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Adsorption-desorption of Ivermectin in an agricultural soil in the province of Corrientes (Argentina)

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Abstract
Ivermectin is widely used in human and veterinary medicine and can contaminate the environment. The degree of adsorption of this drug on soil particles varies considerably, as does its mobility. Adsorption-desorption in an agricultural soil was studied using discontinuous equilibrium experiments. The adsorption and desorption data were correctly fitted to the Freundlich and Langmuir isotherms in a linear fashion ($r > 0.994$) and ($0.988 < r < 0.997$), respectively. An adsorption hysteresis phenomenon was observed, with values ranging from 0.24 to 0.37. Experimental results indicate that Freundlich sorption coefficient (KF) values for ivermectin ranged from 0.18 to 2.6 L g^{-1} for adsorption and ranged from 0.61 to 0.90 L g^{-1} for desorption.

Keywords: Ivermectin, agricultural soil, adsorption-desorption

Introduction
Avermectins are a diverse group of pharmaceutical compounds with a wide range of therapeutic applications. Renowned for their effectiveness, they are primarily utilized as pesticides, playing a crucial role in agriculture and veterinary medicine for the management of harmful pests and parasitic worms. Their potent anthelmintic and insecticidal properties make them indispensable in combating infestations, ensuring healthier crops and livestock. These compounds have become a cornerstone in pest control strategies, celebrated for their ability to target and eliminate unwanted organisms efficiently [21].

The compounds in question are derived from a 16-membered lactone ring. Avermectins are naturally occurring fermentation products of “Streptomyces avermitilis”, an actinomycete isolated from soil. Eight distinct structural variants have been identified and categorized into four primary components (A1a, A2a, B1a, and B2a) and four minor components (A1b, A2b, B1b, and B2b). Notable examples of avermectins include ivermectin, abamectin, doramectin, eprinomectin, moxidectin, and selamectin [12].

Their structures share similarity with antibacterial macrolides and antifungal macrocyclic polynucleotides, but differ in their mechanism of action [2]. In addition, they possess anticancer, antidiabetic, antiviral, antifungal properties and are used for the treatment of several metabolic disorders. Avermectin generally acts by preventing the transmission of electrical impulses in the muscles and nerves of invertebrates, amplifying the effects of glutamate on the gated chloride channel specific to these animals. Avermectin presents undesirable effects or reactions, especially when administered indiscriminately, such as respiratory failure, hypotension, and coma.

Ivermectin, the most widely used derivative of avermectin, is available in various formulations and can be administered through multiple routes. In clinical settings, it is indicated for the treatment of a range of conditions, including strongyloidiasis, scabies, pediculosis, gnathostomiasis, myiasis, and leishmaniasis [5, 6]. Additionally, research has demonstrated that ivermectin is effective in reducing the prevalence and transmission of other infectious diseases associated with soil-transmitted helminths. These diseases include not only strongyloidiasis but also ascariasis, trichuriasis, and enterobiasis, which are caused by hookworms, Trichuris, and Ascaris spp.

In animals, it is used to prevent heartworms, treat ectoparasites, and as a microfilaricide. However, it is particularly hazardous to crustaceans [8] if it leaches into aquatic environments, also to soil-dwelling organisms [13] if present at high levels in topsoil or pasture manure. Sorption is one of the key factors controlling the transport, transformation, and bioavailability of avermectins to these organisms.

The octanol/water ratio (K_{ow}) indicates the affinity of a compound for lipids and hydrophobic compounds. Reported K_{ow} values for different avermectins are 1.651×10^{-3} for ivermectin [1], 9.900×10^{-3} for abamectin [22], and 2.5787×10^{-3} for doramectin [20].

Avermectins are widely recognized for their limited potential to bioconcentrate, primarily due to their high molecular weight, which hinders their ability to cross biological membranes. Despite the scarcity of data on their sorption behavior in soil, it is clear that all three key avermectins—abamectin, doramectin, and ivermectin—remain ionized within the environmentally relevant pH range of 5 to 9. This characteristic indicates that their sorption to soil is largely independent of pH levels.

The avermectin family exhibits a notably high octanol-water partition coefficient, ranging from 5.3 × 10^3 to 8.69 × 10^4. This substantial value underscores their strong tendency to bind with organic carbon, suggesting that they do not leach through soil columns but instead remain firmly adhered to the soil matrix [7, 20].

Importantly, this strong adherence cannot be solely attributed to their lipophilic nature. Avermectins may also interact with soil through alternative adsorption mechanisms, including the formation of adducts or complexes with immobile inorganic materials present in the soil [19]. This multifaceted binding capability reinforces the notion that avermectins are not only persistent in soil but also highlight their significance in agricultural and environmental contexts.

The aim of our study was to rigorously investigate the adsorption and desorption behavior of ivermectin in rice-growing agricultural soil. In Corrientes province, where farms thrive on both agriculture and livestock, the extensive use of ivermectin in livestock presents significant environmental implications. By closely examining this soil, we sought to deepen our understanding of the results derived from toxicity tests on soil-dwelling non-target organisms. Furthermore, we compared our findings with existing literature to critically assess whether current adsorption models can convincingly attribute ivermectin's remarkably high adsorption potential to its lipophilicity or suggest the involvement of more intricate mechanisms. This comparison not only enhances our insights but also underscores the urgency of understanding the environmental impacts of veterinary pharmaceuticals.

Materials and Methods

Chemical

Ivermectin (IVM, CAS No. 70288–86–7, purity 94% B1a and 2.8% B1b) was purchased from Sigma-Aldrich (Steinheim, Germany). Diluted ivermectin standard solutions were prepared by diluting 100 mg L-1 of the purchased stock solution with ethanol. Dilutions of 4, 10, 20, and 30 mg L^{-1} were made in 0.01 mol L^{-1} CaCl. The stock solution was stored at −20°C for up to 6 months. All ivermectin solutions, as well as all test systems containing ivermectin, were wrapped in aluminum foil to prevent photodegradation.

Soil

The soil used is a vertic Epiacualf located on a hill in the rice field of Estancia Aguacerito, part of the Uruguay River basin in the department of Mercedes (M), located in the province of Corrientes (Argentina).

Soil samples were collected at a depth of 0 to 20 cm. The samples were air-dried, sieved to a particle size of ≤2 mm, and stored in plastic bags at room temperature until use. The textural class and some physicochemical properties of the soil are presented in Table 1.

Table 1: Physical and chemical properties of the selected soils

Depth (cm)	properties					
	pH	F(om)%	Texture			CIC meq $100g^{-1}$
			Sand	Silt	Clay	
0-20	6.42	2.53	41.83	34.97	23.20	24.43

Determination of the time required to reach equilibrium.

To this end, an adsorption study was performed using 10 centrifuge tubes containing 1 g of soil and 50 mL of a 10 mg L^{-1} ivermectin solution in 0.01 M $CaCl_2$. The centrifuge tubes were placed in an orbital bath at 25°C, and duplicate samples were taken after 1, 3, 5, 24, 48, or 72 h. Each sample was centrifuged for 15 minutes at 3000 rpm, and the supernatant was collected. Centrifugation and supernatant collection were repeated three times. The aqueous and soil phases were analyzed separately by UV spectrophotometry. The equilibrium time was 24 h.

Experimental Sorption Studies

Adsorption experiments were initially conducted in accordance with OECD Test Guideline 106 [17, 18]. A preliminary study was conducted to determine the optimal ratio (w/v) between soil and ivermectin solution in 0.01 M $CaCl_2$ to ensure the detection of ivermectin in both the solid and aqueous phases during analysis. After testing ratios of 1/1, 1/5, 1/25, and 1/50 (w/v), the test ratio of 1/50 (w/v) was selected for subsequent experiments.

Before selecting the final test conditions, the stability of ivermectin was investigated. The dissipation of ivermectin was found to be negligible for a week. The concentration of the test compound used in the adsorption-desorption equilibrium study was higher than the expected ambient concentrations of 1.5 mg kg−1 dry weight [13] and above the analytical detection limits used.

Working dilutions were placed in centrifuge tubes. The suspensions were thermostated in an orbital bath at 25°C and shaken for 24 hours, the equilibrium time previously determined. They were then centrifuged for 15 minutes at 3000 rpm, the supernatant was removed, and the ivermectin concentration was determined by UV spectrophotometry. Tests were performed in triplicate and at different temperatures (25°C, 35°C, and 45°C).

Experimental desorption studies

Immediately after adsorption, the supernatant was removed and replaced with 0.01 M CaCl2 solution, in the same 1/50 (w/v) ratio, stirred at 25°C for 24 hours, and centrifuged for 15 minutes. The supernatant was collected, and the desorbed ivermectin was determined by UV spectrophotometry.

Theory

The sorption distribution coefficient, K_d, is defined as the ratio of the soil-bound ivermectin concentration (C_s) to the equilibrium concentration in solution (C_e).

$$K_d = \frac{C_s}{C_e} \quad (1)$$

K_{oc} is the normalized distribution coefficient of organic carbon and can be expressed in equation (1). foc% represents the percentage of organic carbon in the soil sample (g g−1).

$$K_{OC} = K_d \cdot \frac{100}{foc\%} \quad (2)$$

In this study, K_d and K_{oc} values were calculated based on the results of C_s and C_e determined with ivermectin.

Adsorption experiments at multiple concentrations allow the construction of adsorption isotherms from which the dependence of the distribution coefficient on Ce can be determined.

The Freundlich sorption isotherm defines the equilibrium between the concentration of ivermectin in the aqueous phase and the soil phase. K_F represents the Freundlich constant, while 1/n is the regression coefficient. If n does not deviate from unity, K_F equals K_d.

$$C_s = K_F C_e^{1/n} \quad (3)$$

The linearized equation:

$$logC_s = logK_F + \frac{1}{n} logC_e \quad (4)$$

The constants K_F and 1/n change with an increase in temperature.
The Langmuir isotherm was initially established for gas-solid interactions, but it is also used for various adsorbents. It is an empirical model based on kinetic standards; that is, surface adsorption and desorption rates are equivalent without aggregation under equilibrium conditions.

The Langmuir isotherm can be described by the equation:

$$C_s = \frac{C_{s,max} K_L C_e}{1 + K_L C_e} \qquad (5)$$

Where C_e is the equilibrium concentration (mg L^{-1}); C_s is the amount of contaminant adsorbed at equilibrium (mg g^{-1}); $C_{s,max}$ is the amount of adsorbate required to form the monolayer (mg g^{-1}) and K_L is the adsorption equilibrium constant (L mg^{-1}).

The linearized equation:

$$\frac{C_e}{C_s} = \frac{C_e}{C_{s,max}} + \frac{1}{K_L C_{s,max}} \qquad (6)$$

The desorption percentage D_t was calculated using equation (7).
The parameter C_s^a is the concentration (mg g^{-1}) of ivermectin adsorbed to the soil at sorption equilibrium, while C_e^d is the total concentration (mg g^{-1}) of ivermectin desorbed from the soil in a given time t.

$$D_t = \frac{m_e^d}{m_s^a} \qquad (7)$$

Data Treatment
In this part of the thesis, the following error functions were chosen:

1. **Correlation coefficient, R^2:**

$$R^2 = \frac{\sum_{i=1}^{n}\left(q_{cal} - \overline{q_{exp}}\right)^2}{\sum_{i=1}^{n}\left(q_{cal} - \overline{q_{exp}}\right)^2 - \sum_{i=1}^{n}\left(q_{cal} - q_{exp}\right)^2} \qquad (8)$$

2. **Mean Sum of Squares Error (MSE):**

$$MSE = \frac{1}{n}\sum_{i=1}^{n}\left(q_{cal} - q_{exp}\right)^2 \qquad (9)$$

Results and Discussion
Sorption Isotherms of Ivermectin by Soil
By plotting the sorption coefficient Kd as a function of time, from 1 to 72 h, it was shown that equilibrium was reached at 24 h. Thus, the time for equilibrium was set at 24 h, to ensure that equilibrium was reached in all experiments.
Soil/0.01 M $CaCl_2$ solution ratios of 1/50, 1/25, 1/5, and 1/1 were tested, containing 0.30, 0.10, and 0.01 mmol of $CaCl_2$ per g of soil. The ratio of 1/50 was selected because it did not affect the distribution of the compound. Between 30 and 60% of the ivermectin applied to the soil was recovered, which is in accordance with OECD conditions.
K_d values for ivermectin ranged from 42 to 63 L kg^{-1} (determined at 10 mg L^{-1}) in the studied soil, while K_{oc} values ranged from 1.6 10^3 to 2.5 10^3 L kg^{-1}. These data agree with those published by other authors for soils with high organic carbon content [16]. These data have then been used to make additional estimates of the sorption correlation.
The experimental results showing how ivermectin adsorbs onto the soil at various temperatures follow an L-type adsorption isotherm. This characteristic shape is consistent with the Langmuir adsorption model, which strongly suggests that the ivermectin molecules are likely engaging in chemical interactions with all the available active sites on the soil's surface. This type of interaction is typically limited to the formation of a single molecular layer of the substance on the adsorbent surface.
The adsorption data were fitted to the logarithmic form of the Freundlich and Langmuir equations. The results obtained from the isotherm parameters at different temperatures are shown in Table 2. (Figure 1)

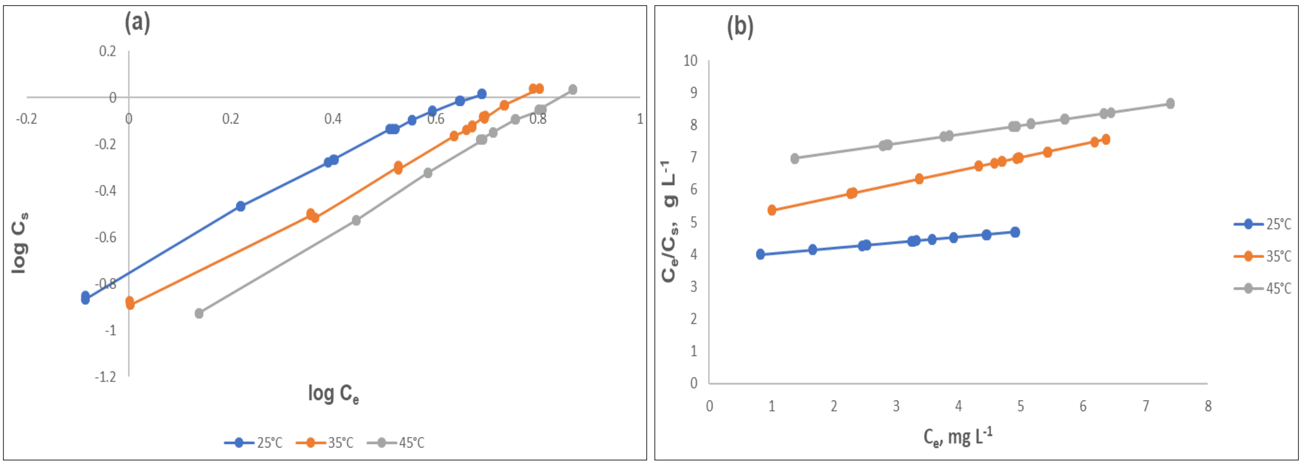

Fig 1: Representation of the models at different temperatures for the adsorption of ivermectin to soil: (a) Freundlich; (b) Langmuir

The results obtained for the adsorption data were fitted to the linealrly form of the Freundlich and Langmuir equation at different temperatures are shown in Table 2. Looking at the Freundlich parameters, we see that the heats of 1/n differ from unity, indicating that the adsorption process is not linear. K_F varied from 78.03 to 183.0 L kg^{-1} depending on the experimental temperature (Table 1). These results agree with those reported by other authors [8, 15]. In addition, regression coefficients (R^2) from the linear regression were calculated. R^2 values ranged from 0.993 to 0.994 decreasing with increasing temperature (Table 1). This model assumes that with the values obtained, at low temperature there is favorable adsorption and at high temperature there is unfavorable adsorption.

The Langmuir equation does not adequately reproduce the experimental data on ivermectin in soil because the hypotheses on which the equation was formulated are not met in reality. That is, the assumption that the adsorbent surface consists of active sites of equal activity is not met, nor is the assumption that there is no interaction between the adsorbate molecules and between them and the adsorbent. A characteristic of the Langmuir isotherm is found in the dimensionless constant R_L, from which the favorability of adsorption on homogeneous surfaces can be inferred and therefore indicates the type of isotherm and the calculations that must be applied, the values found for this parameter in the soil under study are between 0.35 and 0.48, results that are between 0 and 1, from which it can be deduced that adsorption is favorable.

According to the analysis of the two most common methods used in the adsorption process, one would give us a favorable process in the temperature range studied and the other a favorable or unfavorable one, indicating that the process is a bit more complex. Drug interactions with the soil are not considered in the Langmuir and Freundlich models, so we can infer that the interactions with the soil are more complex.

Table 2: Freundlich, Langmuir, and thermodynamic parameters of ivermectin adsorption in the studied soil

Parameters	Temperatura (°C)		
	25°C	35°C	45°C
Freundlich			
1/n	1.136	1.141	1.307
K_F (L g^{-1})	0.183	0.127	0.078
R^2	0.9943	0.9957	0.9964
MSE 10^3	1.336	1.119	3.689
Langmuir			
$C_{s,m}$ (mg g^{-1})	4,508	2.406	2.300
K_L (L g^{-1})	0,0606	0.093	0.091
R_L	0,484	0.355	0.392
R^2	0.9880	0.9968	0.9932
MSE 10^3	1.508	5.681	8.646
Thermodynamic Parameters			
T (°C)	ΔG^{0} (kJ/mol)	ΔH^{0} (kJ/mol)	ΔS^{0} (J/mol K)
25	-13.89	-20.71	-25.14
35	-12.88		
45	-12.76		

Freundlich, Langmuir parameters of ivermectin adsorption in the studied soil decreasing with increasing temperature. These results agree with those reported by other authors.

The analysis shows that both of the analyzed isotherm models, Langmuir and Freundlich, can adequately fit the experimental results obtained at all studied temperatures. While the Freundlich model exhibits slightly better statistics (or lower statistical error), the good correlation obtained with both indicates that further investigation is essential. This requires the exploration of additional theoretical models to accurately distinguish between the possible adsorption mechanisms and precisely define the nature of the ivermectin-soil interaction.

Desorption

Ivermectin desorption was negligible in all experiments. Desorption, like adsorption, presents L-type isotherms at different temperatures. (Figure 2)

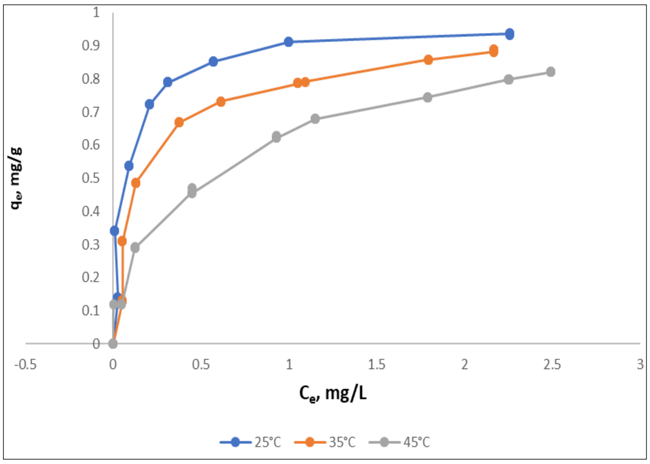

Fig 2: Isoterm desorption of ivermectin at different temperature

The desorption data, calculated as the percentage of desorbed ivermectin (Eq. (4)) in relation to the amount of ivermectin originally adsorbed, were very low, ranging from 2 to 8%, and remained constant at different temperatures. (Table 3) This would indicate that it is strongly retained in the soil and would not leach, therefore it would not contaminate groundwater. The compound's desorption mechanism will depend primarily on the adsorption energy; the higher this energy, the more difficult it will be to desorb the pesticide back into solution from the soil. The desorption mechanism can be studied using the hysteresis phenomenon, which is defined as the degree of reversibility of the adsorption process [7]. Hysteresis can be calculated using the following equation:

$$H = \frac{1/n_d}{1/n_a} \qquad (10)$$

Hysteresis (H) is a commonly observed phenomenon and is attributed to the different forces involved in sorption and desorption. When the Freundlich sorption coefficient is higher at the desorption stage, after reaching apparent equilibrium, than at the adsorption stage, it indicates that the compound, once sorbed, is difficult to desorb. Hysteresis has been reported for many organic compounds where soil or sludge acts as sorbent [11, 14, 4]. A number of experimental artifacts are known to also contribute to hysteresis [3]. The irreversibility of the adsorption process may be due to the entrapment of sorbed molecules in soluble organic matter and inorganic matrices, enhanced by artifacts in the experimental procedure [10, 11] or to entrapment in soils with low organic matter content and high internal surface area [9]. In the present study, a small positive hysteresis was observed in all soils, and its magnitude was assessed by the ratio of the Freundlich isotherm coefficients for adsorption and desorption.

Figure 3 shows how the experimental data fit the chosen models at different temperatures.

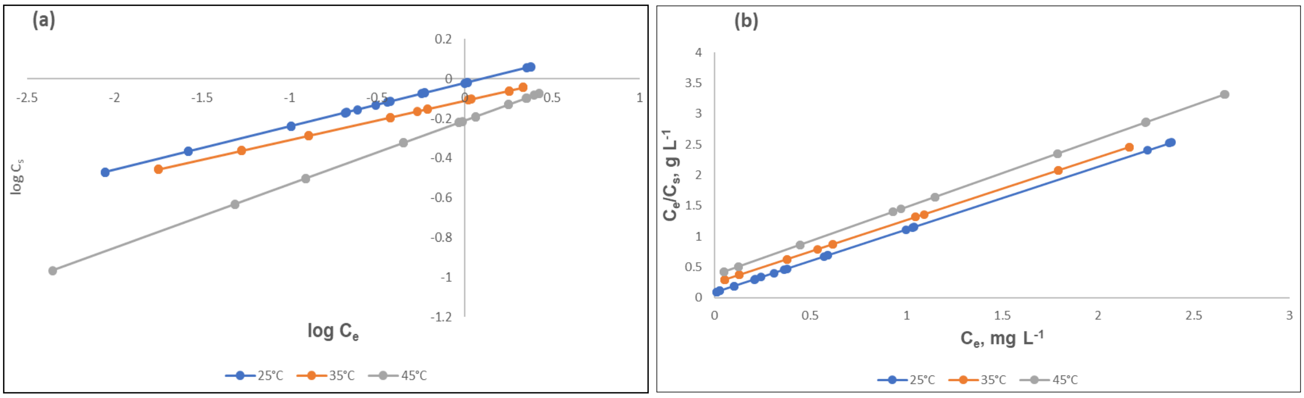

Fig 3: Representation of the models at different temperatures for the desorption of ivermectin to soil: (a) Freundlich; (b) Langmuir

Table 3: Freundlich, Langmuir, and thermodynamic parameters of ivermectin desorption in the studied soil

Parameters	Temperatura (°C)		
	25°C	**35°C**	**45°C**
Freundlich			
1/n	0.293	0.420	0.321
K_F (L g^{-1})	0.905	0.755	0.615
H	0.258	0.371	0.247
R^2	0.9943	0.9958	0.9890
MSE 10^2	1.648	1.544	1.768
Langmuir			
$C_{s,\,m}$ (mg g^{-1})	0.972	0.974	0.902
K_L (L g^{-1})	12.324	4.320	2.998
R_L	0.007	0.021	0.032
R^2	0.9972	0.9925	0.9888
MSE 10^3	8.686	5.728	1.137
Thermodynamic Parameters			
T (°C)	ΔG^{0} (kJ/mol)	ΔH^{0} (kJ/mol)	ΔS^{0} (J/mol K)
25	-21.99	-49.84	-93.39
35	-21.10		
45	-20.13		

The Freundlich constants obtained for desorption are greater than the KF results for adsorption processes. Consequently, the desorption curve is located above the adsorption curve, indicating that the hysteresis coefficients are greater than unity. This behavior confirms that the soil exhibits strong retention of ivermectin after the adsorption stage. (Figure 4)

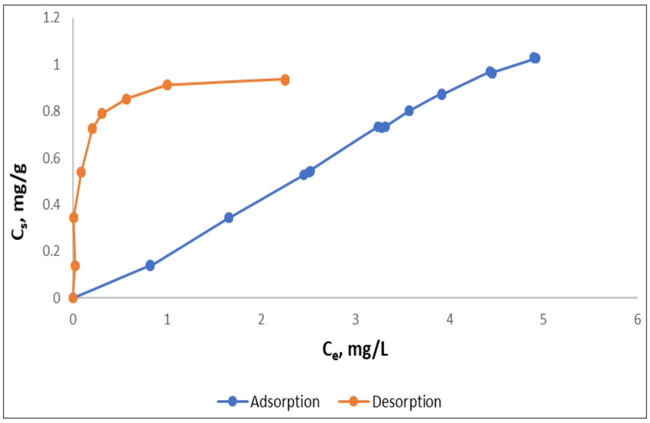

Fig 4: Representation of the adsorption-desorption equilibrium of ivermectin at 25°C

The desorption isotherm exhibits a steeper gradient compared to the adsorption isotherm, indicating a significant hysteresis effect. This disparity implies that the retained ivermectin is poorly desorbed and strongly sequestered by the soil matrix. This strong retention mechanism is likely attributable to the organic matter content in the soil, which significantly favored the binding process over the subsequent release of the compound.

The slope of the adsorption isotherm is particularly noteworthy, as its characteristics suggest strong retention and high affinity of the drug for the soil. This observation is consistent with the known properties of ivermectin, specifically its high hydrophobicity and large molecular size, which synergistically contribute to its greater affinity for the soil matrix through processes like partitioning into soil organic matter.

Figure 5 presents the fit of the experimental data to both models, with the Langmuir model being the most accurate representation.

According to Table 3, both types of linearization exhibit high regression values, with the highest value achieved by the Langmuir model. Additionally, this model demonstrates the best mean sum of squares error (MSE), as illustrated in a typical saturation graph (Figure 5).

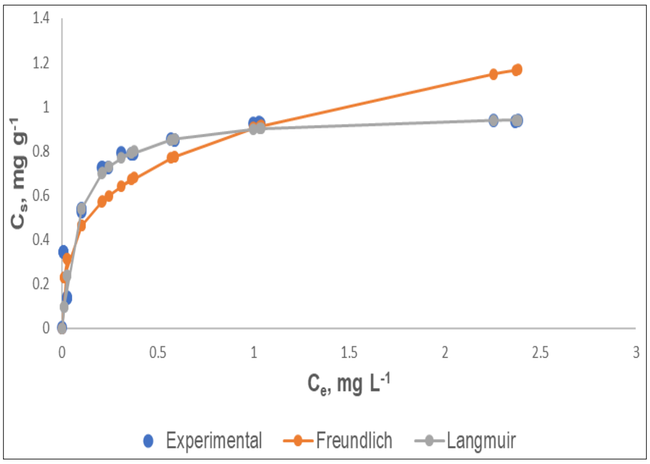

Fig 5: Fitting theoretical models to experimental data on ivermectin desorption at 25°C

Conclusion

The sorption of ivermectin to soil is crucial for understanding its fate and transport in the environment, as well as for assessing its associated risks. This study investigated the adsorption and desorption of ivermectin in agricultural rice soil, following the OECD Test Guideline 106. The experimental data for adsorption were well-described by the Freundlich isotherm, while the desorption data were better represented by the Langmuir isotherm. The results indicated that ivermectin exhibited enhanced adsorption due to the presence of organic carbon and clay within the soil.

Notably, the desorption coefficient (K_{Fdes}) was considerably higher than the adsorption coefficient (K_{Fad}), indicating a positive hysteresis effect. This suggests that once ivermectin is adsorbed to the soil, it is not readily desorbed, particularly within the typical soil pH range. The hysteresis phenomenon points to strong irreversible interactions between the ivermectin molecules and the soil particles.

The relatively high sorption coefficients observed in this study indicate a significant interaction between ivermectin and soil binding sites, suggesting that this drug is unlikely to reach surface and groundwater.

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