

Sorption and Desorption of Cyantraniliprole in Soils with Different Attributes

Sorção e Dessorção de Cyantraniliprole em Solos com Diferentes Atributos

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The applications of insecticides without knowledge of its interactions with the soil solution, that contains humic substances and colloids, can result in serious environmental problems. Cyantraniliprole is an anthranilic diamide insecticide used in seed treatment and therefore can come into direct contact with the soil that can be leached to groundwater. This could result in negative impacts on human and animal health, as well as a reduction in biodiversity. Thus, this work studied the sorption equilibrium of cyantraniliprole in Brazilian soils with different attributes. The sorption percentage was low in soils such as Red-Yellow Latosol and Red Latosol textures (% < 45). Besides, the values of K_f was low ($K_f < 24$). These results suggest the high leaching potential of the insecticide due to low contents of active sites in the soils evaluated.

Keywords: Adsorption isotherms; brazilian soils; HPLC analysis; insecticides.

1. Introduction

Insecticides are extensively used in agriculture to increase the productivity of various crops, protecting them from diseases caused by pests.¹ The main mode of insecticide action on target insect is to block a particular nervous or muscular response pathway, causing the death of the organism.² The Diamides chemical group was introduced to the market at the end of the 1990s.³ The innovative mechanism of these compounds action⁴ has led to their widespread usage and the development of a research studies variety concerning this chemical class.

The main effect of a Diamide on the target insect is inhibition of the correct functioning of the calcium channel.⁵ This disturbance causes a rapid and massive release of calcium ions present in intracellular deposits, causing interruption of feeding, lethargy, paralysis, and finally death of the insect.⁶ The Diamides group includes the second generation diamides known as anthranilic diamides, one of which is the insecticide Cyantraniliprole (Figure 1).

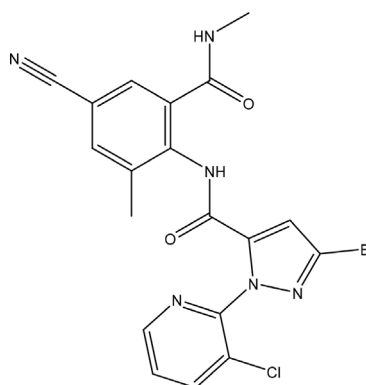


Figure 1. Chemical structure of the cyantraniliprole molecule

Cyantraniliprole ($C_{19}H_{14}BrClN_6O_2$) has been widely used due to its high potential for the control of sucking and chewing insects. It is marketed in the form of a concentrated suspension and is indicated for the treatment of crop seeds. Its chemical name, according to the International Union of Pure and Applied Chemistry (IUPAC), is 3-bromo-1-(3-chloro-

Table 1. Physicochemical properties of cyantraniliprole

Diamide group	Molar mass (g mol ⁻¹)	Log P (20 °C, pH = 7.0)	pKa	Henry's constant (Pa m ³ mol ⁻¹) at 25 °C	Vapor pressure (mPa) at 20 °C	Solubility in water (mg L ⁻¹) at 20 °C
Cyantraniliprole	473.72	2.02	8.8	1.7x10 ⁻¹³	5.13x10 ⁻¹²	14.2

Source: ⁸

2-pyridinyl)-N-[4-cyano-2-methyl-6-(methylcarbamoyl)phenyl]-1H-pyrazole-5-carboxamide.⁷ The physicochemical properties of cyantraniliprole are described in Table 1.

There is a scarcity of information concerning the interactions of Diamides with the soil, considering the effects of edaphological factors and the Physicochemical properties of the insecticide.⁸⁻⁹ Molecules that are weakly sorbed can leach into the soil profile and can contaminate ground and surface waters. Therefore, the mobility and bioavailability of pesticides in the soil are directly related to the capacity of sorption and desorption. The knowledge of these interactions can assist in obtaining information about the effects on ecosystems, as well as on human health.¹¹ The way to obtain this essential information is to stipulate the soil-pesticide interaction, determining the sorption and degradation coefficients, enabling prediction of the movement of the pesticide (in this case cyantraniliprole) through the soil profile.

The half-life of Cyantraniliprole in soil is shorter than 20 days.¹² Work in Spain¹³ obtained a similar half-life of less than 30 days ($DT_{50} < 30$), with Cyantraniliprole therefore being considered a non-persistent compound, while a normalized sorption coefficient (K_{oc}) of about 300 L kg⁻¹ was indicative of the potential for leaching. A study undertaken in Russia¹⁴ indicated that Cyantraniliprole migrated through the soil profile, while recent work in China¹⁵ detected residues of Cyantraniliprole and its main metabolite (J9Z38) in samples of artificial soil and earthworms.

However, there is a scarcity of information concerning the dynamics of Cyantraniliprole in Brazilian soils, which hinders the establishment of concentration levels for residues of this compound in the environment. Therefore, the aim of the present work was to evaluate the sorption and desorption of Cyantraniliprole in soils with different attributes.

2. Materials and Methods

2.1. Solutions and reagents

Acetonitrile (HPLC grade, CAS 75-05-8) was purchased from J.T. Baker Chemical Co. (Phillipsburg, NJ, USA). Methanol (HPLC grade, CAS 67-56-1) was purchased from Merck (Darmstadt, Germany). Calcium Chloride (analytical grade, CAS 10043-52-4) was obtained from ACS (Indaiatuba, São Paulo, Brazil). A Cyantraniliprole standard (95.90% purity, CAS 736994-63-1) was purchased

from Sigma-Aldrich (St. Louis, MO, USA). A commercial product (Fortenza 600 FS[®]) containing 48.80% of the active agent was also obtained.

A stock standard solution of Cyantraniliprole (100 mg L⁻¹) was prepared in Acetonitrile and stored in a freezer at -12 °C. A stock solution of the commercial product containing the cyantraniliprole active agent was prepared at the same concentration (100 mg L⁻¹). Working solutions were obtained by diluting the stock solutions (standard and commercial product) in aqueous 0.01 mol L⁻¹ Calcium Chloride (CaCl₂) solution. All the solutions employed type 1 water obtained from a Milli-Q system (Millipore, Bedford, MA, USA).

2.2. Analytical method

Cyantraniliprole was quantified by high performance Liquid Chromatography (HPLC), using a CBM-20A instrument (Shimadzu, Tokyo, Japan) equipped with high pressure reciprocating pumps (LC-10AD VP), a column oven (CTO-10AS VP) operated at 25 °C, an autoinjector (SIL-10AF) set at 20 µL, a C18 column (Shimadzu VP-ODS, 150 x 4.6 mm, 5 µm), and a UV/Vis detector (SPD-20A) set at 270 nm. The mobile phase was acetonitrile/water (60:40 v/v), at a flow rate of 0.80 mL min⁻¹. The total run time was 7.0 min.

Analytical curves were constructed using the Cyantraniliprole standard or the commercial product containing cyantraniliprole as the active ingredient. The solutions were prepared at concentrations from 0.50 to 10.00 mg L⁻¹.

The analytical resolution (Ar) was determined using Equation 1. This parameter corresponds to the smallest difference of the analyte concentration that can be observed using the analytical curve, where a lower Ar value indicates better quality of the method.¹⁴⁻¹⁵

$$Ar = \frac{s_{res}}{S} \quad (1)$$

where, s_{res} is the estimated residual standard deviation and S is the analytical sensitivity.

2.3. Soils

One soil sample (sandy loam texture) was collected in the municipality of Ponte Nova (Minas Gerais state, Brazil), at coordinates 20°24'16.3"S 42°48'49.5"W. Two other soil samples were collected in Viçosa (Minas Gerais state, Brazil), at coordinates 20°45'47.6"S 42°50'24.7"W

(Viçosa₁, clay texture) and 20°46'11.2"S 42°52'09.3"W (Viçosa₂, clay texture). After collection, any roots were removed from the soils, followed by homogenization, sieving through a 2 mm sieve, and air-drying for 24 h at room temperature (25°C±2). The soils were then submitted to textural and physicochemical analyses (Table 2 and 3).

2.4. Adsorption equilibrium time evaluation

The equilibrium times for Cyantraniliprole adsorption in the soils were determined according to the batch equilibrium method described by the Organization for Economic Cooperation and Development (OECD).¹⁹ In these assays, 2.00 g portions of soil were weighed out into 50.00 mL Falcon tubes and fortified with 10.00 mL of CaCl₂ solution (0.01 mol L⁻¹) containing Cyantraniliprole at 1.5 mg L⁻¹. The tubes were then submitted to vertical agitation, using an orbital shaker at 40 rpm, for periods of 0.5, 1, 2, 4, 8, 12, 18, and 24 h, at a temperature of 24 ± 2 °C. After each time, tubes were removed and centrifuged (model G2300, SISlab) for 7 min at 3500 rpm. Aliquots of the supernatants were collected, filtered through 0.45 µm PTFE membranes, and submitted to analysis by HPLC-UV/Vis.

2.5. Sorption

The sorption assays were performed separately for each soil, according to the OECD method.¹⁹ For this, masses of 2.00 g of the soils were weighed out into 50.00 mL Falcon tubes and fortified with 10.00 mL volumes of solutions containing the commercial Cyantraniliprole product at different concentrations (1.00, 2.00, 4.00, 6.00, and 8.00 mg L⁻¹). The tubes were then submitted to vertical agitation for 6 h at 40 rpm and 24 ± 2 °C, followed by centrifugation for 7 min at 3500 rpm. Aliquots of the

supernatants were collected, filtered through 0.45 µm PTFE membranes, and analyzed by HPLC-UV/Vis.

The amount of Cyantraniliprole adsorbed in the soil (C_s, in mg kg⁻¹) was calculated as the difference between the initial concentration of the analyte added to the soil and the concentration quantified in the equilibrium solution (C_e, in mg L⁻¹).

2.6. Desorption

After the sorption assay, the supernatant in each Falcon tube was discarded, followed by addition of 10.00 mL of CaCl₂ solution (0.01 mol L⁻¹) without Cyantraniliprole. The tubes were then submitted to agitation, centrifugation (7 min at 3500 rpm), and filtration (0.45 µm PTFE membrane), as described above. The amount of Cyantraniliprole desorbed was determined as the difference between the amount sorbed in the soil (C_s) and the concentration determined in the supernatant collected after the desorption assay.

2.7. Sorption and desorption models

The data obtained in the sorption and desorption assays were fitted using the Langmuir (Equation 2), Freundlich (Equation 3), and linear (Equation 4) mathematical models.²⁰ These models enabled evaluation of the behavior of the insecticide present at different concentrations in the soils.

$$[C_s] = [C_m] \frac{K_1 \times C_e}{1 + K_1 C_e} \quad (2)$$

$$[C_s] = K_f [C_e]^{1/n} \quad (3)$$

$$K_d = \frac{C_s}{C_e} \quad (4)$$

Table 2. Chemical analyses of the soils collected in the municipalities of Viçosa and Ponte Nova (Minas Gerais state, Brazil)

Soil	pH	P	K	Ca ²⁺	Mg ²⁺	Al ³⁺	H+Al	BS	t	T	V	m	OM
	H ₂ O	--mg dm ⁻³ --									-----%		dag kg ⁻¹
Viçosa ₁	4.88	1.00	4.00	0.27	0.06	0.00	2.30	0.34	0.34	2.64	12.90	0.00	0.66
Ponte Nova	6.57	36.50	90.00	3.65	1.42	0.00	1.30	5.30	5.30	6.60	80.30	0.20	1.61
Viçosa ₂	5.35	1.90	100.00	2.01	0.59	0.19	4.80	2.86	3.05	7.66	37.30	6.20	3.09

Analyses carried out at the Viçosa Soil Analysis Laboratory, according to the methodology of the Brazilian Agricultural Research Corporation (EMBRAPA, 1997).¹⁸ BS: base saturation; t: effective cation exchange capacity; T: total cation exchange capacity; V: base saturation index; m: Al³⁺ saturation; OM: organic matter.

Table 3. Physical analyses of the soils collected in the municipalities of Viçosa and Ponte Nova (Minas Gerais state, Brazil)

		Clay	Silt	Sand	Textural class
		-----%			
Viçosa ₁	Red-Vermelho Latosol	71.00	71.00	25.70	Clayey
Ponte Nova	Vermelho Latosol	17.40	17.40	76.10	Sandy loam
Viçosa ₂	Red-Vermelho Latosol	57.20	57.20	35.00	Clayey

Analyses carried out at the Viçosa Soil Analysis Laboratory, according to the methodology of the Brazilian Agricultural Research Corporation (EMBRAPA, 1997).¹⁸ BS: base saturation; t: effective cation exchange capacity; T: total cation exchange capacity; V: base saturation index; m: Al³⁺ saturation; OM: organic matter.

where, C_s is the amount of Cyantraniliprole sorbed in the soil (mg kg^{-1}); C_e is the equilibrium concentration of Cyantraniliprole in the soil solution (mg mL^{-1}); $1/n$ indicates the degree of linearity of the isotherm; K_l is the Langmuir model sorption coefficient; K_f is the Freundlich model sorption constant; and K_d is the linear model sorption coefficient.

The sorption coefficient was determined from the organic carbon content using Equation 5.

$$K_{oc} = \frac{100 \times K_f}{foc} \quad (5)$$

where, K_{oc} is the sorption coefficient normalized according to the soil organic carbon (OC) content (L kg^{-1}), and foc is the soil organic carbon content. The latter was calculated by dividing the percentage of organic matter by 1.72.²¹

The Langmuir (K_l) and Freundlich (K_f) coefficients were also normalized according to the OC content, obtaining the values of the coefficients K_{loc} (Equation 6) and K_{foc} (Equation 7), respectively.

$$K_{loc} = \frac{K_l}{OC} \times 100 \quad (6)$$

$$K_{foc} = \frac{K_f}{OC} \times 100 \quad (7)$$

The hysteresis coefficient, H (Equation 8), was determined using the sorption and desorption parameters derived from the Freundlich mathematical model.²⁰⁻²¹

$$H = \frac{1/n_{desorption}}{1/n_{sorption}} \quad (8)$$

3. Results and Discussion

The retention time of cyantraniliprole ($t_R = 3.8 \text{ min}$) in the HPLC analysis was determined by the injection of working standards at different concentrations in the range from 0.50 to 10.00 mg L^{-1} (Figure 2).

The analytical curve presented satisfactory linearity ($R^2 = 0.998$), with sensitivity of 0.046 mg L^{-1} and resolution of $7.14 \times 10^{-4} \text{ mg L}^{-1}$. The Limits of Detection (LoD) and Quantitation (LoQ) were 0.15 and 0.48 mg L^{-1} , respectively. The quantification of the active agent in the commercial product was performed by the external calibration method.

3.1. Adsorption equilibrium time

The Cyantraniliprole sorption kinetics were similar for the three different soils (Figure 3 and Table 4). An initial adsorption stage occurred in the first 2 h, followed by slower adsorption, with equilibrium reached after 4 h. During the

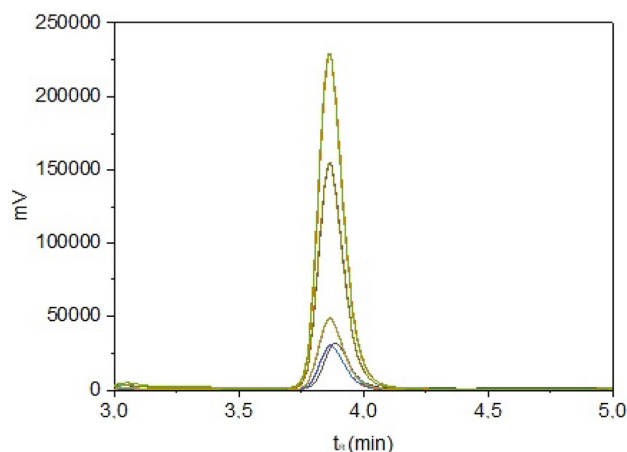


Figure 2. Chromatograms of Cyantraniliprole by HPLC analysis. Monitored wavelength = 270 nm

first stage, sorption of Cyantraniliprole occurred on easily accessible active sites in the soil, while slower sorption during the second stage could be explained by fewer accessible active sites. In order to ensure sufficient time for equilibrium to be reached in the three soils, a time of 6 h was established in the subsequent assays.²²⁻²⁴

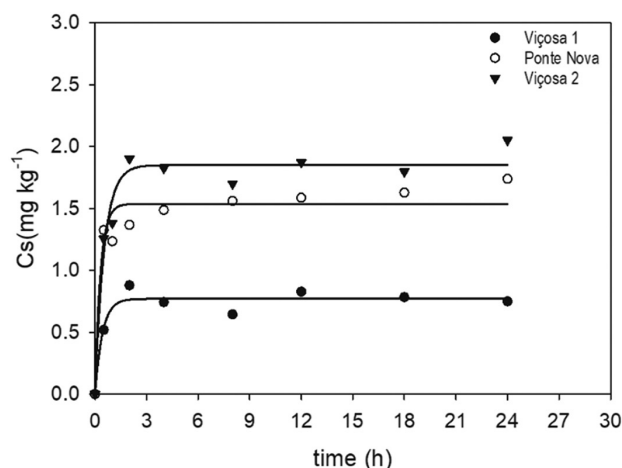


Figure 3. Kinetic curves for sorption of cyantraniliprole in the different soils: (●) Viçosa₁, (○) Ponte Nova, and (▼) Viçosa₂

3.2. Sorption

The parameters obtained for each model are presented in Table 5. Positive linear relationships ($R^2 > 0.99$) were observed between the OM content and K_d and K_f , demonstrating that a greater amount of soil organic carbon (OC) led to higher linear and Freundlich sorption constants. The OC content is the most important factor influencing the adsorption of pesticides in the soil. The OC value is directly proportional to OM, so higher OM should be reflected by higher OC, and, consequently, higher K_d . The K_{oc} values for the Ponte Nova and Viçosa₂ soils were below 300 L kg^{-1} (292.36 and 182.45 L kg^{-1} , respectively), indicating substantial mobility of cyantraniliprole in the

Table 4. Equations of the curves for sorption of Cyantraniliprole in the different soils (Ponte Nova, Viçosa₁, and Viçosa₂)

Soils	Equation	R ²	
Viçosa ₁	$y = 0.7696(1 - e^{-2.3469})$	0.9689	(9)
Ponte Nova	$y = 1.5373(1 - e^{-3.1549})$	0.9650	(10)
Viçosa ₂	$y = 1.8508(1 - e^{-1.8862})$	0.9797	(11)

soil, so it could be considered a potential environmental contaminant.²⁰ Similar results were indicated about Cyantraniliprole in soil.¹⁴

The K_{oc} value is the sorption coefficient normalized according to the organic carbon (OC) content of the soil, as shown in Equation 5. The K_{oc} value was higher for the Viçosa₁ soil (572.21 L kg⁻¹), compared to the Viçosa₂ soil (182.45 L kg⁻¹), even though the two soils had the same textural classification (clayey). Since Cyantraniliprole is a neutral species, it would be expected to partition preferentially in the organic matter, rather than associate with the charged groups of the clay present in the soil. The sorption percentages obtained for the Viçosa₁, Ponte Nova, and Viçosa₂ soils were 33.02, 42.60, and 44.11%, respectively. These results suggested the potential for substantial percolation of the compound through the soil, which could allow it to reach the groundwater.

When the soil pH is lower than the pKa of the pesticide, the molecule is in its neutral form. The pH values for the Viçosa₁ and Viçosa₂ soils were 4.88 and 5.35, respectively, which were both lower than the Cyantraniliprole pKa value of 8.8. In its neutral form, the interaction of Cyantraniliprole with the organic matter present in the soil is more pronounced, due to the partitioning process, formation of charge transfer complexes, and the establishment of hydrogen bonds between cyantraniliprole and organic matter. Electrostatic interaction with clay particles is favored when the chemical species is charged. As shown in Table 2, the organic matter content was higher for the Viçosa₂ soil (3.09 dag kg⁻¹), compared to the Viçosa₁ soil (0.66 dag kg⁻¹). Therefore, the greater amount of organic matter was probably responsible for favoring the sorption of cyantraniliprole in the Viçosa₂ soil.

Values of K_f from 0 to 24 are considered low,²⁶ which means that for the soils evaluated in this work, the retention capacities for Cyantraniliprole were low. These results

suggested low contents of active sites in the soils evaluated, with high potential for leaching of Cyantraniliprole.

The linear coefficient values ($1/n$) were less than 1, between 0.55 and 0.63, indicating that the sorption intensity was not directly proportional to increase of the Cyantraniliprole concentration.

The results obtained in this work were similar to those reported by Vela¹³ and could assist in predicting the environmental behavior of Cyantraniliprole, classified as a potential leacher. Strategies may be required to minimize risks to human health if this compound accumulates in the soil and reaches underground water bodies by leaching.

The isotherms obtained by application of the linear, Freundlich, and Langmuir models for sorption of cyantraniliprole in the soils from Viçosa and Ponte Nova are shown in Figures 4A, 4B, and 4C, respectively.

3.3. Desorption

The parameters obtained for each model are presented in Table 6. The isotherms showed good fits, with positive linear relationships for all the models ($R^2 > 0.99$).

The Freundlich isotherm model provided the best fits to the sorption and desorption results. The $1/n$ parameters obtained for the soils enabled determination of the hysteresis index (H) values. This parameter provides an indication of the amount of the compound sorbed by the soil that could be released into solution,²¹⁻²² which may be reversible or irreversible. A value of $H < 0.7$ indicates a reversible process, while for $0.7 < H < 1.0$, the phenomenon is inconclusive. A value of $H > 1.0$ indicates favorable sorption. In the present case, the hysteresis observed for the Viçosa₁ soil ($H = 0.59$) classified the process as being reversible, while the Ponte Nova soil showed no hysteresis ($H = 1.27$) and the Viçosa₂ soil was classified as inconclusive ($H = 0.89$).

Table 5. Parameters of the linear, Freundlich, and Langmuir isotherm models applied to the sorption of Cyantraniliprole in the clayey and sandy loam soils

Soil	Linear			Freundlich				Langmuir				Sorption (%)
	K _d	K _{oc}	R ²	K _f	K _{foc}	1/n	R ²	K _i	K _{loc}	q _{max}	R ²	
	(L kg ⁻¹)			mg ^(1-1/n) L ^(1/n) kg ⁻¹				(L kg ⁻¹)		(g kg ⁻¹)		
Viçosa ₁	2.20	572.21	0.9997	0.61	159.13	0.93	0.9995	77.16	20108.68	193.01	0.9991	33.02±0.25
Ponte Nova	2.74	292.36	0.9997	4.15	443.28	0.48	0.9997	21.80	2328.46	85.66	0.9982	42.60±0.20
Viçosa ₂	3.28	182.45	0.9996	4.48	249.44	0.71	0.9991	29.42	1637.76	125.99	0.9980	44.11±0.24

$1/n$: degree of linearity of the isotherm; K_l : Langmuir sorption coefficient; K_f : Freundlich sorption coefficient; K_d : linear sorption coefficient; K_{oc} : sorption coefficient normalized according to the organic carbon (OC) content of the soil (L kg⁻¹); foc : soil organic carbon content. The Langmuir (K_l) and Freundlich (K_f) coefficients were also normalized according to OC, where K_{loc} and K_{foc} are the normalized Langmuir and Freundlich coefficients, respectively.

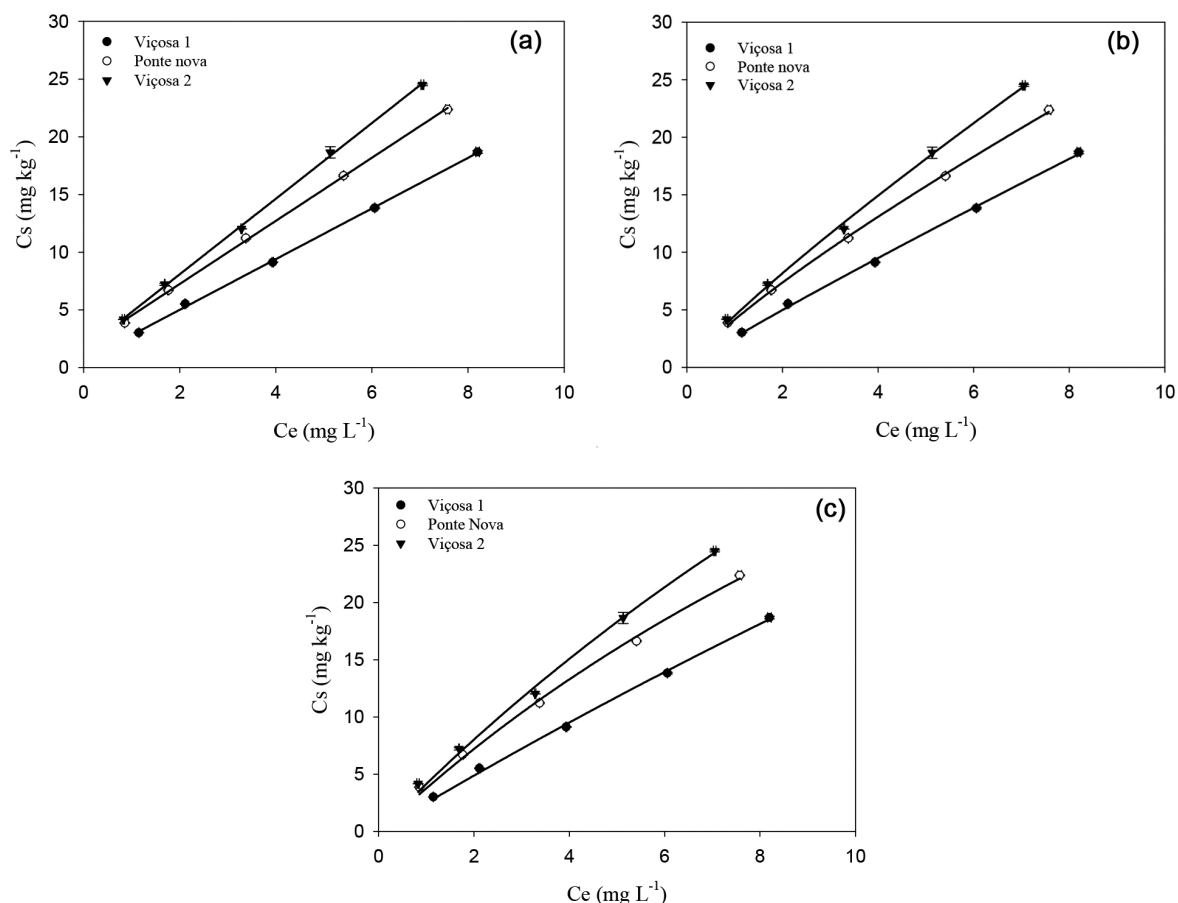


Figure 4. Isotherms for the sorption of Cyantraniliprole in the different soils, obtained using the linear (A), Freundlich (B), and Langmuir (C) models

Table 6. Parameters obtained using the linear, Freundlich, and Langmuir isotherm models applied to the desorption behavior of Cyantraniliprole in the clayey and sandy loam soils

Soil	Linear		R ²	Freundlich					Langmuir			Desorption (%)	
	K _d	K _{OC}		K _f	K _{foc}	1/n	R ²	H	K _l	K _{loc}	q _{max}		
	(L kg ⁻¹)			mg ^(1-1/n) L ^(1/n) kg ⁻¹					(L kg ⁻¹)		(g kg ⁻¹)		
Viçosa ₁	4.91	1276.97	0.9959	7.60	1980.87	0.55	0.9772	0.59	1.35	352.55	3.70	0.9446	44.91±0.29
Ponte Nova	22.47	2400.12	0.9971	22.51	2404.90	0.61	0.9766	1.27	0.86	92.03	2.80	0.9536	82.86±0.14
Viçosa ₂	15.74	876.40	0.9949	18.58	1034.14	0.63	0.9824	0.89	1.23	5.01	68.90	0.9662	74.07±0.10

1/n: degree of linearity of the isotherm; K_l : Langmuir sorption coefficient; K_f : Freundlich sorption coefficient; K_d : linear sorption coefficient; K_{OC} : sorption coefficient normalized according to the organic carbon (OC) content of the soil (L kg⁻¹); foc : soil organic carbon content. The Langmuir (K_l) and Freundlich (K_f) coefficients were also normalized according to OC, where K_{loc} and K_{foc} are the normalized Langmuir and Freundlich coefficients, respectively. H: hysteresis coefficient.

The results of application of the desorption isotherm models (linear, Freundlich, and Langmuir) to the behavior of cyantraniliprole in the different soils are shown in Figure 5.

4. Conclusions

The results obtained in this work showed that the pesticide cyantraniliprole presented low percentage sorption in the soils evaluated (sandy loam texture and clay texture), indicating its high leaching potential. The low sorption coefficients for cyantraniliprole in these soils indicated high potential for the contamination of subterranean water bodies.

These findings could be used as a guide for establishing residue limits for cyantraniliprole in the environment.

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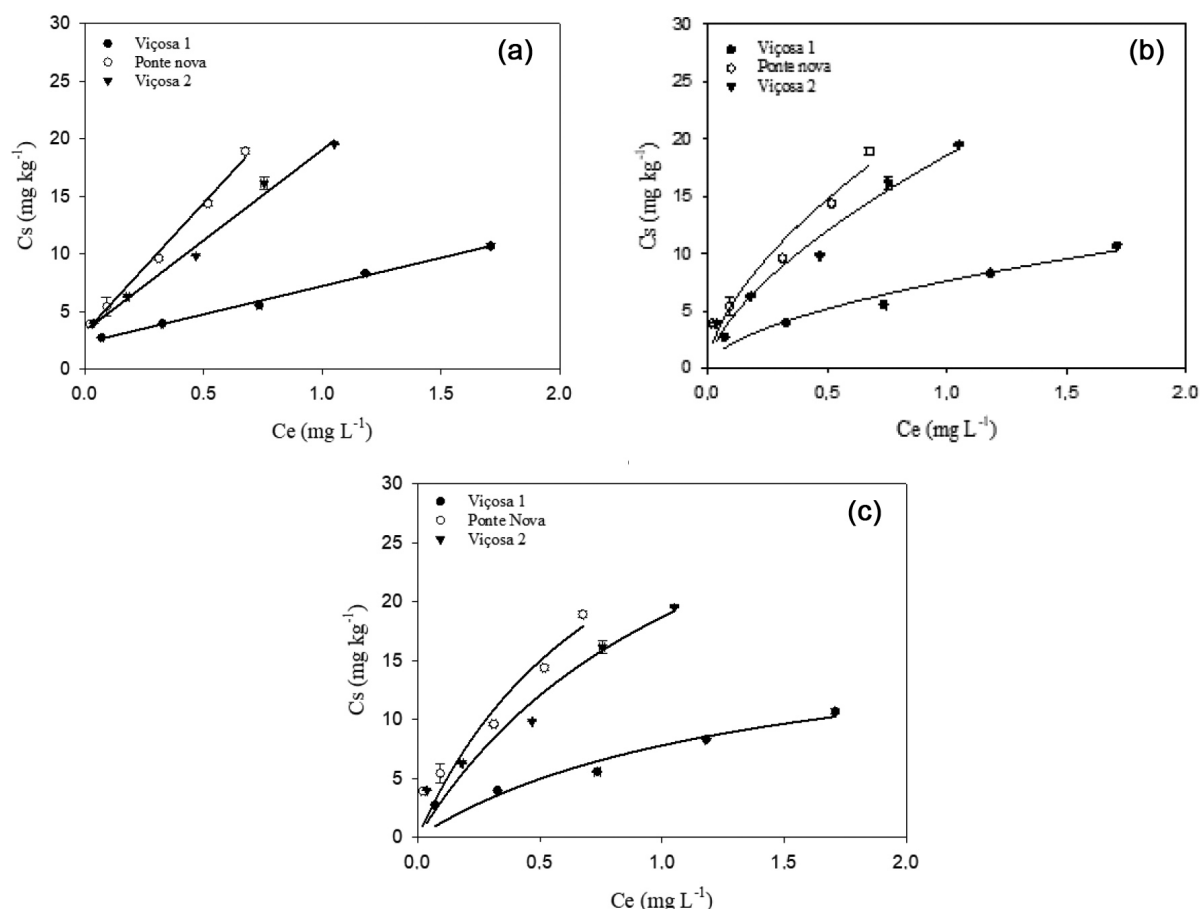


Figure 5. Application of the linear (A), Freundlich (B), and Langmuir (C) desorption isotherm models to the behavior of cyantraniliprole in the different soils

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