

## SORPTION AND DESORPTION BEHAVIOR OF HERBICIDE CLOMAZONE IN SOILS FROM BRAZIL

### COMPORTAMENTO DE SORÇÃO E DESSORÇÃO DO CLOMAZONE EM SOLOS DO BRASIL

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**ABSTRACT:** The knowledge of the processes that affect soil herbicide behavior allows to developing practices that improve efficiency in chemical weed control and reduce the negative impacts of cropping-systems. In this research, we used HPLC to determine sorption and desorption of clomazone in Brazilian soil samples (Red-Yellow Latosol with liming and without liming, Red latosol with and without liming, and Organosol). To study clomazone sorption, we used the batch equilibrium method. Clomazone concentrations in soil samples were determined by HPLC. Desorption was quantitated using the samples contained in tubes after sorption testing, repeating the steps followed to quantify sorption. The Freundlich isotherm adequately describe clomazone sorption in all soils. Variation in pH in Red-Yellow Latosol and Red Latosol did not change clomazone sorption. The values of the Freundlich constant (Kf) indicate that clomazone sorption is higher in Organosol, followed by Red latosol (with liming) = Red latosol (without liming) > Red-Yellow Latosol (with liming) = Red-Yellow Latosol (without liming). We conclude that among the attributes evaluated, soil organic matter has the highest impact on clomazone sorption. There was a direct relationship between Kf values and organic matter content of the substrates. Desorption of clomazone was very low in the evaluated soils, with the exception of Organosol, where quantification of the herbicide was possible.

**KEYWORDS:** Herbicides. Soil behavior. Environmental impact. HPLC.

### INTRODUCTION

Chemical weed control is essential for large-scale agriculture. However, it is crucial that the used herbicides are only recommended after evaluation of their interactions with soil colloids in ensure a high quality of the harvested product and the natural resources that sustain production, especially soil and water (SILVA et al, 2013). The use of pre-emergent herbicides with residual activity in the crop management operation has become a common practice to reduce the initial competition exerted by weeds. The application of these products has as main target the soil. Upon reaching the ground, soil-applied herbicides are redistributed and degraded this process can be short, as in the case of simple molecules which are not persistent or last for months or years in the case of highly persistent compounds. Residence time of herbicides in the environment depends, among other factors, on the sorption capacity of the soil, the dynamics of fluid flow, and the solute transport in addition to degradation rates. In areas with extensive and excessive herbicide use, environmental contamination may occur (CUMMING et al, 2002;

UMILJENDIĆ et al, 2013). Therefore, it is crucial to obtain more information on the dynamics of herbicides in the soil to improve herbicide efficacy and reduce the environmental problems caused using these products. Knowledge of the processes to which the molecules of herbicides are subject to after contact with the ground is critical to predict the herbicide's efficacy and the fate of these substances in the environment (SILVA et al, 2013).

Soil-herbicide interactions alter product availability in the soil solution and are governed by the retention processes of these compounds (GEVAO et al, 2000). The sorption process influences herbicide absorption by plant roots and dissipation processes such as biological degradation, volatilization, leaching, and transport by water erosion (HERWIG et al, 2001; GAVRILESCU, 2005). Sorption (the fractions of the herbicide connected to the soil) and desorption (percentage of the product sorbed which returns to the soil solution) are processes that influence leaching, persistence, and activity of the compound in the environment (YU et al, 2006). These processes can be described by isotherms regarding concentrations in the solution and in the solid soil phase.

The sorption isotherm describes the thermodynamic relationship between water activity and the equilibrium of the moisture content of a soil product at constant temperature and pressure. The knowledge and understanding of sorption isotherms is highly important in soil science and technology for the predictions of quality, stability, and half-life.

The extent to which this phenomenon occurs reflects the reversibility of the sorption process. In some cases, this can be very high, in others, sorption is almost irreversible (hysteresis (H)).

Clomazone [2-(2-Chlorobenzyl)-4,4-dimethyl-1,2-oxazolidin-3-one] is an herbicide from the chemical group isoxazolidinone. Widely used in weed control in crops of mono- and eudicotyledons such as cotton, rice, potatoes, sugarcane, tobacco, and cassava, is used both as pre-emergent and post-emergent. Clomazone is a non-ionizable herbicide and therefore remains in its molecular form in the soil solution (CAO et al, 2013). However, non-ionic herbicides may be polar and therefore affected by pH and complexes adsorbed to clay minerals and soil organic matter (SILVA et al, 2013). Organic matter is the primary attribute of the soil influence the sorption of clomazone (UMILJENDIĆ et al, 2013; GUNASEKARA et al, 2009). However, its role is dependent on its concentration in the soil and chemical composition can vary significantly and

thus influence the nature and extent of herbicide sorption (ĐUROVIĆ-PEJČEV et al, 2008).

Considering the wide range of agricultural land in Brazil, studies of the clomazone sorption Brazilian soils are crucial for obtaining technical recommendations to ensure the agronomic efficiency of this herbicide along with low environmental risks for different soils and climates in Brazil. The aim of this study was to evaluate sorption and desorption capacity of clomazone in three Brazilian soils with different characteristics, using high-performance liquid chromatography

## MATERIAL AND METHODS

### Soil Samples

The experiment was conducted with three soils from distinct Brazilian regions: Red-Yellow Latosol from the state of Tocantins, Red Latosol from the state of Minas Gerais, and Organosol from the state of Espírito Santo. Each Latosol sample was divided into two parts: One sample was limed to reach a soil pH in the neutral range (Table 1). Soil samples were collected from the surface layer (at a depth of 0.0 to 0.20 m), dried in the shade (25°C), sieved through a 2-mm mesh and subsequently characterized chemically and physically (Tables 1 and 2). Acidity correction was performed by acid neutralization curves adding CaCO<sub>3</sub> samples.

**Table 1.** Results of the chemical analysis of substrate samples A (Red-Yellow Latosol, pH = 6.5), B (Red-Yellow Latosol, pH = 5.7), C (Red Latosol, pH = 6.5), D (Red Latosol, pH = 5.1), and E (Organosol, pH = 5.32)

Substrate	pH	K	Ca	Mg	Al	H + Al	SB <sup>(a)</sup>	(t)	OM <sup>(b)</sup>
	H <sub>2</sub> O	cmol <sub>c</sub> kg <sup>-1</sup>							%
A	6.50	79	1.30	0.70	0.0	4.50	33.50	2.30	3.0
B	5.70	79	0.99	0.52	0.0	4.70	26.70	1.71	3.0
C	6.50	9	0.34	0.15	0.0	2.52	60.00	0.79	2.1
D	5.10	9	0.04	0.06	1.6	5.30	2.90	1.66	2.1
E	5.32	185	5.10	3.00	0.6	26.64	31.00	9.17	20.2

<sup>(a)</sup>Sum of bases; <sup>(b)</sup>Organic matter.

**Table 2.** Results of the physical analysis of substrate samples A (Red-Yellow Latosol, pH = 6.5), B (Red-Yellow Latosol, pH = 5.7), C (Red Latosol, pH = 6.5), D (Red Latosol, pH = 5.1) and E (Organosol, pH = 5.32).

Substrate*	Sand %	Silt %	Clay %
A	55	30	39
B	55	30	39
C	21	10	62
D	21	10	62
E	34	20	36

\*Analyses carried out in the Laboratory of Analysis of Soil Viçosa, according to the methodology of the Empresa Brasileira de Pesquisa Agropecuária-EMBRAPA (Brazilian Agricultural Research Corporation).

### Herbicide, Chemicals and Sorption equilibrium time

Determination of the sorption equilibrium time of clomazone by soil colloids was performed using the batch equilibrium method. For this, a solution containing 2.5 mg L<sup>-1</sup> clomazone obtained from a stock-solution of 1.000 mg L<sup>-1</sup> clomazone Pestanal® (Sigma Chem. Co., Standard Analytical 99.7% purity) in CaCl<sub>2</sub> 0.01 mol L<sup>-1</sup> was prepared. Then, 10 mL of this solution and 2.0 g of each soil sample were added to polypropylene tubes. The tubes were properly sealed and placed vertically under agitation for different time periods (0.0, 0.5, 1.0, 2.0, 4.0, 8.0, 12.0, 16.0, 24.0, and 28.0 hours) at room temperature (25 ± 2°C). After agitation, samples were centrifuged at 2,260 x g (3,500 rpm) for seven minutes. Part of the supernatant was filtered through a Millipore filter with a 0.45 µm Politetrafluoretileno (PTFE) membrane and placed directly in a bottle with a capacity of 0.0015 L for subsequent analysis by high-performance liquid chromatography (HPLC). For this experiment, we considered that equilibration time of the analyzed concentration of the solution remained constant.

### Sorption/Desorption Experiments

Sorption analysis was performed using working solutions from the same stock-solution at concentrations of 0.5, 1.0, 2.5, 5.0, and 10.0 mg L<sup>-1</sup> of clomazone in 0.01 mol L<sup>-1</sup> CaCl<sub>2</sub>. We added 0.01 L of these solutions to tubes containing 2.0 g of soil. Then, the tubes were subjected to vertical stirring at room temperature to determine equilibration time. After agitation, samples were centrifuged at 2,260 x g (3,500 rpm) for seven minutes. A portion of the supernatant was filtered through a 0.45 µm membrane and transferred into 0.0015 L vials for HPLC analysis.

Desorption was performed immediately after sorption at all points of the sorption isotherms. The adsorption from the supernatant was completely removed and we subsequently added CaCl<sub>2</sub> 0.01 mol L<sup>-1</sup> of free herbicide until reaching the initial root weight. All replacement volumes were evaluated by weighing on a precision scale (0.1 mg) and ranged from 0.077 to 0.094 L. The vials were agitated to disperse the agglomerated soil and the suspensions were shaken mechanically for the determined equilibration time at room temperature. The suspensions were then centrifuged at 3500 rpm (for 7 minutes) and the supernatants were filtered and analyzed by HPLC. The amount of herbicide still sorbed in the soil in each desorption phase was calculated as the difference between the quantity of sorbed and desorbed herbicide.

### Clomazone Extraction and Analysis

Quantification of clomazone was carried out using a liquid chromatograph with high efficiency (Shimadzu LC 20AT, Japan model), a photodiode array detector (Shimadzu SPD- M20A), and a stainless steel C18 column (Shimadzu VP-ODS Shim-pack 250 mm x 4.6 mm ID, 5 µm particle size). The chromatographic conditions were a mobile phase of acetonitrile: water (acidified with 0.01% v/v orthophosphoric acid) at a ratio of 50:50, injection volume of 20 µL, flow of 0.0012 L min<sup>-1</sup> wavelength of 194 nm, and a temperature of 30°C column. Under these conditions, the retention time for clomazone was approximately 10 minutes. Comparison of retention time of the sample with an analytical standard clomazone allowed herbicide identification, quantification was determined by the external calibration method.

### Data Analysis

The Freundlich isotherm is used to describe this phenomenon, the empirical adjustments  $C_s$  and  $C_e$  refer to herbicide concentrations that are sorbed to the soil and the concentration remaining in solution after the equilibration period, respectively. This adjustment produces  $K_f$  coefficients and  $1/n$ . The Freundlich coefficient ( $K_f$ ) indicates the strength of the sorption while  $1/n$  indicates the nonlinearity of the sorption and desorption isotherm according to Equation 1 (OLIVEIRA, 2002).

$$C_s = K_f * C_e^{1/n} \quad (1)$$

The concentration of sorbed herbicide ( $C_s$ ) in mg kg<sup>-1</sup> was calculated from the difference between the standard solutions initially added to the soil ( $C_p$ ) in mg L<sup>-1</sup> and the amount found in the equilibrium solution ( $C_e$ ) in mg L<sup>-1</sup>. The Freundlich equation was used to interpret the results of sorption and desorption based on the values of  $C_s$  and  $C_e$ .

The method for determining sorption and desorption was validated according to parameter selectivity, linearity, limit of detection, and quantification. Linearity was determined using seven concentrations in triplicate from 0.05 to 1.1 mg L<sup>-1</sup> CaCl<sub>2</sub> 0.01 mol L<sup>-1</sup>. Selectivity was assessed by assaying the supernatants resulting from the agitation of soil with 0.01 L of CaCl<sub>2</sub> solution 0.01 mol L<sup>-1</sup> for the presence of free clomazone.

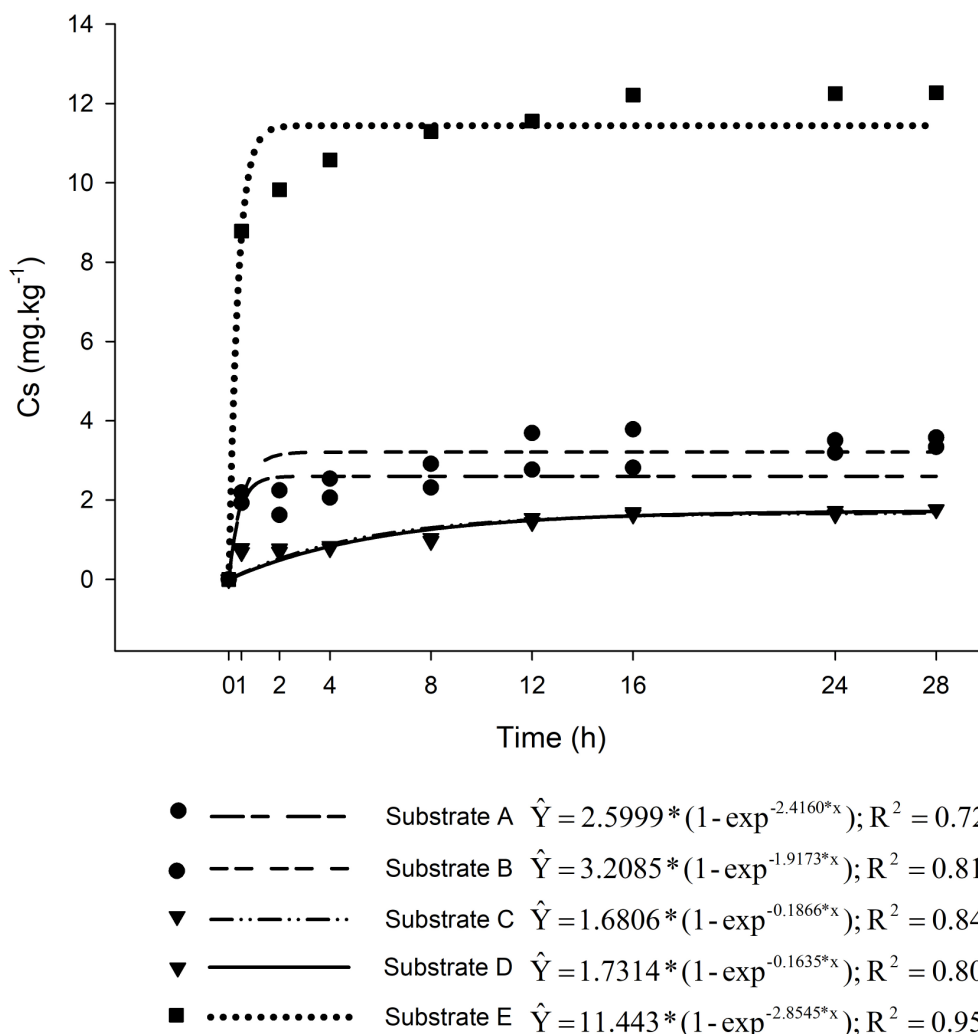
Limit of detection (LD) and limit of quantification (LQ) were determined using the method based on the analytical curve parameters, where these parameters are expressed by  $3.3 \times (s/S)$  and  $10 \times (s/S)$ , respectively, where 's' is the estimated standard deviation of the response, which can be the estimate of the standard linear equation coefficient, and 'S' is the inclination or slope of the

calibration curve deviation (RIBANI et al, 2004). Pearson correlations were also performed between the studied attributes and  $K_f$  observed. In addition, we compared the estimates of the  $K_f$  parameter values with their respective confidence intervals, using the R software (Version 0.99.896 – © 2009-2016 RStudio, Inc.) with the help of the package 'nlme'.

## RESULTS AND DISCUSSION

### Sorption equilibrium time

The sorption of clomazone reached equilibrium after eight hours of agitation for some soils (Figure 1). However, the substrates were stirred for 12 hours to ensure equilibration.



**Figure 1.** Kinetic curve of clomazone sorption in the substrate A (Red-Yellow Latosol, pH = 6.5), B (Red-Yellow Latosol, pH = 5.7), C (Red Latosol, pH = 6.5), D (Red Latosol, pH = 5.1) and E (Organosol, pH = 5.32).

In all substrates, sorption kinetics were characterized by two phases: rapid sorption at the initial stage, the first four hours, followed by a slower phase (PASSOS et al. 2013). Also observed the for atrazine and 2,4-D, reporting that 90% of the compounds sipped the first four hours under stirring, followed by a slower subsequent sorption and an equilibrium. This result can be explained by the fact that in the first hour, many sorption sites on the ground and in the next phase are available, albeit the

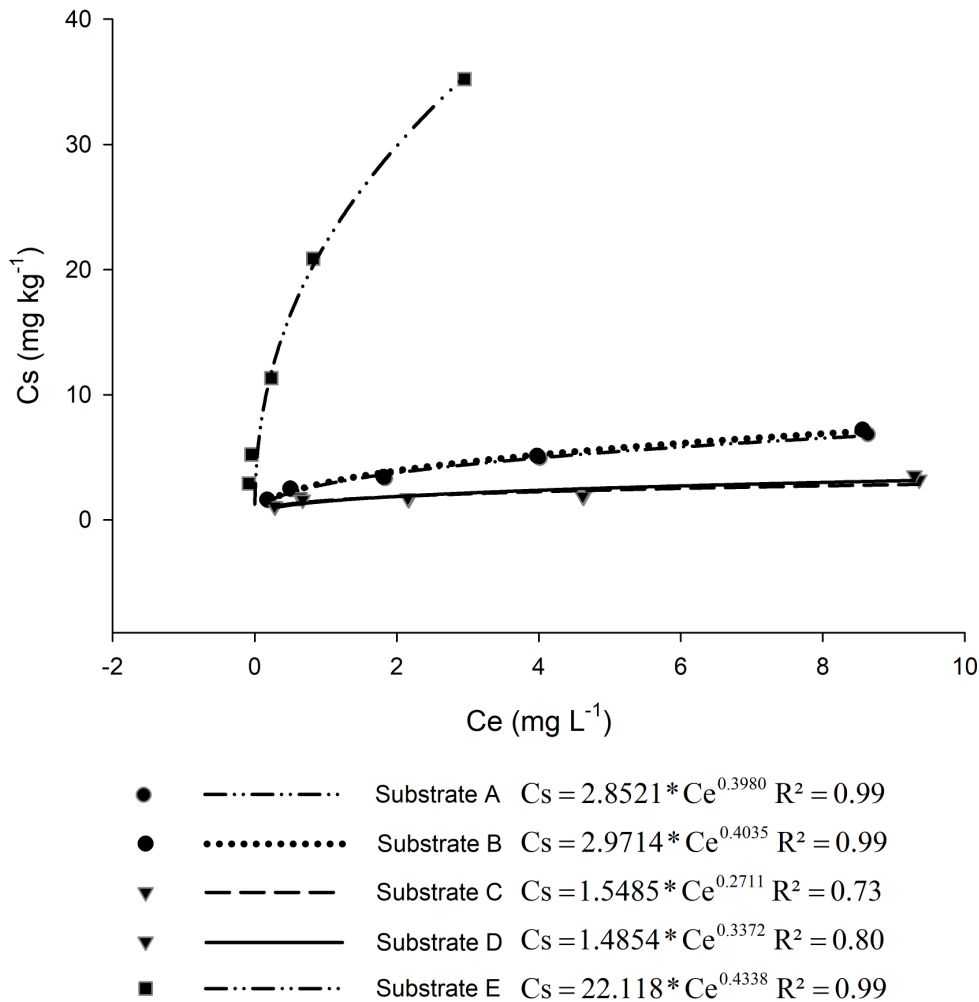
vacant surface sites are difficult to be occupied due to the repulsive forces between the molecules of the solute in the solid phase and molecules in solution (LIU et al, 2010; ROCHA et al, 2013).

The method is selective because there are not interfering in the retention time of the analyte and obtaining good linearity ( $y = 118883x + 5687.8$ ) with a coefficient of determination of 0.99. The limits of detection and quantification were 0.35 and 1.07 mg kg<sup>-1</sup>, respectively.

**Sorption Isotherm**

The Freundlich isotherm was adjusted appropriately to describe sorption of clomazone to the substrates studied, as indicated by the

determination coefficients of the equations ( $r^2 = 0.73$  to  $0.99$ ) (Figure 2). The  $K_f$  values ranged from 1.48 to 22.12 (Table 3).



**Figure 2.** Clomazone of sorption isotherms in the substrate A (Red-Yellow Latosol, pH = 6.5), B (Red-Yellow Latosol, pH = 5.7), C (Red Latosol, pH = 6.5), D (Red Latosol, pH = 5.1), and E (Organosol, pH = 5.32).

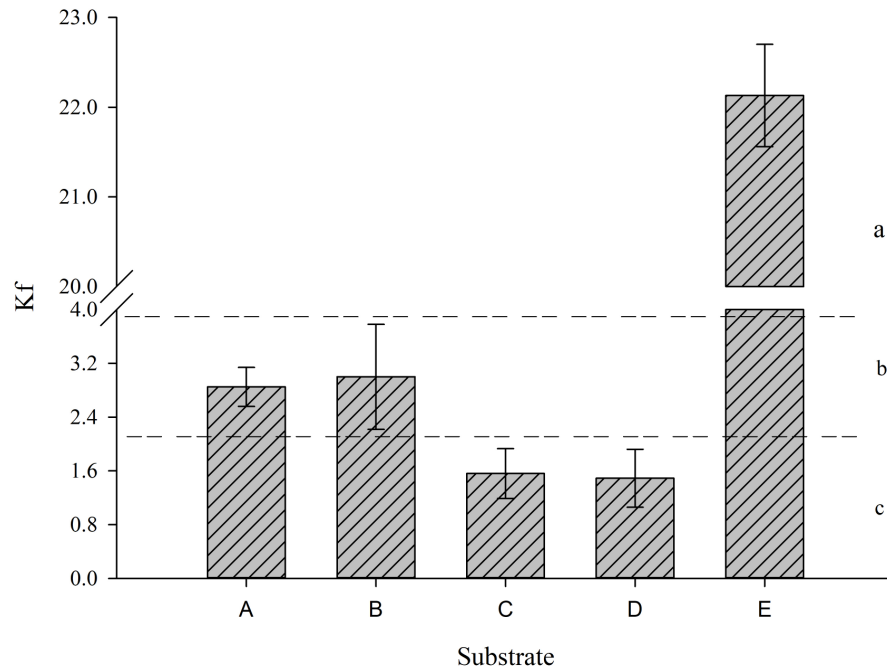
**Table 3.** Parameters of clomazone sorption of Freundlich equation in the substrate A (Red-Yellow Latosol, pH = 6.5), B (Red-Yellow Latosol, pH = 5.7), C (Red Latosol, pH = 6.5), D (Red Latosol, pH = 5.1) and E (Organosol, pH = 5.32).

Parameters	Substrate				
	A	B	C	D	E
$K_f$	2.8521	2.9714	1.5485	1.4854	22.118
$1/n$	0.3980	0.4035	0.2711	0.3372	0.4338
$R^2$	0.99	0.99	0.73	0.80	0.99

The pH variations in Latosols did not change the clomazone sorption (Figure3). The  $K_f$  values confirm that clomazone was more sorbed in Organosol, followed by Red-Yellow Latosol (pH = 5.7) = Red-Yellow Latosol (pH = 6.5) > Red Latosol (pH = 6.5) = Red Latosol (pH = 5.1). Organic matter content and soil CTC decreased in the same order, confirming the results of the correlation between the attributes of substrates and  $K_f$  values (Table 4). The other attributes were not correlated with the  $K_f$  values.

Organic matter contents of latosols were smaller to the Organosol, and clomazone sorption was lower in latosols (Figure 3 and Table 4). These results can be explained by the influence of organic matter on the sorption of non-ionic molecules, such

as clomazone (UMILJENDIĆ et al, 2013; BENOIT et al, 2008). Several studies demonstrated a direct relationship between clomazone sorption and soil organic matter (UMILJENDIĆ et al, 2013; BENOIT et al, 2008; SILVA et al, 2012).



**Figure 3.** Parameter estimates "Kf" and its confidence intervals for the substrates A (Red-Yellow Latosol, pH = 6.5), B (Red-Yellow Latosol, pH = 5.7), C (Red Latosol, pH = 6.5), D (Red Latosol, pH = 5.1) and E (Organosol, pH = 5.32). Confidence intervals separated by dashed lines and identified by different lower case letters are statistically different.

**Table 4.** Correlations between soil (organic matter, clay, silt, sand, pH and CTC) and Kf values in the substrate A (Red-Yellow Latosol, pH = 6.5), B (Red-Yellow Latosol, pH = 5.7), C (Red Latosol, pH = 6.5), D (Red Latosol, pH = 5.1) and E (Organosol, pH = 5.32).

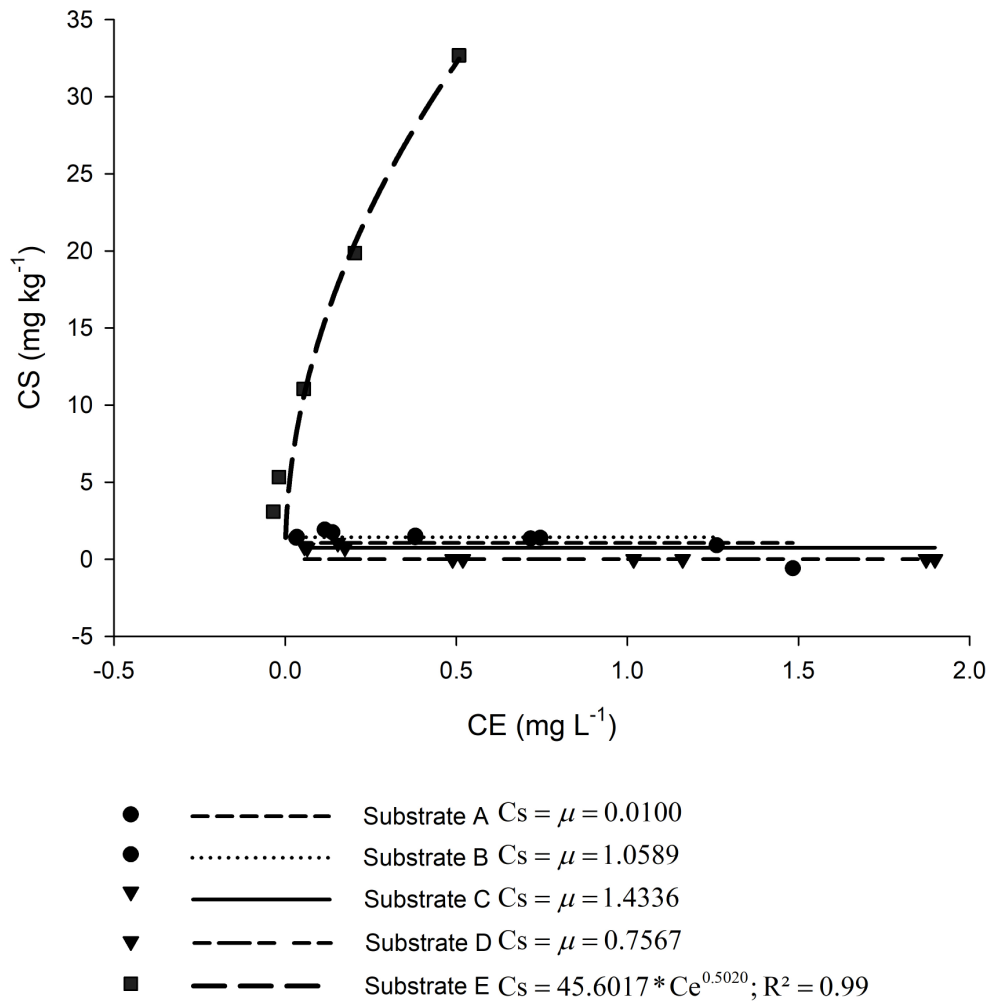
	OM	Clay	Silt	Sand	pH	CTC	Kf
OM	1	-0.54	0.06	-0.05	-0.41	0.99**	0.99**
Clay		1	-0.87**	-0.81**	0.01	-0.58	-0.56
Silt			1	0.99	0.23	0.11	0.08
Sand				1	0.27	0.01	-0.03
pH					1	-0.44	-0.41
CTC						1	0.99**
Kf							1

\*\* significance 1% error probability.

**Desorption Isotherm**

The Kf value of desorption for Organosol (with higher organic matter contents) was higher than Kf or desorption (Figure 4) indicating that desorption was hindered by the strong interaction between organic matter and clomazone (VIEIRA et

al, 1999). Several soil characteristics can affect herbicide sorption with organic matter, such as the source material, predominant types of minerals in the clay fraction, the presence of saturating ions of functional groups of organic matters, and specific ions (SILVA et al, 2013).



**Figure 4.** Desorption isotherms of clomazone in substrate A (Red-Yellow Latosol, pH = 6.5), B (Red-Yellow Latosol, pH = 5.7), C (Red Latosol, pH = 6.5), D (Red Latosol, pH = 5.1), and E (Organosol, pH = 5.32)

Clomazone desorption for other substrates could not be quantified, possibly to the low Kf values observed in these substrates. As sorption was too low, the desorption of the remaining clomazone, irrespective of the concentration used in this study, was not significant, which prevented the use of the Freundlich isotherm model.

## CONCLUSION

Organosol presents higher sorption capacity of clomazone. The results confirm the importance of knowledge of the physical and chemical

characteristics of the soil, especially the organic matter content, before recommending the use of the herbicide clomazone. Information on the soil characteristics is crucial to make safe recommendations from an agronomic and environmental point of view.

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**RESUMO:** O conhecimento dos processos que afetam o comportamento de herbicidas no solo permite desenvolver práticas que melhorem a eficiência no controle químico de plantas daninhas e reduzam os impactos negativos do seu uso em sistemas agrícolas. Nesta pesquisa, utilizando a cromatografia líquida de alta eficiência, foram determinadas a sorção e dessorção do clomazone em amostras de solos do Brasil (Latosolo VermelhoAmarelo com e sem calagem,

Latossolo Vermelho com e sem calagem, e Organossolo). Para o estudo da sorção do clomazone foi utilizado o método Batch Equilibrium. As concentrações do clomazone nas soluções das amostras dos solos foram determinadas por cromatografia líquida de alta eficiência após centrifugação e filtração das soluções. A dessorção foi quantificada utilizando as amostras contidas nos tubos, após os ensaios de sorção repetindo-se os passos seguidos para quantificar a sorção. A isoterma de Freundlich ajustou-se adequadamente para descrever a sorção do clomazone em todos os solos estudados. As variações de pH nos Latossolo Vermelho-Amarelo e Latossolo Vermelho não alteraram a sorção do clomazone. Os valores de Kf indicam que o clomazone é mais sorvido no Organossolo seguindo a sorção em ordem decrescente para Latossolo Vermelho-Amarelo, Latossolo Vermelho-Amarelo (com calagem), Latossolo Vermelho e Latossolo Vermelho (com calagem). Concluiu-se que dentre os atributos dos solos avaliados a matéria orgânica foi o que mais influenciou a sorção do clomazone. Observou-se relação direta entre valores de Kf e teores de matéria orgânica nos substratos. A dessorção do clomazone foi muito baixa nos solos avaliados, com exceção do Organossolo, onde foi possível a quantificação do herbicida.

**PALAVRAS-CHAVES:** Herbicidas. Comportamento do solo. Impactos ambientais. HPLC.

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