

Adsorption and Desorption of Triasulfuron by Soil

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The adsorption and desorption of the herbicide triasulfuron [2-(2-chloroethoxy)-*N*-[[[4-methoxy-6-methyl-1,3,5-triazin-2-yl]amino]carbonyl]benzenesulfonamide] by three soils, soil organic matter (H⁺ and Ca²⁺-saturated), and an amorphous iron oxide were studied. Adsorption isotherms conformed to the Freundlich equation. It was found that pH is the main factor influencing the adsorption in all of the systems. Indeed, the adsorption on soils was negatively correlated with pH. The highest level of adsorption was measured on soils with low pH and high organic carbon content. Moreover, it was found that humic acid is more effective in the adsorption compared with calcium humate (the pH values of the suspensions being 3.5 and 6, respectively). Experiments on amorphous iron oxide confirmed the pH dependence. Desorption was hysteretic on soils having high organic carbon content.

KEYWORDS: Triasulfuron; herbicide; adsorption; desorption; organic matter; montmorillonite; iron oxide

INTRODUCTION

Triasulfuron, the active ingredient of Amber (Ciba-Geigy Corp.), is a relatively new selective sulfonylurea herbicide (**Figure 1**) used at very low rates (15–25 g of active ingredient/ha) for weed control in cereals (1). Degradation and retention processes are among the most important factors influencing the fate of an agrochemical in soil environment. Similarly to other sulfonylureas, triasulfuron is more persistent in neutral or weakly basic media than in acidic media (2). Studies on the hydrolysis kinetics of triasulfuron revealed that over a wide range of pH the main mechanism pathway is the cleavage of the sulfonylurea bridge (3). Photolysis can be an alternative to the chemical hydrolysis of triasulfuron. Pusino et al. (4) proposed a mechanism that accounts for the formation of triasulfuron photo-products. The degradation of triasulfuron in soil was observed under both sterile and nonsterile conditions (5, 6). The adsorption and degradation of triasulfuron were observed on aluminum smectites (7) and iron and aluminum montmorillonites (8).

Because of the potential mobility of triasulfuron due to its acidic nature (9), extensive research was done on the mobility, leaching, and persistence of this chemical in soil by means of field and laboratory studies. The results are often contradictory. In a field study on leaching and degradation in an alkaline sandy loam soil [pH 8.5; organic matter (OM) 1.1%, in the 0–10 cm layer], it was found that triasulfuron, applied in May, leached beyond 50 cm to a 21% extent by late winter under natural rainfall conditions and leached almost completely in the following season between late autumn and midspring (10). On

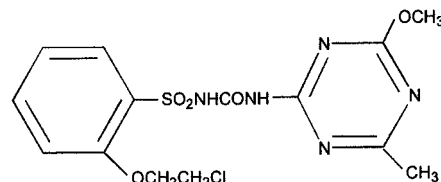


Figure 1. Structure of triasulfuron.

the contrary, in another field study triasulfuron remained in the top layer and distributed in the range 0–40 cm, respectively, in a loamy soil [pH 7.3; organic carbon (OC) 2.1%] and a sandy soil (pH 7.0; OC 1.0%) after the addition of 90 mm of water (11). The different results from field studies may be mainly ascribed to the different hydric regimes and the different soil constitutional characteristics. In fact, in a laboratory study on a quarry sand enriched with increasing amounts of peat (pH 5.7), the movement of triasulfuron was directly related to the amount and frequency of rain and inversely related to the OM content (12).

In a further laboratory study of triasulfuron adsorption K_d values of 1.05 and 0.26 were measured on a sandy loam soil (pH 7.1; OC 0.54%) and a loamy sand soil (pH 8.7; OC 1.6%), respectively (6). Similarly, both field and laboratory mobility studies on a silty clay soil (pH 8.1; OM 1.8%) and in a sandy loam soil (pH 6.5; OM 1.2%) substantiated that triasulfuron is more mobile in alkaline than in acidic soil (13, 14). These findings are consistent with the enhancement of mobility of triasulfuron with increasing pH values. On the contrary, in a study on columns, the mobility of triasulfuron in a Norfolk loam soil (pH 5.9; OM 1.1%), a Rion loamy sand soil (pH 6.7; OM 0.9%), a Farnum sandy loam soil (pH 6.7; OM 1.9%), and a Webster silt loam soil (pH 7.4; OM 6.0%) followed the order

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Table 1. Selected Physical and Chemical Properties of Investigated Soils

property	Fornace	Bassona	Monghidoro
OM (%)	1.3	4.4	6.3
clay (%)	18.0	61.0	20.0
sand (%)	57.0	21.0	58.0
silt(%)	25.0	18.0	22.0
pH ^a	7.8	7.6	5.4
Fe ^b	0.6	1.0	0.8
CEC ^c	17.5	51.2	23.5

^a Soil/water = 1:1. ^b Oxalate extractable (g/100 g). ^c mequiv/100 g.

Table 2. Mineralogical Composition of Soils Investigated

composition	Fornace	Bassona	Monghidoro
smectite (%)	12	46	3
illite (%)	42	32	
chlorite (%)		9	34
kaolinite (%)	24	7	3
quartz (%)		6	53
potash feldspar (%)	22		7

Norfolk (R_f 0.52) \approx Rion (R_f 0.48) > Farnum (R_f 0.40) > Webster (R_f 0.26) (15). In this study the mobility of triasulfuron was inversely related to the OM content; however, no relationship between mobility and pH could be found. These discrepancies suggest that it is not easy to understand how and to what extent soil characteristics and constituents affect the behavior of triasulfuron in soil. Moreover, no information is available on the reversibility of the adsorption process.

In this work we have investigated the adsorption–desorption behavior of triasulfuron by three soils with different physico-chemical properties as a function of pH. The adsorption of triasulfuron on humic acid and iron oxide was also examined.

MATERIALS AND METHODS

Materials. Triasulfuron (99.5% purity) was supplied by Ciba-Geigy, Saronno, Italy. Its purity was checked by high-performance chromatography (HPLC). All of the solvents were of HPLC grade (Carlo Erba Reagenti, Milano, Italy) and were used without further purification.

Three Italian soils, a sandy loam Entisol from Fornace (Emilia-Romagna), a clay loam Vertisol from Bassona (Emilia-Romagna), and a loamy sand Inceptisol from Monghidoro (Emilia-Romagna), were examined. The samples were air-dried and sieved to <2 mm. The particle size distribution was measured by Purdue University Soil Testing Laboratory using the pipet method (16). The organic carbon content was determined according to the modified Walkley–Black (17) method. The cation exchange capacity (CEC) was determined according to the procedure of Hendershot and Duquette (18). Ammonium oxalate-soluble Fe was extracted according to the method of Schwertmann (19). Soil pH was determined on slurries with a soil/water ratio of 1:1. Selected physicochemical properties of the soils are listed in **Table 1**. Clay mineralogy was determined by X-ray diffraction (XRD) analysis, see **Table 2**.

Hydrated ferric oxide was prepared by the addition of an equivalent amount of FeCl₂ to 250 mL of 2 N KOH under rapid stirring (20). The fresh precipitate was immediately washed with distilled water and dried under vacuum. It was amorphous according to X-ray analysis and completely soluble in ammonium oxalate (pH 3).

Humic acid was obtained from Monghidoro soil according to the procedure of Stevenson (21). After precipitation, it was centrifuged, redissolved, and precipitated three times, then dialyzed against distilled water until salt-free, and finally freeze-dried.

Adsorption on Soil. The adsorption of triasulfuron was determined at 25 ± 2 °C. In general, triplicate samples of 5 g of air-dried soil sieved through a 2-mm mesh screen were equilibrated in polyallomer

centrifuge tubes with 10 mL of aqueous herbicide solution (the range 5.8–29.2 μM was chosen on the basis of herbicide solubility). The tubes were shaken (end over end) for 24 h. Generally, 95% of the adsorption took place within the first 5 h. After equilibration, the suspension was centrifuged at 19000g for 15 min; the supernatant was then pipetted off and analyzed immediately. The amount adsorbed by soil was calculated from the difference between the initial and final concentrations of triasulfuron in solution. The effect of soil solution pH on triasulfuron adsorption was examined by adding HCl or NaOH to soils and equilibrating overnight with 10 mL of a 11.7 μM solution of herbicide. The amount adsorbed was quantified according to the same procedure described for natural soils.

Adsorption on Humic Acid and Iron Oxide. Duplicate samples of humic acid (50 mg), calcium humate, and iron oxide (50 mg) were equilibrated in polyallomer centrifuge tubes with 10 mL of aqueous herbicide solution. Triasulfuron concentrations before equilibration ranged from 5.8 to 29.2 μM. The tubes were shaken (end over end) for 12 h. After centrifugation, the supernatant solution was removed and analyzed.

Desorption. After the adsorption process described above, 5 mL of the supernatant solution was withdrawn and the amount of adsorbed herbicide was calculated. The remaining slurry was again brought to 10 mL by the addition of 5 mL of water, equilibrated for 24 h, and centrifuged. These steps (supernatant withdrawing and replacing followed by re-equilibrating) were repeated four times consecutively. The concentration of herbicide in the desorption solutions was determined, and the amount of herbicide adsorbed on the soil after each desorption step was calculated by difference. Each level of dilution thus provided one point to the desorption isotherms.

HPLC Analyses. The concentration of triasulfuron was determined by HPLC. The system was assembled as follows: a Waters 510 pump equipped with a Waters 490 UV–vis programmable detector operating at 224 nm, Breeze chromatography software, and a Spherisorb C₈ analytical column (5 μm, 4.6 × 250 mm) eluting with acetonitrile plus water (40 + 60 by volume, pH 2.7) at a flow rate of 1 mL min⁻¹. The retention time for triasulfuron under the chromatographic conditions described previously was 10.5 min. The quantitative determination of triasulfuron was performed by using an external standard. Calculations were based on the average peak areas of the external standard. The detection limit for triasulfuron was 0.05 mg L⁻¹, as calculated from the concentration of herbicide needed to obtain a detector response approximately twice the background signal.

Data Analysis. Adsorption–desorption data were fit to the logarithmic form of the Freundlich equation, $\log C_s = \log K_f + 1/n \log C_e$, where C_s (in μmol kg⁻¹ units) is the amount of herbicide adsorbed by soil, humic acid, and iron oxide, respectively, C_e (in μM units) is the equilibrium concentration in solution, and $\log K_f$ and $1/n$ are empirical constants representing the intercept and the slope of the isotherm, respectively. The hysteresis coefficient, H , for the adsorption–desorption isotherms was calculated according to the formula $H = (1/n_{des})/(1/n_{ads})$, where $1/n_{ads}$ and $1/n_{des}$ are the Freundlich slopes obtained for the adsorption and desorption isotherms, respectively (22).

RESULTS AND DISCUSSION

Adsorption Studies. The adsorption isotherms of triasulfuron on three test soils are shown in **Figure 2**. The empirical Freundlich equation fit well the behavior ($r \geq 0.987$). The calculated constants K_f and $1/n$ and the coefficients (r) for the linear fit are given in **Table 3**. In all cases the isotherms are nonlinear with slopes ($1/n$) of <1, resembling the L-type curve described by Giles et al. (23). This shape suggests a relatively high affinity of the herbicide for the adsorbing sites (**Figure 2**). The K_f values indicate that the adsorption is highest on Monghidoro followed, in the order, by Bassona and Fornace soils, which is also the order of decreasing value of both organic matter content and pH of the soils.

Despite the small number of samples, linear regression analyses between selected soil properties and the Freundlich constant, K_f , were performed. The highest correlation coefficient,

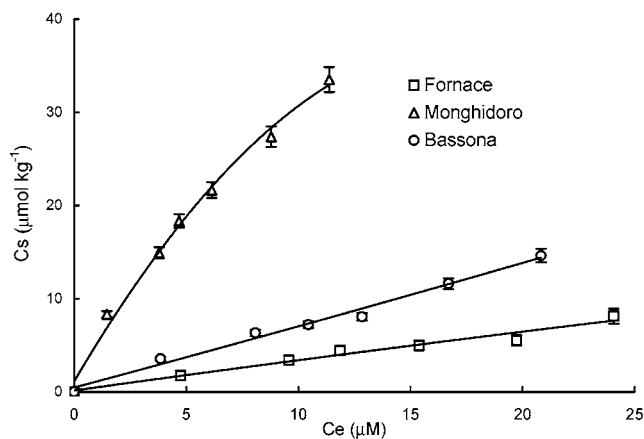


Figure 2. Adsorption isotherms on soil.

Table 3. Freundlich Parameters for the Adsorption of Triasulfuron on Soil and on Humic Acid and Iron Oxide

sample	pH ^a	K_{ads}^b	$1/n_{ads}^b$	r
Monghidoro soil	5.4	6.34 (± 0.11)	0.68 (± 0.02)	0.999
Bassona soil	7.6	1.13 (± 0.22)	0.81 (± 0.03)	0.988
Fornace soil	7.8	0.46 (± 0.13)	0.88 (± 0.05)	0.987
humic acid	3.5	41.00 (± 1.35)	0.93 (± 0.07)	0.971
humic acid	4.6	28.97 (± 0.98)	0.96 (± 0.04)	0.969
calcium humate	6.0	3.60 (± 0.12)	1.19 (± 0.07)	0.963
iron oxide	3.7	41.05 (± 2.27)	0.76 (± 0.10)	0.943
iron oxide	6.7	4.46 (± 0.27)	0.86 (± 0.08)	0.933

^a pH of aqueous suspension. ^b The value in parentheses is the 95% confidence interval.

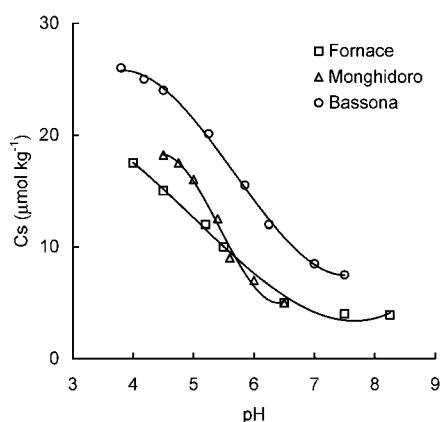


Figure 3. Effect of pH on the adsorption of triasulfuron on soil.

$r = 0.9995$, was obtained when pH was used as the soil property (significant at $P = 0.02$). There is no significant correlation ($P < 0.05$) between K_f and other soil properties. The correlation between K_f and pH is negative, indicating that the extent of adsorption increases as pH decreases. The calculated equation is

$$K_f = 19.80 - 2.47 \text{ pH}$$

Multiple correlation analyses were not significant.

The dependence of triasulfuron adsorption on soil solution pH was also tested (Figure 3). Actually, it was found that the herbicide adsorption increases as the pH of the soil decreases, according to the general trend observed for sulfonylureas (24, 25). It is interesting to note that, if the same pH value is considered, Bassona soil shows the greatest adsorption capacity (Figure 3). This soil has the highest clay (mainly of the smectite

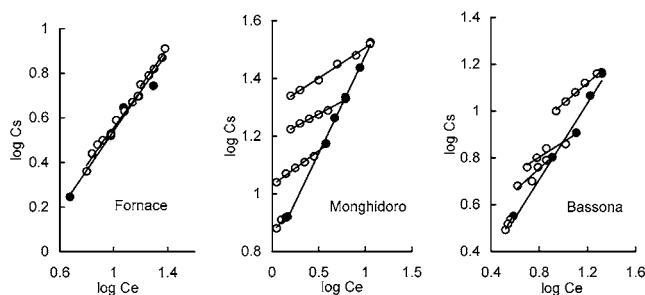


Figure 4. Freundlich adsorption-desorption for triasulfuron: (●) adsorption; (○) desorption.

type, see Table 2) and iron oxalate extractable content together with a high OC content (Table 1). The effect of expanding layer silicate clays such as smectites on the adsorption of triasulfuron was already reported (8). Montmorillonite clay minerals are active in the adsorption of triasulfuron, and the adsorption of triasulfuron on clay is affected by the type of exchangeable cation. Indeed, the affinity of triasulfuron for smectites increases with increasing acidity of the hydration water of the exchangeable cation.

Because soil pH is responsible for the dissociation or protonation processes of both the herbicide and the adsorbent surfaces, the effect of pH on the triasulfuron adsorption on humic acid and iron oxide was investigated.

The extent of triasulfuron adsorption on humic acid is rather high at low pH and decreases with increasing pH of the suspension. The slopes of the adsorption isotherms ($1/n$) vary from 0.93 at pH 3.5 to 1.19 at pH 6.0 (Table 3). Therefore, the shape of isotherms changes from L-type (high affinity) at low pH to S-type (low affinity) at high pH (23). This indicates that the availability of the adsorption sites to triasulfuron molecules becomes reduced as the pH value is increased.

Triasulfuron is a weak acid with a pK_a value of 4.5 (9), due to the dissociation of the NH group within the sulfonamide bridge. The high extent of adsorption measured at low pH values may be due to the lack of charge and higher lipophilicity of the neutral molecular species of the herbicide. The increase of solution pH increases the fraction of negatively charged triasulfuron species. At pH 6 only a negligible amount of neutral herbicide species is expected. Therefore, the repulsion of the anionic molecules with the negatively charged surfaces of the organic matter (pK_a value around 5; 21) makes unfavorable the adsorption. This trend accounts for the almost insignificant correlation ($P = 0.35$) observed between K_f and the organic carbon content of soil.

Moreover, a low solubility of triasulfuron in water at low pH could contribute to the high adsorption level, because the retention of a pesticide onto organic surfaces is often inversely correlated to its water solubility. Indeed, the solubility of triasulfuron in water is 32 mg L⁻¹ at pH 5 and 815 mg L⁻¹ at pH 7 (data from the manufacturer).

Experiments on amorphous iron oxide substantiated the same pH dependence as described above (Table 3), although this adsorbent is the only colloid surface that exhibits a significant affinity to the triasulfuron molecule at pH values near neutrality (6.7). The zero point of charge (ZPC) of amorphous iron oxides occurs generally in the pH range 7.5–9 (26). Therefore, below ZPC, iron oxide surfaces, being positively charged, are able to interact with triasulfuron anionic species.

Desorption Studies. The Freundlich desorption parameters for triasulfuron determined on the test soils are given in Table 4. The hysteresis coefficient, H , for the adsorption and desorp-

Table 4. Freundlich Parameters and Hysteresis Coefficients^a for the Desorption of Triasulfuron by Soil

	C_i^b (μM)	Fornace ^c	Bassona ^c	Monghidoro ^c
K_{des}	5.8	nd	1.07 (± 0.10)	7.37 (± 0.21)
$1/n_{\text{des}}$			0.90 (± 0.07)	0.34 (± 0.11)
r			0.984	0.952
H				0.50
K_{des}	11.7	0.48 (± 0.08)	2.41 (± 0.18)	10.69 (± 0.31)
$1/n_{\text{des}}$		0.88 (± 0.05)	0.46 (± 0.04)	0.24 (± 0.05)
r		0.943	0.958	0.989
H		1.00	0.57	0.35
K_{des}	17.8	0.52 (± 0.06)	3.49 (± 0.11)	15.47 (± 0.18)
$1/n_{\text{des}}$		0.88 (± 0.04)	0.54 (± 0.09)	0.17 (± 0.03)
r		0.979	0.973	0.977
H		1.00	0.67	0.25
K_{des}	29.2	0.53 (± 0.05)	3.93 (± 0.08)	19.76 (± 0.14)
$1/n_{\text{des}}$		0.85 (± 0.03)	0.44 (± 0.08)	0.21 (± 0.02)
r		0.988	0.993	0.997
H		0.96	0.54	0.31

^a Hysteresis coefficient $H = (1/n_{\text{des}})/(1/n_{\text{ads}})$. ^b Initial concentration. ^c The value in parentheses is the 95% confidence interval.

tion isotherms (Table 4) is a measure of the extent of hysteresis in the desorption. A value of 1 means that the desorption proceeds as fast as adsorption does and no hysteresis occurs. For all equilibrium points, triasulfuron desorption from the Fornace sandy loam soil is almost completely reversible, practically with unitary H coefficient values. Fornace soil exhibits a lower adsorption capacity and no hysteresis effect, which means that the small amount of adsorbed herbicide is readily desorbed. This behavior could be attributed to its high sand content and moderate organic carbon content. In the more sorptive Bassona and Monghidoro soils, some hysteresis is observed, as indicated by the low $1/n_{\text{des}}$ and H values. For these two soils, the hysteresis coefficients are not greatly dependent on the initial amount of triasulfuron adsorbed, although there is an increase in K_{des} as the initial concentration of triasulfuron increases. The highest hysteresis effect (lowest H) is observed in Monghidoro soil, which is the most effective in retaining triasulfuron, presumably because of the highest organic carbon content and lowest pH. It is expected that the matrix of polymeric organic colloid may be more H-bonded and have finer pores than under higher pH conditions. It is worth noting that desorption hysteresis is often an artifact due to several phenomena, such as chemical or biological degradation or non-equilibrium conditions (27). However, according to preliminary studies, the equilibration was complete within 20 h. In addition, over the pH range investigated (5.4–7.8) chemical degradation is negligible, as revealed by published kinetic data (3). Accordingly, at the end of each desorption cycle the degradation products were below the analytical detection limit.

Conclusions. This investigation clearly demonstrated that the adsorption of triasulfuron on soil and soil colloids is largely influenced by pH. At pH >6 the positively charged oxide surfaces are the most effective in triasulfuron retention. Humic matter is an important adsorbent primarily at pH values <6, when both triasulfuron and humic acids are partly in the undissociated form. Literature data indicate that the affinity of triasulfuron for smectites increases with increasing acidity of the hydration water of the exchangeable cation (8). Most likely clay and organic components contribute to the adsorption capacity to a higher extent in the acidic pH region, whereas iron oxides contribute more at higher pH values. Therefore, soils with low pH and rather high contents of amorphous oxides and

organic matter will retain triasulfuron to the greatest extent and resist leaching of the compound.

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