Sorption of Tribenuron-methyl, Chlorsulfuron, and Imazamethabenz-methyl by Soils

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The sorption at 25 °C of the acidic herbicides tribenuron-methyl, chlorsulfuron, and imazamethabenz-methyl on three soils (clay content percentages ranging from 7.2 to 54.6, pH values ranging from 6.5 to 7.6, and organic matter percentages <1.5) was studied. Only imazamethabenz-methyl presented significant sorption in these soils. Sorption isotherms of imazamethabenz-methyl were well correlated to the Freundlich model, although linearity can be reasonably assumed. Desorption studies of this herbicide presented remarkable hysteresis and nonlinearity. Chlorsulfuron sorption was low, and tribenuron-methyl presented negligible or negative adsorption. The effect of the soil/solution ratio employed in batch experiments on the estimated distribution coefficient of the three herbicides was also investigated. By changing the soil/solution ratio from 1:3 to 1:1, an increase in the distribution coefficient of imazamethabenz-methyl and chlorsulfuron was observed. On the other hand, the distribution coefficient of tribenuron-methyl decreased within the same range of soil/solution ratios. Results indicate that these herbicides, but especially chlorsulfuron and tribenuron-methyl, could be mobile in the soils studied. The effect of the soil/solution ratio or hysteresis in desorption seems to be more important than the isotherm nonlinearity in modeling the transport of these hebicides in soil.

Keywords: *Sorption; soil; tribenuron-methyl; chlorsulfuron; imazamethabenz-methyl*

INTRODUCTION

Sorption of ionic and ionogenic organic contaminants has recently become the focus of much attention due to the effect of the soil and solvent properties on the extent of sorption of these chemicals in soils. In contrast to nonpolar organic contaminants, the ionic composition and concentration of the medium have been shown to have a profound impact on the sorption of organic acids and bases (McConnell and Lloyd, 1985; Weber, 1993; Pusino et al., 1994).

Castilla y León is the largest region of the European Community and holds one-fifth of the total area available for agriculture in Spain, with cereals being the main crop produced. Several acidic herbicides are being used in this region. Tribenuron-methyl (TRB) [methyl 2-[4-methoxy-6-methyl-1,3,5-triazin-2-yl(methyl)carbamoylsulfamoyl]benzoate] and chlorsulfuron (CHL) [1-(2-chlorophenylsulfonyl)-3-(4-methoxy-6-methyl-1,3,5-triazin-2-yl)urea] are two herbicides belonging to the sulfonylureas, a group that controls a wide range of weeds at very low application rates. Both herbicides are weak acids having pK_a values close to 3.6 and 5, respectively (Tomlin, 1994). Imazamethabenz-methyl (IMZ) [methyl 6-(4-isopropyl-4-methyl-5-oxo-2-imidazolin-2-yl)-*m*- (and -*p*)-toluate] is an effective postemergence herbicide, belonging to the imidazolinones, with a pK_a value of 2.9 (Tomlin, 1995). It is expected that these herbicides exist as the dissociated anion in most of our agricultural soils, since the pH of these soils usually exceeds by >2 units the pK_a of the aforementioned herbicides. Negatively charged adsorption sites are therefore not expected to contribute to the adsorption process, provoking an enhanced mobility of these chemicals in soils. Accordingly, information on the behavior of these herbicides is essential in predicting their effectiveness, as well as their potential for crop damage and environmental pollution.

The most commonly used method to study sorption–desorption of contaminants in soils has been the use of the batch equilibration technique for calculation of the coefficients of different sorption isotherms. The values of these coefficients can be used to obtain direct measurements of the parameter values in further transport experiments, especially for the retardation factor (Brusseau et al., 1989). Nevertheless, certain discrepancies have been reported between the sorption coefficients obtained in batch experiments and the values of the transport parameters obtained in column experiments (Brusseau and Rao, 1989). Differences in the experimental conditions (Koskinen and Cheng, 1983; Schrap et al., 1994) seem to be responsible for this disagreement. Among the different experimental variables, the soil/solution (s/s) ratio is perhaps the most important due to the wide range of soil/solution values present in experiments conducted under field conditions. This ratio also can be a major source of discrepancies between the values of the distribution coefficients obtained in batch experiments and those required for column experiments. Hence, it is interesting to investigate the effect of the soil/solution ratio on the herbicide sorption coefficients.

The principal objective of this study was to characterize the sorption of the three above-mentioned acidic

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herbicides on agricultural soils. Three typical cereal soils, covering the range of soil properties of the Castilla y León croplands, were selected. An IMZ sorption study on three soils of close texture was previously conducted (Cartón et al., 1997). In this paper we will extend the study to cropland soils showing higher textural differences, comparing the behavior of IMZ with that of two extensively used sulfonylureas. In addition, the effect of the soil/solution ratio on sorption was also investigated to determine if these results could be extrapolated to other experimental situations, particularly to solute transport experiments.

Table 1. Selected Chemical and Physical Properties of the Soils Used in This Study

	texture				
soil	% sand	% silt	% clay	OM[a] %	pH
A	88.8	4.0	7.2	0.6	6.5
B	71.1	8.6	20.3	1.1	7.5
C	19.2	26.2	54.6	1.5	7.6

[a] Organic matter.

MATERIALS AND METHODS

Soils. Soil samples were collected from the top 15 cm of the tilled surface soil (Ap horizons) of various locations in the Castilla y León region (Spain) to obtain a variety of physical characteristics. Three soils from the original set of soils sampled were selected as representatives of the typical soils in this region. This selection took into account the clay content of the soils, because this parameter is considered to be the most important in determining the amount of sorption of these herbicides [organic matter (OM) contents of the soils of Castilla y León are low and within a short range, usually below 1.5%]. Table 1 shows a description of selected physical and chemical properties of these soils. Although the clay content was the main parameter in the soil selection, the variation of OM content, shown in Table 1, follows the same tendency as clay content (A < B < C). In all cases, the soils used in this work were air-dried and passed through a 2 mm sieve.

Herbicides. Granstar (Dupont Ibérica), Glean (Dupont Ibérica), and Assert (Cyanamid Ibérica), were the commercial pesticides used in this research. Figure 1 shows the molecular structures of their active ingredients, TRB, CHL, and IMZ, respectively. A description of the main characteristics of these herbicides (Tomlin, 1995) and relevant analytical data can be found in Table 2. In addition to pK_a, other properties are influential in determining the sorption of these herbicides in soils. Water solubility shows significant differences among these three herbicides (TRB ≫ CHL ≫ IMZ). Half-life is reported to be IMZ > CHL > TRB (Tomlin, 1995). In the case of TRB, the short half-life has practical consequences in the experimental methodology, which will be described under Analytical Methods.

Experimental Procedure. All herbicide solutions were made in a 0.01 M $CaCl_2$ matrix to hold constant the ionic strength of the soil solution. Other effects of this salt were discussed by Koskinen and Cheng (1983). Additionally, blanks with initial solution without soil were prepared to account for possible losses due to adsorption on PE bottles or filters, volatilization, or hydrolisis.

Kinetic experiments were carried out by adding 10 g of 11.5 μg/g herbicide solution to 5 g of soil in centrifuge tubes. The tubes were introduced to a thermostatic bath controlled to 25.0 ± 0.1 °C and magnetically stirred over different periods of time (0.25, 0.5, 1, 2, 3, 14, and 45 h, respectively). The tubes' contents were then centrifuged at 3500 rpm for 10 min, and a fraction of the supernatant clear liquid was filtered and stored in vials at temperatures <4 °C until the analysis. The amount of chemical in the sorbed phase was estimated by a mass balance

$$Sq = L(c_0 - c - D) \qquad (1)$$

where S and L are, respectively, the masses (g) of soil and solution, q is the concentration in the sorbed phase (μg/g), c_0 is the initial concentration in solution (μg/g), c is the final concentration in solution (μg/g), and D (μg/g) is the term accounting for degradative losses in the blank experiments. In this work we will use also $c_0{}^*$ to represent $c_0 - D$ (i.e., the final concentration in the blank).

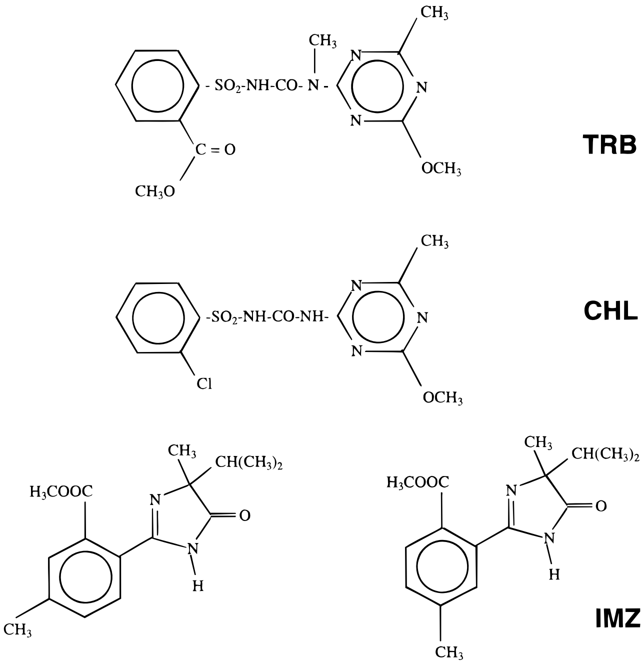

Figure 1. Structures of the molecules of the three herbicides studied in this work.

Herbicide solutions containing 0.5, 1, 2, 4, 6, 8, 10, and 12 μg/g herbicide were prepared for the sorption equilibrium experiments, using a batch equilibration method. Samples of 10 g of herbicide solution and 5 g of air-dried soil were placed in centrifuge tubes, thermostated, and magnetically stirred during the equilibration time determined in the previous kinetic study. Samples for analysis were obtained as in the kinetics experiments, and the amounts of sorbed herbicide were estimated by the mass balance given by eq 1. Equilibrium adsorption results were correlated to the linear and Freundlich equations, which are the most extended isotherms used in solute transport models

$$q = K_D c, \qquad q = K_F c^{1/n} \qquad (2)$$

where q is the soil pesticide concentration (μg/g) and c is the concentration of the pesticide in the solution phase (μg/g). K_D, K_F, and $1/n$ are parameters for each isotherm.

Desorption of IMZ was conducted at the end of each adsorption stage by replacing a fraction of the clear supernatant liquid with a 0.01 M $CaCl_2$ pesticide-free solution, keeping the soil/solution ratio unaltered. The initial pesticide solution concentration was diluted so that, after a new equilibration time, a fraction of the retained pesticide would be extracted from the soil, resulting in a new equilibration point. This operation was repeated four times for each desorption isotherm. No desorption experiments were carried out with TRB and CHL because of the small sorbed amounts obtained in the sorption experiments.

The effect of the soil/solution ratio on the sorption equilibrium was studied by preparing 4 μg/g herbicide solutions on 0.01 M $CaCl_2$. Following the above adsorption procedure, different quantities of soil and solution were placed in the tubes (4 g/4 g, 4 g/8 g, and 2 g/6 g), resulting in 1:1, 1:2, and 1:3 soil/solution ratios. The weight of soil was limited to 4 g to maintain the agitation system under the optimum condi-

Table 2. Selected Properties of the Herbicides and Relevant Analytical Data

herbicide	water solubility (g/L, 25 °C, pH 7.0)	pK_a	field dissipation (days, half-life)	HPLC analysis: mobile phase (% acetonitrile/% water, v/v)	HPLC analysis: flow (mL/min)	HPLC analysis: λ (nm)
tribenuron-methyl	280[a]	4.0–5.0	1–7	48:52 (H_3PO_4 0.01%)	1.0	232
chlorsulfuron	27.9	3.60	28–56	45:55 (H_3PO_4 0.01%)	1.0	210
imazamethabenz-methyl	1.370 (meta) 0.857 (para)	2.90	30–276	30:70	1.0	210

[a] pH 6.

tions. Among the several magnitudes that can be used to represent the amount of sorption (q, K_D, c/c_0*, etc.) the estimation of the ratio c/c_0* offers several advantages from a practical point of view. Rearranging eqs 1 and 2 gives the relation between K_D and c/c_o* by

$$K_D = \frac{(1 - c/c_0^*)}{c/c_0^*}\,\frac{L}{S} \qquad (3)$$

If c_0* is determined as described under Analytical Methods, then the sorbed amount q can be obtained from eq 1, as we did in the kinetics and sorption–desorption equilibrium experiments. Nevertheless, the determination of c/c_0* is rapid when it is performed by a comparison of the peak areas of a sample and blank, respectively, and the estimation of K_D is carried out using eq 3, once c/c_0* is known. This assumes a constant response factor of the HPLC system, which is very reasonable under most of the experimental conditions. Following this procedure, a considerable reduction of the number of analyses and an improvement on repetitivity were achieved.

Analytical Methods. Herbicide concentrations in the kinetics and sorption–desorption experiments were determined by HPLC, performed with an LDC-Analytical 4100 series apparatus (LDC Analytical, Riviera Beach, FL) composed of a vacuum mobile phase degasifier, alternative pump CM4100, automatic injector, and UV detector SM4100. The column was a C_{18} Waters Nova-Pak, 150 × 3.9 mm, 5 μm particle diameter. The studies on the effect of soil/solution ratio were performed using a DAD:HP-1100 detector and an HP-1050 HPLC system (Hewlett-Packard, Madrid, Spain). The working conditions are shown in Table 2. Standard solutions were prepared using chromatographically pure herbicides (Ehrenstorfer GmbH, Augsburg, Germany) in a 0.01 M $CaCl_2$ matrix. The standard solutions were periodically replaced to ensure no degradation. The limits of detection (estimated as 3 times the background noise) were 0.04 μg/g for TRB and CHL and 0.05 μg/g for IMZ (Atienza et al., 1996: Bernal et al., 1997).

Figure 2 shows the chromatograms of three samples after equilibration with the soil. The half-life of the tribenuron-methyl was too short to perform sorption experiments (because ≈24 h was required to reach equilibrium). For this reason, we selected a more stable metabolite, which is the form in which TRB is usually present in our soils. Figure 3 shows the UV spectra of TRB and the selected metabolite. Standards of TRB remained unaltered at −20 °C in methanol, but they were transformed in the metabolite in <24 h.

RESULTS AND DISCUSSION

Kinetics. TRB and CHL presented negligible or negative adsorption in the kinetics experiments performed at the ambient pH of the soils. These results will be confirmed in further adsorption equilibrium experiments, shown in the next section. The kinetic experiments for IMZ suggest that this herbicide adsorbs in two steps: an initial fast adsorption during the first few hours, followed by a slower adsorption. The performance of IMZ is similar to that found by Cartón et al. (1997) for close experiments. Pseudoequilibrium was reached in <24 h.

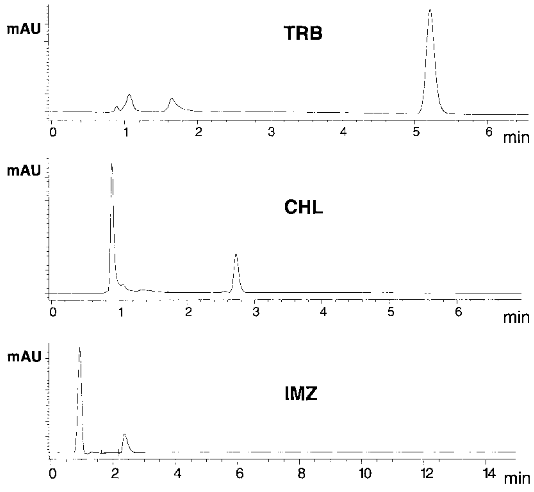

Figure 2. Chromatograms of the soil extracts after equilibration.

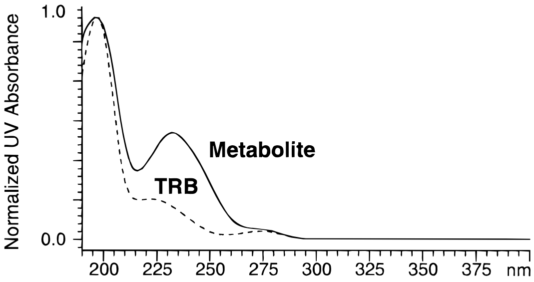

Figure 3. UV spectra of tribenuron-methyl and its stable metabolite.

Sorption. In all cases, only IMZ presented adsorption in the experiments carried out at the ambient soil pH. The fitted parameters, corresponding to eq 2, were obtained by the least-squares minimization of the linearized form of these equations. Their values and deviations are summarized in Table 3. The estimation followed the Dixon criterium (Miller and Miller, 1988) to eliminate the outlying values. From the calculated values of the sum of the squares (SSQ) for the linear and Freundlich models, it is obvious that the Freundlich model leads to the lowest errors. Nevertheless, as there are no significant differences between the isotherms fitted to the Henry and Freundluch models, linearity can be reasonably assumed for the sorption process, the relative error in the estimation always being <5%.

IMZ desorption studies showed a marked hysteresis and high nonlinearity. Several authors (Brusseau and Rao, 1989; Schrap et al., 1994; among others) reported this phenomenon to be, partially, an apparent effect due to a failure to reach equilibrium and degradative losses. In our case, once ensured that degradative losses were negligible for the conditions of these experiments,

Table 3. Henry and Freundlich Constants and Coefficients for IMZ Adsorption and Desorption

		Henry		Freundlich		
soil	isotherm	K_D	SSQ	K_F	$1/n$	SSQ
A	adsorption	0.170 (±0.005)	0.049	0.19 (±0.01)	0.96 (±0.04)	0.048
	desorption 1			0.22 (±0.08)	0.90 (±0.30)	0.006
	desorption 2			0.47 (±0.01)	0.32 (±0.02)	0.001
	desorption 3			0.86 (±0.02)	0.21 (±0.02)	0.008
	desorption 4			1.15 (±0.02)	0.22 (±0.02)	0.005
B	adsorption	0.295 (0.011)	0.236	0.40 (±0.02)	0.86 (±0.03)	0.097
	desorption 1			1.04 (±0.01)	0.20 (±0.01)	0.004
	desorption 2			1.39 (±0.04)	0.20 (±0.02)	0.006
	desorption 3			1.77 (±0.04)	0.21 (±0.01)	0.009
C	adsorption	0.99 (0.03)	0.623	1.40 (±0.08)	0.81 (±0.04)	0.447
	desorption 1			1.81 (±0.01)	0.16 (±0.01)	0.027
	desorption 2			2.59 (±0.05)	0.16 (±0.03)	0.058
	desorption 3			2.71 (±0.06)	0.41 (±0.02)	0.016
	desorption 4			2.82 (±0.14)	0.49 (±0.03)	0.070

a true hysteresis effect seems to be responsible for the desorption behavior of IMZ. Table 3 shows the values of the Freundlich parameters. The low values of the $1/n$ parameter are representative of this low linearity, the reason for which the Henry isotherm could not represent the desorption processes.

TRB and CHL sorption experiments showed negligible or negative adsorption (i.e., the herbicide concentration in suspension was slightly higher than the initial added concentration). The lack of herbicide on the starting soils was checked by direct estimation of the soil-phase concentration after a solvent extraction process of the herbicide, following the procedure described by Bernal et al. (1997). The phenomenum of negative adsorption has been reported earlier in studies concerning the sorption of acidic chemicals (Weber et al., 1965).

Borggaard and Streibig (1989) reported negligible adsorption of CHL at pH >7−8, and similar results were found by Shea (1986) and Walker et al. (1989). Additionally, experiments performed using soil thin-layer chromatography showed that CHL mobility was positively correlated with pH (Mersie and Foy, 1986). The negative adsorption of CHL seems to be reasonable, since this chemical at the soil pH values (see Table 1) is predominantly in the anionic form and would be repelled by the negatively charged clay particles. Since we have considered a degradative term in the estimation of the soil-phase concentration (eq 1), the resulting negative adsorption for TRB and CHL should not be attributed to a deficient mass balance calculation.

Similar negative adsorption results were found in the TRB sorption experiments. At pH 6.5−7.6 this herbicide is predominantly in the anionic form. Therefore, the explanation given for CHL may be valid in interpreting this behavior. Although data for this recently developed herbicide are scarce in the literature, pH seems to play a major role in TRB sorption (Riise, 1994).

Two effects should be considered in the interpretation of the role of the pH of the soil solution. First, the molecular forms of both CHL and TRB herbicides are favored as the pH decreases, promoting an increase of their sorption on soils. Thus, it is well-known that the octanol/water partition coefficients and water solubilities of sulfonylureas are functions of pH. In addition to this effect, the rate of hydrolysis of sulfonylureas is pH dependent (Hay, 1990), and specifically, CHL degradation has been reported to be pH dependent (Joshi et al., 1985; Walker and Welch, 1989; Walker et al., 1989). Despite this possible explanation, based upon the two coupled effects, the study of the effect of the pH on the sorption of TRB and CHL will not be considered here beyond the pH range of the soils shown in Table 1. That study would require more sophisticated analytical techniques to identify and quantify the different forms in which the herbicides are present and their main metabolites. In our experiments, the dependence of the absorbance/concentration ratio on pH was avoided by selection of the appropriate wavelength (Table 2).

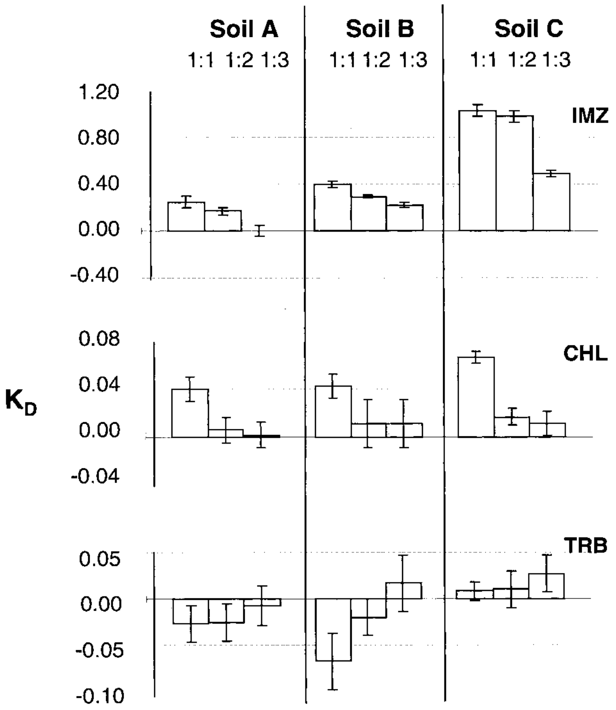

Figure 4. Effect of the soil/solution ratio on the sorption of the three herbicides arranged by the magnitude of the sorption.

Soil/Solution Ratio. The sorbed amount of IMZ increased (i.e., K_D increased) as the soil/solution ratio increased (Figure 4). The adsorption of this herbicide increased, at each soil/solution ratio, while the clay content of the soil increased. This is reasonable, since as the soil/solution ratio increases, there are more available sites for sorption. This effect was less evident in the experiments performed with CHL, due to the low sorption of this herbicide in the studied soils. Whereas the typical values for c/c_0* in the IMZ experiments ranged from 0.5 to 0.99, CHL experiments were in a considerably shorter range, from 0.96 to 0.99. The behavior of TRB (or, more precisely, its metabolite) was different. Although there were no significant differences among different soil/solution ratios, the general trend was a decrease in sorption as the soil/solution ratio increased. The K_D values for soils A and B were close to 0 and negative in some of the experiments. As the soil/solution ratio increases, the anionic exclusion (responsible for negative sorption) is expected to increase, in agreement with the results obtained for these soils.

The reason for changes in the adsorption coefficients K_D and K_F with changes in the soil/solution ratio have not been elucidated in the literature, and the results concerning experiences with weak acids are inconsistent: Farmer and Aochi (1974) found that the value of K_F for picloram increased when the ratio changed from 1:5 to 1:2, whereas Koskinen and Cheng (1983) observed a decrease in K_F by changing the ratio from 1:3.3 to 1:1. The increase of the soil/solution ratio (in a $CaCl_2$ solvent matrix) affects not only the ionic strength of the supernatant but other slurry properties as well. The pH, ionic composition, and concentration of the solvent, the shape of the diffuse double layer in the vicinity of soil particles, and the nature and surface character of the organic matter may be included among them. Thus, it is reasonable to expect a change in the amount of adsorption in addition to the change in the solubility of the ionic or ionogenic compounds with the soil/solution ratio.

SUMMARY AND CONCLUSIONS

Tribenuron-methyl presented a high degree of hydrolysis, and a more stable metabolite was used for this study. The UV spectrum of this metabolite was used to identify the form in which TRB was present in the soil extracts. This herbicide showed negligible or negative adsorption in all of the soil/solution ratios (from 1:3 to 1:1) investigated in soil with low OM and clay contents, whereas the sorption was higher in soil C, with higher OM and clay contents. CHL exhibited slight sorption in all soils. Due to this low sorption, kinetics studies did not reveal valuable information. Increasing the soil/solution ratio from 1:3 to 1:1 increased the distribution coefficient in the three soils, following a similar trend. Nevertheless, despite some variations in the adsorption coefficients, this study demostrated that, in general, TRB and CHL were not strongly adsorbed, and consequently, they must be expected to be mobile in the soils considered in this work.

Imazamethabenz-methyl presented adsorption under different soil/solution ratios. Although a slight nonlinearity was observed in the sorption processes of this herbicide, isotherms can be reasonably assumed as linear. The desorption processes showed a high degree of hysteresis and nonlinearity, with Freundlich $1/n$ parameters in the range 0.16−0.90. Adsorption was higher in soil with higher clay and OM contents.

The use of K_D in modeling the transport of these herbicides in soil seems to be a reasonable assumption. Nevertheless, the nonsingularity of sorption−desorption isotherms requires the use of different coefficients for the sorption and desorption processes, respectively, and the implementation of nonlinear isotherms for desorption (such as the Freundlich model). Additionally, the effect of the soil/solution ratio on the value of the distribution coefficient K_D must be considered to avoid possible discrepancies between the batch and the transport experiments.

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