

Derivation and application of a generalised exchange-adsorption isotherm for the adsorption of hydrophobic compounds on soils

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Abstract: A general adsorption model able to provide a good description of the multilayer adsorption of ions and molecular compounds under any circumstances is proposed in this paper. In order to reach this goal, a general form of isotherm was deduced based on the Gapon equation and classical isotherm forms. The adsorption of molecular compounds with diverse hydrophobicity and acid-base characteristics were investigated and validated with a new model for the adsorption of four pesticides on different soils. The adsorption capacity and adsorption energy estimated by the new model were found to be related to the organic matter content of the soil. Great differences were found in the isotherm parameters (A , K , and n) among both the soils and compounds investigated here.

Keywords: isotherm parameters; multilayer adsorption; organic matter; pesticide

Nowadays, the adsorption of organic and inorganic pollutants in the environment is a very important research area because the mobility and fate of these materials could be critical for the preservation of the environment. Most sorption processes (e.g., adsorption, exchange) in the environment occur on the solid-liquid interface and these processes can be described for various molecules and elements using isothermal models. The parameters of these models could be used in environmental issues like the fate and transport of materials such as organic matter and heavy metals (Huang et al. 2018) and the health risks involved in the transfer of the potentially toxic elements (Aghili et al. 2018). They could also provide information on the behaviour of the pollutants in the course of water treatment (Anastopoulos et al. 2017) and soil remediation (Liang et al. 2016).

Many theoretical isotherms have been designed to describe sorption processes, one of the first of which was the adsorption isotherm proposed by Langmuir (1918). This model assumed that adsorption takes place on an energetically homogeneous surface, that the molecules or ions can form a monolayer on the surface, and that there is no interaction between the adsorbed molecules (Kapoor et al. 1990). Although soils cannot be considered to be an energetically homogeneous adsorbent, as they involve binding sites with a different affinity and reactivity and contain functional groups, hydrophilic and hydrophobic moieties, the Langmuir equation has been successfully used to describe the adsorption of both ionic substances and uncharged molecules (Olsen & Watanabe 1957; Zhang & Selim 2005; Subramanyam & Das 2009; Chen et al. 2017).

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Multilayer isotherms that can be classified as type VI isotherms having more than one inflection point and local maxima (Schneider 1995) can be used to describe the adsorption process on mesoporous solids, such as soils (Silberberg 1971; Saadi et al. 2015), where the Langmuir equation does not adequately describe the adsorption, presumably because (i) the soil contains non-homogeneous energy adsorption sites and (ii) the adsorption is not a monolayer (Polley et al. 1953; Shahbeig et al. 2013).

Czinkota et al. (2002) presented a new general isotherm model, a modification of the Langmuir isotherm, which adequately described the stepwise adsorption of certain pesticides on soils. However, the derivation of the isotherm and the interpretation of the model parameters were lacking. The present paper attempts to find a solution to one of the basic problems of physical chemistry: the shortcomings of the Freundlich and Langmuir isotherms at certain concentrations, and to compile an adsorption isotherm that is able to provide a good description of the multilayer adsorption of ions and molecular compounds under any circumstances. Therefore, the objectives were (i) to derive a new generalised isotherm, and (ii) to interpret the physical meaning of the model parameters by evaluating the adsorption of four pesticides on three different soils.

MATERIAL AND METHODS

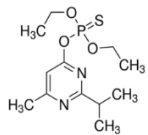
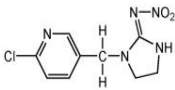
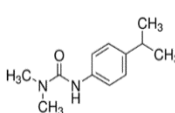
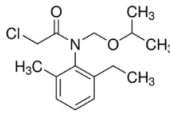
Adsorption experiments. The experiment for investigating the adsorption of the compounds in organic pesticides was clearly described in the stud-

ies of Czinkota et al. (2002) and Konda et al. (2002). Briefly, the sorption experiments were conducted on representative Hungarian soils with different physico-chemical properties (Table 1).

Pesticide solutions were prepared in 0.01 mol/L of calcium chloride to maintain a constant ionic strength (Table 2). The concentration ranged from 1 to 15.0 mg/L for propisochlor, diazinon, imidacloprid and isoproturon. The adsorption of propisochlor was studied for all three soil types, whilst for diazinon, imidacloprid and isoproturon, the batch experiments were carried out with Cambisol only.

The soil samples were then shaken for 1 h with the pesticide solutions and left to stand overnight for equilibration at 25 °C. The equilibrium phases were separated by centrifugation (3 000 rpm). After filtration, the pesticide concentrations were analysed by High Performance Liquid Chromatography (HPLC). Every setup in the experiment was repeated three times and the results were analysed statistically. The isotherm models for the nonlinear fitting were made using the ORIGIN program (Ver. 7.5, 2003). The fraction of the pesticides present as neutral, negatively or positively

Table 2. Properties of the pesticides used in the sorption studies

Diazinon		MW: 304.3
		S: 60 mg/l (@20 °C) logKow: 3.81 pK _a : 2.6 ¹ α ₀ : 100%
Imidacloprid		MW: 255.7
		S: 350 mg/l (@20 °C) logKow: 0.65 pK _a : 5.9 ² α ₀ : 95.5, 91.9 and 90.5% ³ α ₊ : 4.5, 8.1 and 9.5%
Isoproturon		MW: 206.3
		S: 70 mg/l (@20 °C) logKow: 2.87 pK _a : non-ionic α ₀ : 100%
Propisochlor		MW: 283.8
		S: 15.6 mg/l (@25 °C) logKow: 3.50 pK _a : 8.9 α ₀ : 100%

¹fraction of pesticide in a neutral form; ²fraction of pesticide in a neutral form at the soil pH of (7.73, 6.35 and 6.27); ³fraction of pesticide in a positive form at pH of soils (7.73, 6.35 and 6.27); MW – molecular weight; S – solubility

Table 1. Selected properties of the soils

	Calcic Chernozem	Mollic Cambisol	Albic Arenosol
pH (H ₂ O)	7.73	6.35	6.27
SOC (mg C/g soil)	41.0	22.0	13.2
Silt + clay (%)	55.9	42.6	18.4
CaCO ₃ (%)	5.9	–	–
BET surface (m ² /g)	21.6	5.9	2.5
Mineral composition (%)			
Albite	9.8	12.6	15.1
Chlorite	5.3	6.6	2
Mica	4.2	7.8	2.4
Quartz	30.6	48.4	63.9
Smectite	32.4	14.3	8.3

SOC – soil organic carbon

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charged species ($\alpha 0$, $\alpha -$, $\alpha +$) were calculated with MarvinSketch from ChemAxon (Budapest, Hungary).

Derivation of the new isotherm equation. The derivation was performed for the charged particles (ions), but the equation is also valid for compounds without a charge (molecules).

The derivation of the isotherm equation was based on the Gapon equation (Gapon 1933), assuming a unified surface charge, where the surface is loaded with the ion Y, and the solution contains the ion X. For the sorption of ionic compounds, the equation takes the form:

$$\begin{aligned} n \times Y^{(1/n)+} + S &= Q + m \times X^{(1/m)+} \text{ or} \\ n \times Y^{(1/n)-} + S &= Q + m \times X^{(1/m)-} \end{aligned} \quad (1)$$

and for the sorption of molecules:

$$n \times Y + S = Q + m \times X \quad (2)$$

where:

- Y – the equivalent concentration of the adsorbed material,
- X – the equivalent concentration of the desorbed material,
- S – the surface charge adsorbed by ion X,
- Q – the surface charge adsorbed by ion Y,
- n, m – the number of adsorbed ions (which equals the fraction number if the ion has a charge of more than 1),
- 1/n – the charge of ion Y, or the number of molecules bound to the surface,
- 1/m – the charge of ion X, or the number of molecules bound to the surface.

The reciprocals of the n and m values are equal to the charge of the adsorbed ions, or to the number of molecules per binding site in the case of molecules or associations.

Based on Equation (2), the equilibrium constant can be expressed as:

$$K' = Q \times [X]^m / [Y]^n \times S \quad (3)$$

where:

K' – the constant for the adsorption exchange rate.

Applying the mass balance equation for the sorption processes gives:

$$A = Q + S \text{ and } S = A - Q \quad (4)$$

where:

A – the charge for the whole surface (adsorption capacity of the soil).

Using the mass balance Equation (4), Equation (3) can be rewritten as:

$$K' = (Q \times [X]^m) / ([Y]^n \times (A - Q)) \quad (5)$$

Expressing Q is the next step in the transformation (by analogy with the Langmuir isotherm derivation):

$$Q = (K' [Y]^n \times A) / ([X]^m + K' \times [Y]^n) \quad (6)$$

If $[Y] = c$, the classical form of the isotherm equation is obtained:

$$Q = (A \times K' \times c^n) / ([X]^m + K' \times c^n) \quad (7)$$

Simplification of the model at a near constant desorbed concentration.

Equation (7) is the general form of the exchange isotherm. It can be used to describe the ion exchange process (for example, in the case of ammonium-calcium) if the equilibrium concentrations of both the adsorbed and desorbed ions are known. The general form of Equation (7) can be used if the two concentrations can be measured in the given experiment and we know exactly what materials take place in the exchange process. The main problem is that we can never be sure that only one type of material has a role during desorption. Consequently, it is necessary to make certain simplifications.

There could be many cases (for example, in soils) where the desorbed ion concentration $[X]$ is nearly constant, either due to a buffer reaction or to the much lower concentration of the desorbed material which means that it can be omitted. In this case we can write:

$$K = K' / [X]^m \text{ or } K' = K \times [X]^m \quad (8)$$

where:

K – the ion equilibrium exchange rate related to constant desorbed ion concentration.

By substituting $K \times [X]^m$ for K' in Equation (7), we obtain:

$$Q = (A \times K \times [X]^m \times c^n) / ([X]^m + K \times [X]^m \times c^n) \quad (9)$$

If we assume that $[X]^m = 1$, we obtain:

$$Q = (A \times K \times c^n) / (1 + K \times c^n) \quad (10)$$

This simplification can only be made if the change in the amount of desorbed material can be neglected and considered constant. This is valid for the following situations:

- (i) if the surface was empty at the beginning (similarly to the Langmuir isotherm), if solvent molecules (for example, water) were adsorbed on the surface, but the concentration change caused by the desorption can be neglected
- (ii) if the desorption of the hydrogen or hydroxide ions does not change their concentration due to a buffer system in the soil
- (iii) if the desorption of the ions (for example, Al^{3+} , Fe^{3+} , HCO_3^- , ammonium, etc.) does not alter their concentration because they are in hydrolytic equilibrium with the hydrogen and hydroxide ions
- (iv) if the ions present in the soil solution in a high concentration are desorbed, then they can be neglected (for example, sodium, chloride, sulfate in salt-affected soils)
- (v) if the concentration of desorbed ions remains at a constant level due to the precipitation processes (for example, phosphate ions, calcium ions)
- (vii) if the concentration of desorbed ions is nearly constant due to a complex equilibrium (for example, a high amount of metal ions in a humic environment)
- (viii) if the concentration of the desorbed ions is nearly constant due to a carbonate equilibrium system and the calcium ion concentration is regulated by the solubility of CaCO_3 .

If the ions, molecules, molecule ions or associates with different charges can be adsorbed consecutively in several layers, the process can be written as:

$$Q = \sum_{i=1}^s \left(\frac{A_i \times K_i \times c_i^{n_i}}{1 + K_i \times c_i^{n_i}} \right) \quad (11)$$

where:

s – the number of steps of the isotherm,

A_i – the adsorption capacity,

K_i – the equilibrium constant,

n_i – the n values.

Each adsorption step can be characterised by three parameters: A_i , K_i and n_i . No evident physical explanation could be attributed to n (Konda et al. 2002). The sum of the maximum quantities adsorbed in each part process ($\sum A_i = A$) must equal the total maximum adsorbed quantity, taking into account that more than one layer may be formed in the adsorption process, in which case, the adsorbed associate ions may behave as different materials. For two-step isotherm, we can write:

$$Q = \frac{A_1 \times K_1 \times c_1^{n_1}}{1 + K_1 \times c_1^{n_1}} + \frac{A_2 \times K_2 \times c_2^{n_2}}{1 + K_2 \times c_2^{n_2}} \quad (12)$$

while the three-step isotherm is:

$$Q = \frac{A_1 \times K_1 \times c_1^{n_1}}{1 + K_1 \times c_1^{n_1}} + \frac{A_2 \times K_2 \times c_2^{n_2}}{1 + K_2 \times c_2^{n_2}} + \frac{A_3 \times K_3 \times c_3^{n_3}}{1 + K_3 \times c_3^{n_3}} \quad (13)$$

So, the multistep isotherm can be calculated from the adsorption isotherm of the individual steps (Equation 12 and 13), where each isotherm starts from the previous limit value of c_i . The limit values of the distinct isotherms can be calculated by determining the inflection point of the curve using the second derivative of the function: at inflection point the second derivative has an isolated zero. The limit concentrations are presented in Table 3, 4 and 5.

RESULTS AND DISCUSSION

Application of the new model for the adsorption of pesticides on soils: adsorption of propisochlor on three different soils. The adsorption isotherm of a pesticide (propisochlor) on three different soils is shown in Figure 1. Only the new isotherm (Equation (11)) was fitted to the data, as the measured values could not be described satisfactorily using classical adsorption isotherms. The parameters of the new model are presented in Table 3. The Chernozem soil had the highest value of A_1 (0.506 $\mu\text{mol/kg}$) and also the highest K values ($4.6 \cdot 10^{-4}$ and $6.64 \cdot 10^{-18}$), which are proportional to the adsorption energy indicating that the energy of adsorption is the highest in the chernozem soil. The organic matter (OM) of the soils was found to be the principal factor in controlling the sorption of organic compounds (Lambert 1968), which is why the highest affinity is found in the soil with the highest SOM content.

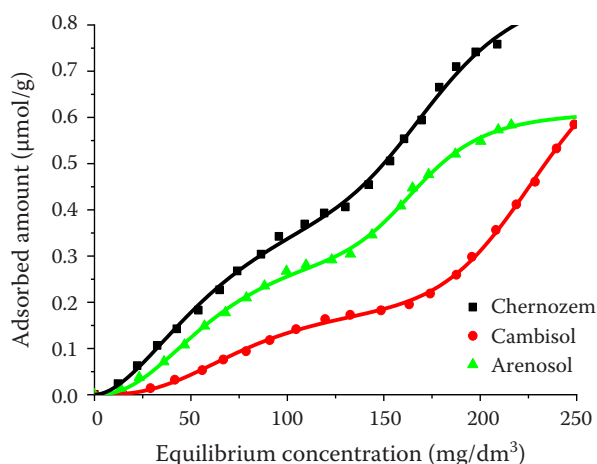


Figure 1. Adsorption of propisochlor on the different soils

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Table 3. Adsorption parameters for propisochlor

	A_1 ($\mu\text{mol/kg}$)	K_1 ($\text{dm}^3/\mu\text{mol}$)	n_1	C_1 ($\text{mg}\cdot\text{dm}^3$)	A_2 ($\mu\text{mol/kg}$)	K_2 ($\text{dm}^3/\mu\text{mol}$)	n_2	R^2
Cambisol	0.21 ± 0.01	$5.2 \cdot 10^{-6} \pm 5.8 \cdot 10^{-6}$	2.77 ± 0.28	149 ± 11	0.58 ± 0.07	$1.6 \cdot 10^{-22} \pm 4.8 \cdot 10^{-22}$	9.21 ± 0.50	0.999
Arenosol	0.35 ± 0.02	$7 \cdot 10^{-5} \pm 5 \cdot 10^{-5}$	2.30 ± 0.23	128 ± 9	0.27 ± 0.02	$1.81 \cdot 10^{-22} \pm 5.9 \cdot 10^{-22}$	9.77 ± 0.63	0.998
Chernozem	0.51 ± 0.06	$4.6 \cdot 10^{-4} \pm 3.3 \cdot 10^{-4}$	1.80 ± 0.22	121 ± 8	0.42 ± 0.06	$6.64 \cdot 10^{-18} \pm 5.7 \cdot 10^{-17}$	7.65 ± 1.63	0.998

A , K , n , C – isotherm parameters

Table 4. Parameters of the new model for the adsorption of diazinon and isoproturon

	A_1 ($\mu\text{mol/kg}$)	K_1 ($\text{dm}^3/\mu\text{mol}$)	n_1	C_1 ($\text{mg}\cdot\text{dm}^3$)	A_2 ($\mu\text{mol/kg}$)	K_2 ($\text{dm}^3/\mu\text{mol}$)	n_2	R^2
Isoproturon	18.1 ± 20.4	0.13 ± 0.16	0.71 ± 0.30	4.31 ± 0.25	9.9 ± 1.4	$7.7 \cdot 10^{-10} \pm 6.9 \cdot 10^{-9}$	12.4 ± 5.3	0.997
Diazinon	29.8 ± 2.2	0.56 ± 0.08	1.9 ± 0.2	4.08 ± 0.33	31.6 ± 2.0	$2.3 \cdot 10^{-8} \pm 6.3 \cdot 10^{-8}$	10.4 ± 1.6	0.999

A , K , n , C – isotherm parameters

Application of the new model for the adsorption of pesticides on soils: adsorption of diazinon and isoproturon on brown forest soil. The adsorption of two further pesticides (isoproturon and diazinon) was examined on a brown forest soil to demonstrate the ability of the new isotherm to describe the multilayer adsorption (Figure 2).

Similar to propisochlor, the adsorption isotherms of both isoproturon and diazinon were found to be stepwise. In the lower concentration range and in the first layer, the number of molecules in the association was about 1 for isoproturon and 2 for diazinon, indicating that diazinon was adsorbed on the soil as a dimer (Table 4). In the higher concentration range, forming a second adsorption layer, the number of pesticide molecules in the association increased to 12 for isoproturon and 10 for diazinon.

Since diazinon has greater hydrophobicity ($\log K_{ow}$: 3.81) than isoproturon ($\log K_{ow}$: 2.87), the A and K values are greater for diazinon than for isoproturon, indicating that diazinon has a greater affinity for the organic matter in the soil. For example, the adsorption capacity of the first layer for diazinon was $29.8 \mu\text{mol/kg}$, while this value was only $18.1 \mu\text{mol/kg}$ for isoproturon. The K_1 values were $0.56 \text{ dm}^3/\text{mg}$ for diazinon and $0.13 \text{ dm}^3/\text{mg}$ for isoproturon (Table 4). Surprisingly, a difference of two orders of magnitude was found between the K_2 values of the two molecules at higher concentrations (7.7×10^{-10} for isoproturon and 2.3×10^{-8} for diazinon).

At the pH in the soil (6.35), the diazinon molecule can be found in an ionised form (pK_a value = 2.6), so the adsorption of this molecule can be explained by the electrostatic attraction.

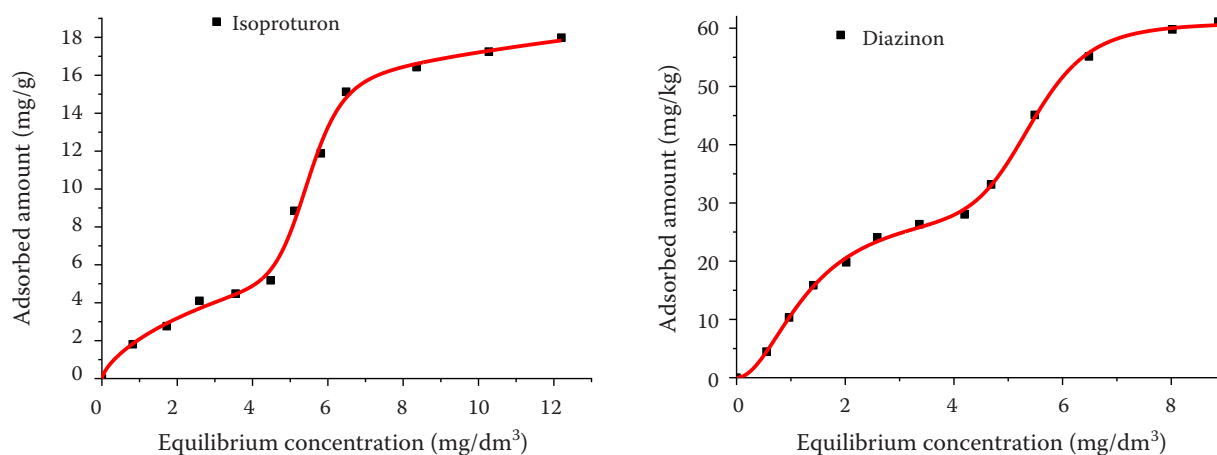


Figure 2. Adsorption of isoproturon and diazinon

Table 5. Adsorption isotherm parameters for imidacloprid on the brown forest soil

A_1 (mg/kg)	K_1 (dm ³ /μmol)	n_1	C_1 (mg.dm ³)	A_2 (mg/kg)	K_2 (dm ³ /μmol)
16.1 ± 55.6	0.21 ± 0.89	0.52 ± 0.61	3.2 ± 0.3	7.04 ± 2.04	2.1·10 ⁻⁸ ± 3·10 ⁻⁷
n_2	C_2 (mg.dm ³)	A_3 (mg/kg)	K_3 (dm ³ /μmol)	n_3	R^2
12.1 ± 9.8	7.2 ± 0.6	5.7 ± 1.8	5.9·10 ⁻¹⁷ ± 9·10 ⁻¹⁵	18.1 ± 75.4	0.997

A , K , n , C – isotherm parameters

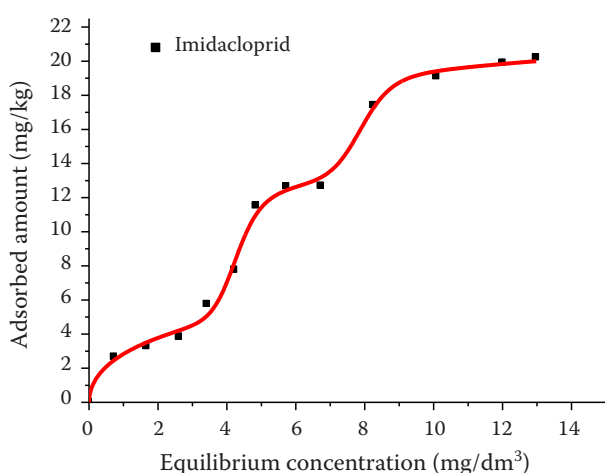


Figure 3. Adsorption of imidacloprid on the brown forest soil

Application of the new model for the adsorption of pesticides on soils: adsorption of imidacloprid on brown forest soil. When the adsorption of a further pesticide, imidacloprid, was tested, it exhibited a stepwise adsorption behaviour on the brown forest soil, with three adsorption layers (Figure 3). Although the hydrophobicity of imidacloprid was much lower than that of isoproturon, the adsorption capacity and affinity calculated using the model were comparable (Table 5). This could be attributed to the fact that imidacloprid can be adsorbed in the interlayer space of smectites, mainly by the hydrophobic interactions with the alkyl chains in organic smectites and with the uncharged siloxane surface in the Fe(III)-smectite. The protonation of imidacloprid also enhanced the sorption on the Fe-smectites (Cox et al. 2001).

CONCLUSION

In the present study, a general-purpose isotherm model was derived, which was then analysed and validated with experimental data. The new isotherm provides a good description of the multilayer adsorp-

tion of compounds with different hydrophobicity. The adsorption capacity of the adsorbent and the adsorption energy, which is proportional to the K value of the isotherm, the size of the associates and the charge of the ions could be estimated using the model, which could help to elucidate the mechanism of the exchange and adsorption processes. The isotherm has the advantage of avoiding the errors observed for the Freundlich isotherm at high concentrations and for the Langmuir isotherm at low concentrations, making it suitable for the risk assessment for both ionic and molecular compounds.

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