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Sorption and desorption of Atrazine in horizons of the Red-Yellow Latosol

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Abstract: **Background:** Studies of sorption and desorption of herbicides in soils are essential in predicting their transport, bioavailability, and transformation in the soil profile. These studies are also vital to predict the agronomic efficiency of the herbicides in controlling weeds.

Objective: In this study, atrazine's sorption and desorption kinetics were evaluated in samples composed of single and a mixture of horizons from the Red-Yellow Latosol profile.

Methods: This was performed by the Batch equilibrium method, and the quantification was carried out using a High-performance liquid chromatography (HPLC).

Results: Freundlich isotherms were adjusted appropriately to describe the atrazine sorption. The K_f values obtained varied between 0.188 and 2.592, and the Koc values ranged between 37.76 and 143.81, which corresponded to horizons C and A, respectively. The desorption was only possible to determine in horizon A. The other substrates presented low sorption, and the desorption of the remaining atrazine was not significant, therefore not adjusting to the Freundlich model. The organic matter (OM) was the main attribute to the atrazine sorption in the soil, presenting a high correlation with K_f and Koc values. In the horizon A samples, the K_{fd} value obtained in desorption was higher than the K_f obtained in sorption, which indicated a low desorption capacity and, therefore, low potential mobility of the herbicide in this soil horizon.

Conclusions: It was concluded that the organic matter present in greater quantity in horizon A of the Red-Yellow Latosol has a crucial role in the dynamics of atrazine in the horizons of this soil.

Keywords: Sorption kinetics; Freundlich coefficient; Chromatography

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1. Introduction

Agricultural activities are closely related to a productive model that includes mechanization, the use of fertilizers, and use of synthetic pesticides to control weeds, diseases and pests in crops, leading to higher productivity. Among these substances, since the Green Revolution, herbicides stand out (Rigotto et al., 2014), and in Brazil, they correspond to the most commercialized pesticides (Sindiveg, 2017).

Herbicides are substances used to control weeds. When not controlled at the right time and appropriately, weeds reduce crop productivity (Costa et al., 2018). The chemical method of weed control is cheap, efficient, and allows quick operation, and because of that, it is highly accepted by the crop producers (Martins and Christoffoleti, 2014).

Herbicides are exogenous compounds that, after application, will enter the soil system. Several factors, such as the soil's physical-chemical characteristics, the properties of the herbicide, and the environmental conditions, will influence its final destination in the environment (Silva and Silva, 2007).

In corn, sorghum, and sugarcane crops, atrazine is one of the most used herbicides to control dicotyledonous weeds. It is an old herbicide widely used today in several countries (Ibama, 2018). Atrazine belongs to the triazine group, has a pKa of 1.68, solubility in water of 30 mg L^{-1}, and is soluble in organic compounds such as ether, acetone, benzene, ethanol, and ethyl acetate (ATSDR, 2017).

Atrazine is one of the herbicides with the highest number of reports of contamination in surface and groundwater (Machado et al., 2016; Moreira et al., 2012; Schwab et al., 2006; Soares et al., 2017), and therefore is associated with adverse effects on aquatic biota (Moreira et al., 2014; Wirbisky et al., 2016).

When in contact with the soil, the herbicide atrazine is distributed between aqueous and solid soil phases. Herbicide sorption influences its final destination in the environment, affecting its absorption by plants, its chemicals and biological degradation, its retention in soil, and transport. This transport will be affected due to its relative concentration in the phases (Kovaios et al., 2011).

The colloidal fraction of soils tends to absorb and retain atrazine molecules, modifying this herbicide's degradation and transport processes (Márquez and Hansen, 2014). The bioavailability of herbicides applied to the soil is primarily driven by the dynamic balance between the adsorption processes of the compounds present in the soil colloids and their solubility in the soil-water phase (Takeshita et al., 2018).

The behavior of herbicides in soil depends on the sorption and desorption processes. These processes allow predicting the mobility of herbicides in the soil since sorption influences the leaching processes and control the persistence and irreversible binding of the herbicide. The desorption is related to the release and the mobility of the herbicide in the soil (Peña-Martínez et al., 2018).

As one of the oldest herbicides, there are many studies in the literature on atrazine's behavior in sorptive processes. These are influenced by the physicochemical properties of different types of soils. In Brazil, the primary studies using atrazine were carried out in soils from regions of subtropical climates (Ben-Hur et al., 2003; Martinazzo et al., 2010). Almost all of these studies investigated the behavior of atrazine in the superficial horizon of soils. Therefore, our research aimed to quantify atrazine's sorption and desorption in the different horizons of a Red-Yellow Latosol (RYL) where this herbicide is commonly used. It is believed that the data obtained in this study can contribute to adjustments in the mathematical models used to predict the environmental risks caused by the use of herbicides.

2. Material and Methods

Equilibrium time, sorption, and desorption studies of atrazine were carried out using samples of an RYL, collected in the region of Viçosa-MG, with no previous herbicide application. Three samples were collected at different depths according to the color change in the soil profile, called horizon A (HA), horizon B (HB), and horizon C (HC). In the laboratory, two samples were prepared by mixing the soils. One sample was composed of a mixture of the horizons A and B (HAB), and another was composed of a mixture of the horizons A, B, and C (HABC). The HA horizon corresponded to the topsoil and was collected in a layer between 0 to 5 cm. The HB was collected below the transition layer between HA and HB in a 20 cm layer. The HC was collected below the transition layer between HB and HC in a 20 cm layer. The soils were dried in a greenhouse and then sieved in a 5 mm mesh. The samples composed of the RYL were prepared by mixing a portion of the HA with three portions of the HB to obtain the HAB sample to simulate common collection conditions that corresponded to a depth of 20 cm. The HABC sample was prepared by mixing the same amounts of HA, HB, and HC. The soil samples studied were submitted to textural and physical-chemical analyses (Table 1).

A 1,000 mg L^{-1} of stock solution was prepared using an atrazine PESTANAL® standard (Sigma Chem. Co, Analytical Standard, 98.8%) in acetonitrile (MERCK). 100 mg L^{-1} of the working solution was prepared using the stock solution in a 0.01 mol L^{-1} of $CaCl_2$ solution.

Initially, the equilibrium time was determined in the five substrates using the Batch Equilibrium method (OECD, 2000). From the working solution, 10 mg L^{-1} of atrazine solution in 0.01 mol L^{-1} of $CaCl_2$ was prepared. 10 mL of this atrazine solution and 2 g of soil were added to falcon tubes. The tubes were sealed and placed under vertical agitation using an orbital shaker (Fisatom-801) for different times (0; ½, 1, 2, 4, 8, 12, 16, 24, and 28 hours) at 45 rpm at room temperature (24 ± 2 °C). The tubes containing the samples were then centrifuged at 960 rpm for 6 minutes (SISLab-G2300). After centrifuging the samples, the supernatants' aliquots were collected using a volumetric pipette, filtered in a 0.45 μm Millipore PTFE membrane, and placed in a vial (1.5 mL volume) for further analysis using an HPLC. The equilibrium time is considered the period the concentration of the analyzed solution remained constant.

In the sorption test, from the atrazine 100 mg L^{-1} working solution, solutions with concentration levels of 0.25 were prepared; 0.5; 1.0; 5.0; 10.0; 15.0 and 25.0 mg L^{-1} atrazine. 2 g of soil and 10 mL of these solutions were added in falcon tubes. The tubes were then submitted to vertical agitation for the equilibrium time determined in the previous step. After this agitation, the tubes were centrifuged, and the supernatants were filtrated, under the same conditions used in the equilibrium time test, followed by analysis using HPLC. The amount of atrazine absorbed into the soil (Cs) in mg Kg^{-1} was calculated by the difference between the concentration of the standard solution added to the soil (Cp) in mg L^{-1} and the amount that was found in the equilibrium (Ce) in mg L^{-1}, for each concentration.

Table 1 - Physico-chemical properties of soil samples collected in horizons A (HA), B (HB), and C (HC) and mixtures of samples from horizons A + B (HAB) and A + B + C (HABC) of a Red-Yellow Latosol (LVA).

Horizons	pH	K	P	Mg^{2+}	Ca^{+2}	Al^{3+}	H+Al	CEC	Fe	Mn	OM	Clay	Silt	Sand
		mg dm^{-3}			$cmol_c$ dm^3				mg dm^{-3}			dag kg^{-1}		
HA	5.4	46.0	1.4	0.5	0.9	0.4	5.0	1.9	40.0	15.8	3.1	43.0	12.0	45.0
HB	5.3	36.0	0.6	0.2	0.4	0.2	3.4	0.9	18.0	7.3	1.8	56.0	14.0	30.0
HC	5.0	7.0	0.6	0.0	0.0	1.2	2.3	1.3	8.8	1.5	0.9	5.0	35.0	60.0
HAB	5.3	36.0	0.8	0.3	0.5	0.3	4.1	1.1	22.4	9.6	2.2	56.0	9.0	35.0
HABC	5.3	30.0	0.8	0.3	0.4	0.5	3.8	1.3	25.9	8.2	2.0	37.0	18.0	45.0

OM: Organic matter; CEC: Cation exchange capacity.
Analyzes were carried out at the Soil Analysis Laboratory Viçosa, according to Embrapa (1997).

In the desorption test, the excess supernatant from the substrates in the tubes of the sorption test was discarded. 10 mL of a 0.01 mol L^{-1} of herbicide-free $CaCl_2$ solution was added to the tubes, followed by vertical agitation of the tubes for the equilibrium time. The tubes were centrifuged, and the supernatants were filtered, similarly to the previous step. The filtrate was collected in a vial to be analyzed by HPLC. The amount of atrazine desorbed was determined by the difference between its concentration in the soil before the desorption test and the solution's concentration after the desorption test for each concentration level. The equilibrium time, sorption, and desorption studies were performed in triplicate.

Atrazine quantification was performed using HPLC (model Shimadzu LC 20AT, Japan), equipped with a Photodiode Array Detector (Shimadzu SPD-M20A), and a stainless steel C18 column (Shimadzu VP- ODS Shim- pack 250 mm x 4.6 mm, 5 µm of particle size). The chromatographic conditions were: mobile phase acetonitrile: water (acidified with 0.01% v/v orthophosphoric acid) at a ratio of 50:50, injection volume of 20 µL, flow of 1.0 mL min^{-1} and a wavelength of 254 nm, at 40 °C. An atrazine standard was first injected to identify and determine the retention time. The quantification was performed by the external calibration method, with the preparation of an analytical curve in the concentrations between 0.05 and 30 mg L^{-1}. The limits of detection (LD) and quantification (LQ) were determined based on parameters of the analytical curve and the standard deviation of the controls and were expressed by 3.3x (s/S) and 10x (s/S), respectively, in which s is the standard deviation of the controls and S is the slope of the analytical curve (Anvisa, 2017).

For the interpretation of the results, Sigma Plot 12.0® software was used to construct the sorption isotherms, using the Freundlich equation:

$$Cs = K_f{}^*Ce^{1/n}$$

where Kf is the sorption coefficient, 1/n is the sorption intensity index, Ce is the concentration of atrazine in the equilibrium, and Cs is the amount of atrazine absorbed. The coefficient of sorption was determined regarding the organic carbon content, according to the equation:

$$K_{oc} = \frac{100\ K_f}{foc}$$

where, Koc is the normalized sorption coefficient according to the organic carbon content of the soil (L kg^{-1}). f_{oc} is the soil's organic carbon content, which was calculated by dividing the percentage of organic matter by 1.72 (Silva and Silva, 2007).

3. Results and Discussion

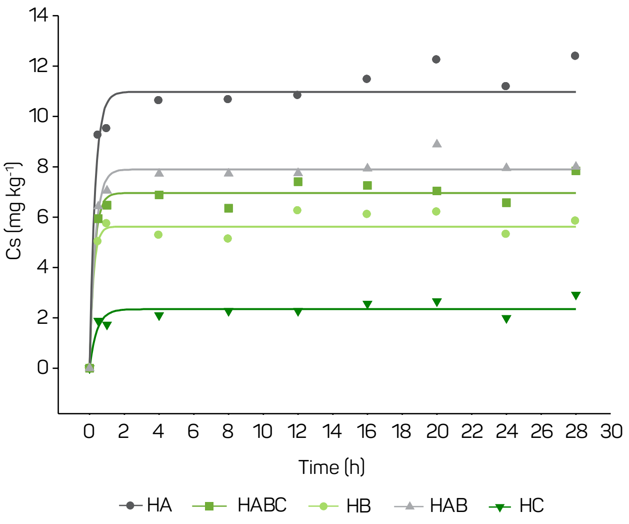
According to the chromatographic analyses, the chosen method proved to be selective for atrazine, as no interferences from the soil matrix extract were observed. The retention time of 7.6 minutes showed linearity according to the coefficient of determination of the analytical curve, which was 0.998. The detection and quantification limits were 0.007 and 0.025 mg L^{-1}, respectively.

The sorption kinetics (Table 2 and Figure 1) was similar in the samples composed of one and more than one soil horizon and occurred in two stages. In the first stage, rapid sorption occurs between two and four hours, followed by a slow step, with the equilibrium reaching six hours. It is suggested that in the first stage, atrazine sorption occurs in easily accessible locations in the soil (Kovaios et al., 2011). In the second stage, the sorption of atrazine becomes slower and occurs in less accessible places.

Table 2 - Equations of atrazine sorption kinetic curves in horizons A (HA), B (HB), and C (HC) and mixtures of samples from horizons A + B (HAB) and A + B + C (HABC) of a Red-Yellow Latosol (LVA).

Horizons	Equation	R^2
HA	$y = 10.9785 * (1 - (e^{-3.2108X})$	0.92
HB	$y = 5.6291 * (1 - (e^{-4.5405X})$	0.92
HC	$y = 2.3492 * (1 - (e^{-2.3645X})$	0.84
HAB	$y = 7.8967 * (1 - (e^{-3.0795X})$	0.97
HABC	$y = 6.9599 * (1 - (e^{-3.6577X})$	0.95

R^2: Determination coefficient.

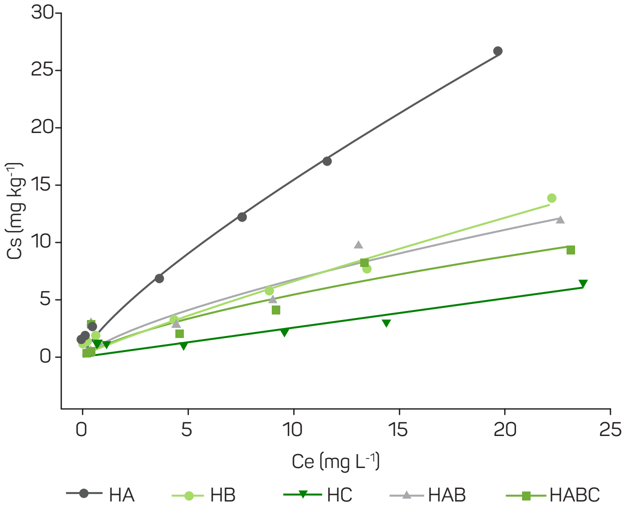


Cs: Concentration of herbicide absorbed in the soil.

Figure 1 - Estimation of atrazine sorption kinetic curves in horizons A (HA), B (HB), and C (HC) and sample mixtures from horizons A + B (HAB) and A + B + C (HABC) of an Oxisol Red-Yellow Latosol (LVA), depending on the stirring time.

For work safety and to guarantee the balance in the five substrates, an equilibrium time of eight hours was established for the samples' sorption and desorption studies. Similar values of equilibrium time (six hours) were found by Queiroz and Lanças (1997), studying soils from different regions of São Paulo and Minas Gerais's states. The behavior in the sorption kinetics of atrazine described by Schmidt et al. (2015) was similar to what was observed in this study. However, the equilibrium times found were higher in a dystrophic Red-Yellow soil submitted to different management systems.

The Freundlich model adjusted appropriately to describe the sorption isotherms (Figure 2) of atrazine in the five horizons of the RYL, obtaining coefficients of determination between 0.85 to 0.98.



Cs: Concentration of herbicide absorbed in the soil; Ce: Concentration of herbicide in equilibrium solution.

Figure 2 - Sorption isotherms of atrazine in horizons A (HA), B (HB), and C (HC) and mixtures of samples from horizons A + B (HAB) and A + B + C (HABC) of a Red-Yellow Latosol (LVA).

The K_f values varied from 0.188 to 2.592 (Table 3), which were similar to the ones obtained by Inoue et al. (2006) in samples of Red Latosol, Red Argisol, and Red Nitossol collected in Mamboré-PR, which varied from 1.47 to 2.10. Archangelo et al. (2005) also reported similar K_f values to those found in this study in dystrophic Red Latosol collected in São Paulo and Minas Gerais's states, which varied between 1.14 to 3.47. After evaluating samples at different soil depths, these authors observed a behavior similar to the present study. As the sampling depth increased, the sorption coefficient decreased. Regarding the normalized sorption coefficients according to the soil's organic carbon content (K_{oc}), values ranging from 37.76 to 143.81 were observed. The highest values were achieved in the HA and HAB that correspond to the superficial layers.

Table 3 - Estimations of the sorption coefficient (Kf) and sorption coefficient normalized by the organic carbon content (Koc) of atrazine in horizons A (HA), B (HB), and C (HC) and sample mixtures in horizons A + B (HAB) and A + B + C (HABC) of a Red-Yellow Latosol (LVA).

Horizons	Equation	Kf	Koc	R^2
		L kg^{1}	L kg^{1}	
HA	$Cs = 2.592 * Ce^{0.7780}$	2.5920	143.81	0.98
HB	$Cs = 0.8612 * Ce^{0.8834}$	0.8612	84.16	0.95
HC	$Cs = 0.1880 * Ce^{1.105}$	0.1880	37.76	0.85
HAB	$Cs = 0.9929 * Ce^{0.810}$	0.9929	78.34	0.96
HABC	$Cs = 0.8344 * Ce^{0.7893}$	0.8344	72.85	0.94

Cs: Concentration of the herbicide absorbed in the soil; Ce: Concentration of the herbicide in the equilibrium solution; R^2: Coefficient of determination.

According to the data obtained, the K_f values followed the increasing order: HC <HABC <HB <HAB <HA. The correlation matrix between K_f of each RYL horizons' sorption and their physical and chemical properties (Table 4) shows a positive correlation between K_f and the OM content. The sorption of atrazine in the soil is related to the OM content that contains active adsorption sites. Higher OM content results in atrazine's higher sorption (Dick et al., 2010; Schmidt et al., 2015).

Table 4 - Correlation matrix between the mean sorption coefficient (Kf) of atrazine, hydrogen potential (pH), organic matter content (OM), and cation exchange capacity (CEC) of the Red-Yellow Latosol (LVA).

	Kf	pH	MO	CTC
Kf	1.00	-	-	-
pH	0.77	1.00	-	-
MO	0.94*	0.91*	1.00	
CTC	0.75	0.26	0.59	1.00

* Significant at 5% by t test (n = 5).

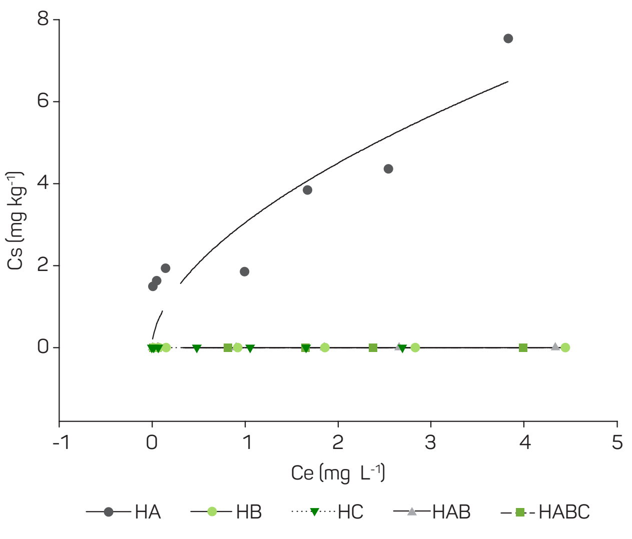
The behavior of sorption isotherms for atrazine in the RYL profile studied is classified as type C. In this type of isotherm, the sorption is linear as the adsorbate concentration increases. New groups are created in the adsorption process allowing the process to continue (Silva and Silva, 2007).

The OM present in the soil can influence various processes (Schmidt et al., 2015). The humic acids present in OM help with atrazine's sorption through the hydrogen bonds, Van der Waals forces, and hydrophobic bonds, which are all involved in herbicide adsorption (Yue et al., 2017).

The low K_f values of the HB, HC, and HAB suggested that the atrazine is highly mobile in these horizons. Variation of K_f values within the soil profile is expected since the soil OM does not present a homogeneous composition and degree of humification. Therefore the OM content changes with the soil profile's depth (Cox et al., 2000). Correia and Langenbach (2006), in a study using Red-Yellow Argisol, observed that around 75% of atrazine was distributed in the 5 cm surface of the soil and that 15% was gradually distributed and decreasing up to 25 cm in depth. This demonstrates the critical role of OM in the sorption processes. However, the authors have not ruled out the possibility of returning the herbicide to the solution, which can provide leaching if the process forces are weak. In fact, in the same study, leaching was evaluated up to 90 days after the herbicide application. The atrazine was found 50 cm deep, but the authors believe it could have reached greater depths.

The overturning of the soil during soil preparation for conventional planting or large-scale crops and atrazine application promotes the destruction of the surface layer. This can lead to the infiltration and percolation of water in the soil profile (Pinheiro et al., 2011). In this way, the precipitation would directly influence the herbicide's transport between the soil profile's horizons and reach the groundwater.

The Freundlich model (Figure 3 and Table 5) describes desorption behavior in the HA with an adjustment of 0.76. It was not possible to quantify the desorption in the other soils. The K_f value of desorption was higher than the K_f of sorption (Table 3), which suggests that the herbicide return to the soil solution is difficult. The strong interaction between atrazine and organic matter (Abate et al., 2004) indicates less desorption capacity and low mobility of the herbicide in this soil horizon.



Cs: Concentration of herbicide absorbed in the soil; Ce: Concentration of herbicide in equilibrium solution).

Figure 3 - Atrazine desorption isotherms in horizons A (HA), B (HB), and C (HC) and mixtures of samples from horizons A + B (HAB) and A + B + C (HABC) of a Red-Yellow Latosol (LVA).

Table 5 - Estimation of the desorption coefficients (Kfd) of atrazine in the A (HA), B (HB), and C (HC) horizons and sample mixes from the A + B (HAB) and A + B + C (HABC) horizons Red-Yellow Latosol (LVA).

Horizons	**Equation**	**Kfd**	**R^2**
		L kg^1	
HA	$Cs = 3.0442 * Ce^{0.5638}$	3.044	0.76
HB	$Cs = \mu = 1.470$	ND	-
HC	$Cs = \mu = 0.852$	ND	-
HAB	$Cs = \mu = 1.411$	ND	-
HABC	$Cs = \mu = 1.278$	ND	-

Cs: Concentration of the herbicide absorbed in the soil; Ce: Concentration of the herbicide in the equilibrium solution, μ average value; ND: not determined; R2: Coefficient of determination.

It was not possible to quantify the atrazine desorption for the other horizons, which can be attributed to the low values of K_f in the sorption test. In these cases, as the sorption was low, the desorption of the remaining atrazine in the study was not significant, regardless of the concentration. This led to the lack of adjustment of the isotherm to the Freundlich model. However, according to Mamy and Barriuso (2007), the time of exposure of the soil to the herbicide can influence the determination of sorption and desorption. Regarding desorption, it tends to decrease by increasing the contact time between the soil and the herbicide due to the diffusion processes in the soil (Alletto et al., 2010).

4. Conclusions

Each layer that composes the soil profile presents different physical, chemical, and biological characteristics. This can lead to atrazine leaching in the soil profile, with consequent contamination of groundwater, as the atrazine's sorting capacity is different between the horizons in the soil profile. In the studied RYL, the following increasing order of sorption was observed; HC < HABC < HB < HAB < HA, and the organic matter is one of the factors that mostly contributes to the atrazine sorption. The HA of the RYL profile plays an essential role in the desorption of atrazine due to its ability to retain organic matter.

Author' contributions

ARAP, MSMJ, NMEM: Preparation and conduct of the experiment in the laboratory. GAMP, CFL, AAS: Planning, guidance, and correction of the written material of the experiment.

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