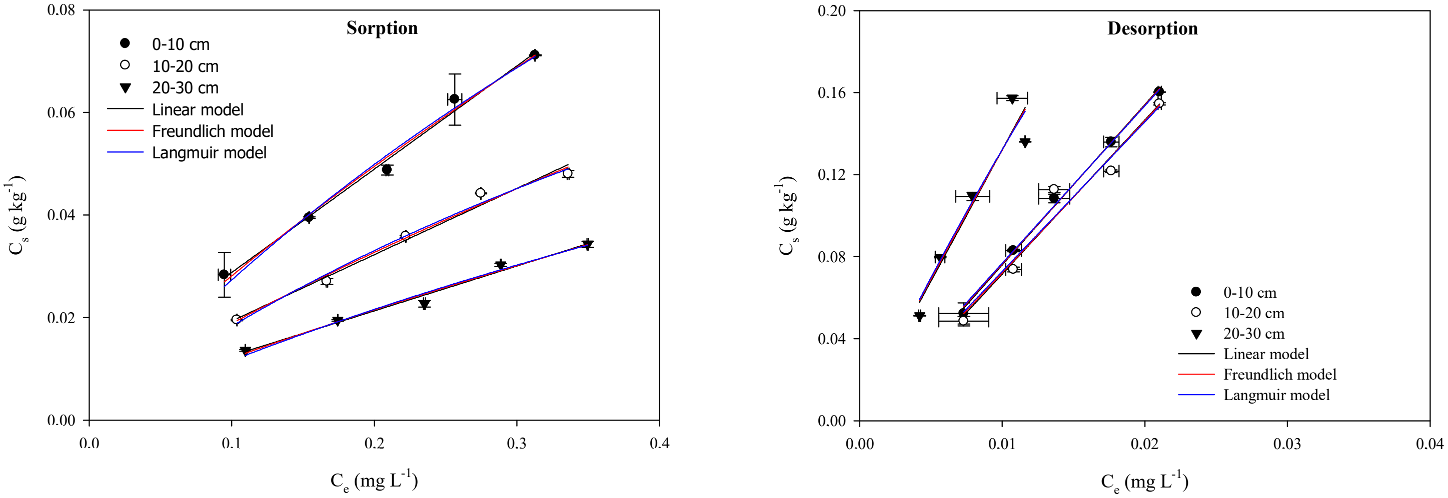
A study conducted by Koskinen et al. (1996), using six types of sandy soils with depths varying from 0-5 to 90-95 cm also demonstrated the relationship between OC and the sorption of hexazinone, obtaining low values of K_d and K_{oc}, which ranged, respectively, from 1.10 to 0.0119 L kg^{-1} and from 25.7 to 20.9 L kg^{-1} at the respective depths of 0-5 and 90-95 cm. Santos et al. (2018) also reported low K_f values of hexazinone, ranging from 0.06 to 0.73 $mg^{1-1/n}$ kg^{-1} $L^{1/n}$ in Brazilian soils. The values obtained in the q_{max} were <0.28 g kg^{-1} (0-10 cm soil depth) (Table 2) and represent the maximum capacity of hexazinone sorption in the different soil profiles.

The coefficients of sorption normalized according to the OC content (K_{loc} and K_{foc}) for hexazinone also decreased proportionally with the increase in depth, varying between 28.57 and 19.51 L kg^{-1} for Kl_{oc} and between 25.71 and 19.51 $mg^{1-1/n}$ kg^{-1} $L^{1/n}$ for K_{foc}. According to Pereira-Junior et al. (2015), in a study using contrasting tropical soils of sugarcane crops, at a depth of 0-20 cm, hexazinone had greater retention (K_f = 0.8 mg^{1-N} L^N kg^{-1}) in soils with higher OC and clay contents when compared with sandy soils (K_f = 0.2 mg^{1-N} L^N kg^{-1}). Mendes et al. (2019) reported that the addition of bovine bone coal to the soil increased the OC content and, consequently, the sorption of hexazinone. Therefore, the results of this study reinforce the importance of soil OC in the sorption of hexazinone, as already evidenced by Oliveira Jr. et al. (2001), Pang et al. (2005), and Sousa et al. (2018).

The sorption isotherms of Freundlich, Linear, and Langmuir for hexazinone at different soil depths are shown in Figure 1. Values of 1/n near 1 in the sorption of hexazinone indicate that the process was not, in its greatest potential, reversible to the solution of the soil (Table 2).

According to the parameters obtained for the sorption isotherms, the rate of reduction in the amount of the product increased by approximately 4.12% from the superficial layer at 0-10 cm to the deepest layer at 20-30 cm, confirming that deeper layers of the soil tend to retain less hexazinone. The percentages of hexazinone sorbed at depths of 0-10, 10-20, and 20-30 cm were 50.49%, 47.58%, and 46.37%, respectively (Table 2). These results indicate that hexazinone is weakly sorbed by the soil, because this herbicide is a weak base and will be poorly laden in the soil, and it seems that hexazinone is sorbed in the soil mainly by means of non-polar mechanisms (Bouchard and Lavy, 1985).

Determination of the leaching rate of hexazinone in the first 30 cm of the profile of Tropical soils (Mendes et al., 2016; Reis et al., 2017) is important for understanding the sorption process in this soil layer, as reported in this study and, consequently, for evaluating the bioavailability of the product in the soil solution for better efficacy of chemical control of weeds in arable areas.



The vertical and horizontal bars represent the standard deviation of the mean (*n* = 2) of the C_e (concentration of equilibrium herbicide in the soil solution) and C_s (concentration of sorbed herbicide in the soil), respectively. The symbols can cover the bars.

Figure 1 - Sorption and desorption isotherms of the Linear (black), Freundlich (red) and Langmuir (blue) model for hexazinone applied at different soil depths.

Hexazinone desorption and soil depth

The values of the desorption coefficients at all soil depths ranged from 7.96 to 9.82 mg $^{(1-1/n)}$ L$^{1/n}$ kg^{-1} for K_f; 7.81 to 12.82 L kg^{-1} for K_d; and from 12.05 to 0.08 L kg^{-1} for K_l (Table 3). Similarly to the non-normalized desorption coefficients, the K_{foc} values were 1137.14 to 2395.12 mg $^{(1-1/n)}$ L$^{1/n}$ kg^{-1}, and K_{oc} from 1115.71 to 3126.90 L kg^{-1}, increasing with soil depth. The values of 1/n in the desorption of hexazinone were also ~1, indicating that the process did not occur to its greatest potential extent (Table 3). These results corroborate those of Pereira-Junior et al. (2015), who found desorption values for hexazinone ranging from 59% to 89% of the product that was sorbed in the soil particles, in agreement with the low hysteresis values found by the same authors of 2.1 to 1.6, directly proportional to the OC content of the soil. A study conducted by Bouchard and Lavy (1985) showed desorption of 82.3%-89.5% of hexazinone initially sorbed in the particles of a sandy soil with OC levels varying between 0.63% and 7.31%, corroborating data from this study suggesting that soils with lower OC levels tend to retain less hexazinone.

Freundlich, Linear, and Langmuir desorption isotherms for hexazinone at different soil depths are shown in Figure 1, and are type C, as in the sorption isotherm (Giles et al., 1974). The type C isotherm describes the relationship between C_e and C_s as constant at any concentration. The use of this type of isotherm is restricted to very small initial concentrations, when one is sure that the solute will not occupy all the sorption sites of the surface (Limousin et al., 2007).

The values of the hysteresis coefficients (H) of hexazinone at depths of 0-10, 10-20, and 20-30 cm were low and close to 1, meaning that the desorption process is, in general, reversible, that is, no hysteresis occurs (Table 3). Hysteresis occurs due to the heterogeneity of the sorption sites of the soil colloid surfaces and, consequently, to the various sorption mechanisms between

Table 3 - Parameters of the desorption isotherms of the Linear, Freundlich and Langmuir model and hysteresis coefficient (H) for hexazinone applied at different soil depths

Soil depth (cm)	Linear[1]			Freundlich					Langmuir				Desorption (%)
	K_d	K_{oc}	R^2	K_f	K_{foc}	1/n	R^2	H	K_l	K_{loc}	q_{max}	R^2	
	(L kg^{-1})			(g $^{(1-1/n)}$ L$^{1/n}$ kg^{-1})					(L kg^{-1})		(g kg^{-1})		
0-10	7.81±0.32	1115.71	0.99	7.96±0.45	1137.14	1.01±0.03	0.99	1.25	12.05±0.82	1721.42	92.74±1.23	0.99	26.17
10-20	7.50±0.41	1630.43	0.98	7.93±0.38	1723.91	1.02±0.04	0.98	1.29	10.32±0.75	2243.50	87.30±1.86	0.98	26.58
20-30	12.82±1.26	3126.90	0.96	9.82±0.64	2395.12	0.93±0.06	0.96	1.12	0.08±0.01	19.51	1.24±0.13	0.96	28.68

(1) Mean value of each parameter, ± standard deviation of the mean (*n* = 2).

the herbicide and the soil. Even in the first phase of sorption, many sites exhibit the characteristic of irreversibility, that is, the molecule is strongly retained in soil colloids, which prevents the desorption process from occurring (Celis and Koskinen, 1999).

The percentages of hexazinone remaining in the soil varied with increasing depth, being 26.17%, 26.58%, and 28.68% at depths of 0-10, 10-20, and 20-30 cm, respectively (Table 3). This demonstrates that more than half of the initially sorbed product returned to the soil solution, again becoming bioavailable for weed control.

The depths of the soil profile interfered in the process of sorption and desorption of hexazinone, because, as the soil depth increased, there was a decrease in the amount of the herbicide and a small increase in the amount desorbed. These results indicate that small differences in OC content between soil depths affect the retention and transport of hexazinone, interfering in the bioavailability of this product in the soil solution for weed control under field conditions. Further research is needed to evaluate the efficacy of control by this herbicide in the seed bank and/or propagules present in the different layers of the soil profile.

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