

# Adsorption of Triclopyr on Soil and Some of Its Components

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The adsorption of triclopyr [(3,5,6-trichloro-2-pyridinyl)oxy]acetic acid by homoionic clays, soil organic matter (H- and Ca-saturated), and seven soils was studied. The extent of adsorption on clays decreased in the order of decreasing acidity of the saturating cations. The H-organic matter was more active in triclopyr adsorption than Ca-organic matter and Ca-clay, whereas studies on mixtures of clay and organic matter indicated that the interaction between these two soil components reduced the surfaces available for triclopyr adsorption. Adsorption isotherms conformed to the Freundlich equation. It was found that in soil the Freundlich adsorption constant decreased linearly with organic matter ( $r = 0.930$ ) and adsorption decreased with increasing pH. Multiple-regression analysis indicated that  $k_f$  values were better correlated if cation-exchange capacity and pH ( $r = 0.989$ ) were simultaneously taken into account.

## INTRODUCTION

Triclopyr, [(3,5,6-trichloro-2-pyridinyl)oxy]acetic acid, is a systemic herbicide highly effective for the control of many woody plants and broadleaf weeds (Byrd *et al.*, 1980). Triclopyr has been widely used for several years to control brush in forestry (Bovey and Mayeux, 1980). Like its two close analogues, 2,4,5-T and picloram, triclopyr induces characteristic auxin-type responses in affected broad-leaved plants (Radosevich and Bayer, 1979).

Field and laboratory studies (Norris *et al.*, 1987; Solomon *et al.*, 1988; Stephenson *et al.*, 1990) indicate that this herbicide exhibits scarce mobility and persistency. However, little is known about its interaction with soil.

Because of the presence of carboxylic acid and basic pyridine functions, triclopyr exhibits amphoteric properties. The behavior of most ionic herbicides in soil is strongly affected by pH and the nature of the colloidal fractions of the soil. Generally, their mobility and efficacy are affected by adsorption onto soil colloids.

The adsorption of triclopyr by montmorillonite, humic acid, and seven soils of various physical and chemical properties is described in this study. The effect of the exchangeable cation and pH of the soil solution was also examined to better characterize the mechanisms involved in adsorption. Finally, to identify the soil constituents involved in triclopyr binding, triclopyr adsorption was correlated to selected soil parameters.

## MATERIALS AND METHODS

**Materials.** Triclopyr ( $C_7H_4Cl_3NO_3$ , aqueous solubility 440 mg L<sup>-1</sup>) was supplied by Dow Chemical Co. as a fluffy colorless solid (99.6% purity). Seven Italian soils, namely, a clay loam Histosol from Torba (Emilia-Romagna), a clay loam Andosol from Macomer (Sardinia), a silt clay Entisol from Carpi (Emilia-Romagna), a loamy sand Cambisol from Monte Acuto (Sardinia), a sandy loam Alfisol from Vercelli (Piemonte), a loamy sand Inceptisol from Coazze (Piemonte), and a clay loam Entisol from Cadriano (Emilia-Romagna), were examined in this study (Table 1). The samples were air-dried and sieved to <2 mm. Particle size distributions were performed by Purdue University Soil

**Table 1. Selected Physical and Chemical Properties of Soils Investigated**

property	Cadriano	Vercelli	Carpi	M. Acuto	Coazze	Macomer	Torba
OM (%)	1.43	1.45	1.70	2.40	5.51	16.00	25.60
clay (%)	23.63	10.77	46.58	9.10	2.21	14.60	30.68
sand (%)	32.56	48.56	16.44	70.10	72.60	32.90	5.09
silt (%)	38.73	36.83	29.92	16.80	25.20	26.20	24.86
water (%)	3.25	2.22	4.50	1.40	2.22	9.50	12.48
pH <sup>a</sup>	7.40	6.28	7.72	5.24	4.72	6.05	4.43
CEC <sup>b</sup>	17.92	7.68	23.73	4.03	11.63	17.62	53.32

<sup>a</sup> Soil:water = 1:1. <sup>b</sup> mequiv/100 g.

Testing Laboratory using the pipet method (Day, 1965), and organic carbon contents were determined according to the modified Walkley-Black (Jackson, 1958) method. Cation-exchange capacity (CEC) was determined according to the procedure of Hendershot and Duquette (1986). Soil pH was determined on slurries with a soil:water ratio of 1:1. Clay minerals of Carpi soil were identified by X-ray diffraction, which indicated a mixed mineralogy consisting of illite, kaolinite, and smectite. Selected physicochemical properties of the soils are given in Table 1. Montmorillonite No. 25 (bentonite) from Upton, WY, supplied by Ward's Natural Science Establishment, Rochester, NY, was used in this study. The <2- $\mu$ m fraction was obtained by sedimentation. The CEC, as determined by literature methods (Hendershot and Duquette, 1986), was 90.2 mequiv/100 g. Al<sup>3+</sup>-, Fe<sup>3+</sup>-, Cu<sup>2+</sup>-, Ca<sup>2+</sup>-, K<sup>+</sup>-, and Na<sup>+</sup>-exchanged samples were prepared by immersing the clay into 1 N solutions of the corresponding metal chlorides. The samples were centrifuged, washed repeatedly with deionized water until Cl<sup>-</sup>-free, and dried at room temperature. Humic acid was obtained from Macomer soil by the procedure of Stevenson (1982). After precipitation, it was centrifuged, redissolved and precipitated three times, dialyzed against distilled water until salt-free, and finally freeze-dried.

**Adsorption by Soil.** The adsorption of triclopyr was determined at 25  $\pm$  2 °C. In general, triplicate samples of 5 g of air-dried soil sieved through a 2-mm mesh screen were equilibrated in centrifuge tubes with 10 mL of triclopyr solution. Triclopyr concentrations before equilibration ranged from 23.28 to 581.90  $\mu$ M. 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, and the supernatant was pipetted off and analyzed immediately. The amount adsorbed by soil was calculated from the difference between the initial and final concentrations of triclopyr in solution. The effect of varying pH was examined by adding HCl or NaOH and equilibrating overnight.

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**Table 2. Triclopyr Adsorption by Mixtures of Ca-Clay and Humic Acid**

M-humic acid (mg)	Ca-clay (mg)	$K_d$	$K_{dcalc}^{a,b}$
50 <sup>c</sup>	0	38.12	
50 <sup>d</sup>	0	23.30	
0	50	0.33	0.33
15 <sup>c</sup>	35	3.80	11.67
25 <sup>c</sup>	25	10.79	19.22
35 <sup>c</sup>	15	16.68	26.78

<sup>a</sup>  $K_{dcalc} = K_d$  expected in the absence of interaction between the adsorbents.  $K_{dcalc} = 38.12$  wt fraction of humic acid + 0.33 wt fraction of Ca-clay. <sup>b</sup>  $C_0 = 90 \mu\text{M}$ . <sup>c</sup>  $M = H$ . <sup>d</sup>  $M = Ca$ .

**Adsorption by Clay.** Batch distribution isotherms on  $\text{Al}^{3+}$ ,  $\text{Fe}^{3+}$ ,  $\text{Cu}^{2+}$ ,  $\text{Ca}^{2+}$ ,  $\text{K}^+$ , and  $\text{Na}^+$ -clays were determined at  $25 \pm 2^\circ\text{C}$ . In general, duplicate samples of 50 mg of air-dried clay were equilibrated in centrifuge tubes with 5 mL of aqueous herbicide solution. Triclopyr concentrations before equilibration ranged from 23.28 to 394.14  $\mu\text{M}$ . The tubes were shaken (end-over-end) for 24 h.

Generally, 95% of the adsorption was complete within 5 h. After equilibrium was reached, the suspension was centrifuged at 30000g for 15 min. The supernatant was pipetted off and analyzed immediately by high-performance liquid chromatography (HPLC). The amount adsorbed by clays was calculated from the difference between the initial and final concentrations of triclopyr in solution.

**Adsorption by Clay and Humic Acid and Their Mixtures.** Duplicate samples of Ca-montmorillonite (50 mg) or humic acid (50 mg) were shaken for 24 h with 5 mL of triclopyr solution. After shaking, the suspension was centrifuged at 30000g for 15 min. The supernatant was removed and analyzed. Similar measurements were made using mixtures of Ca-clay and humic acid in different ratios as listed in Table 2.

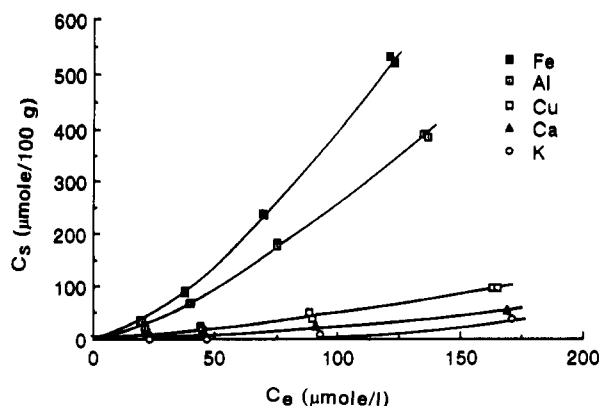
**Triclopyr Analyses.** Triclopyr concentrations were determined by HPLC analyses. A Waters 501 liquid chromatograph equipped with a  $300 \times 4$  mm i.d. Waters  $\mu\text{Bondapak C}_{18}$  (10  $\mu\text{m}$ ) analytical column, a multiwavelength Waters 490 programmable detector operating at 295 nm, and a Waters Baseline 810 chromatography workstation, was used. The mobile phase (1 mL  $\text{min}^{-1}$ ) was composed of acetonitrile + water (70 + 30 by volume, pH 3). In these conditions the retention time of triclopyr was 4.1 min.

**Potentiometric Titrations.** Dissociation constants for triclopyr acid were determined by the Gran method from titrations carried out on a series of solutions (30 mL) at  $25^\circ\text{C}$ . Measurements were made with a Metrohm 716 DMS Titrino, equipped with combined pH glass electrode. Small amounts of alkali (NaOH) were added with a micropipet under nitrogen to the end points. All solutions were of ionic strength 0.1 M ( $\text{NaClO}_4$ ) with acid concentration of  $1 \times 10^{-3}$  M.

**Data Analysis.** The data were fitted to the logarithmic transform of the Freundlich equation,  $\log c_s = \log K_f + 1/n \log c_e$ , where  $c_s$  ( $\mu\text{mol kg}^{-1}$ ) is the amount of herbicide adsorbed by soil,  $c_e$  ( $\mu\text{M}$ ) is the equilibrium concentration in solution, and  $K_f$  and  $1/n$  are empirical constants representing the intercept and the slope of the isotherm, respectively. In the case of adsorption on clay  $c_s$  is expressed as micromoles per 100 g of adsorbent. In the case of adsorption on homoionic montmorillonite and humic acid, the values of  $1/n$  deviate considerably from unity; therefore, it was preferred to use the distribution coefficient  $K_d$  to measure the adsorption extent. This coefficient is defined by the relationship  $c_s/c_e$ , where  $c_s$  ( $\mu\text{mol}/100\text{ g}$ ) is the amount of herbicide adsorbed and  $c_e$  ( $\mu\text{M}$ ) is the equilibrium concentration in solution. Fitting was performed by a least-squares regression analysis program using the Stat View 512 statistical package (Brain Power, Inc., Calabasas, CA) on a Macintosh IIsi computer (Apple Computer). This statistical package was also used to determine the correlation between adsorption and soil parameters.

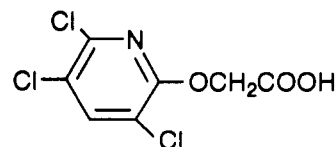
## RESULTS AND DISCUSSION

**Adsorption on Clays and Humic Acid.** Figure 1 shows the adsorption isotherms of triclopyr on  $\text{Al}^{3+}$ ,  $\text{Fe}^{3+}$ ,  $\text{Cu}^{2+}$ ,  $\text{Ca}^{2+}$ ,  $\text{K}^+$ , and  $\text{Na}^+$ -montmorillonites. All of the

**Figure 1.** Adsorption isotherms of triclopyr on homoionic clays.**Table 3. Freundlich Constants ( $K_f$  and  $1/n$ ) and Determination Coefficient ( $r^2$ ) for the Adsorption of Triclopyr on Montmorillonite**

sample	pH <sup>a</sup>	$K_f^b$	$1/n^b$	$r^2$	$K_d^c$
$\text{Fe}^{3+}$	4.2	0.397 ( $\pm 0.019$ )	1.501 ( $\pm 0.023$ )	0.999	3.77
$\text{Al}^{3+}$	4.5	0.338 ( $\pm 0.067$ )	1.441 ( $\pm 0.078$ )	0.983	2.11
$\text{Cu}^{2+}$	5.9	0.219 ( $\pm 0.116$ )	1.119 ( $\pm 0.127$ )	0.936	0.58
$\text{Ca}^{2+}$	6.2	0.172 ( $\pm 0.034$ )	1.119 ( $\pm 0.037$ )	0.994	0.33
$\text{K}^+$	7.0	0.001 ( $\pm 0.171$ )	2.056 ( $\pm 0.263$ )	0.968	0.09
$\text{Na}^+$	7.2				

<sup>a</sup> pH of aqueous suspension. <sup>b</sup> Values within parentheses are 95% confidence intervals. <sup>c</sup>  $C_0 = 90 \mu\text{M}$ .

**Figure 2.** Structure of triclopyr.

isotherms are of the S-type according to the classification of Giles *et al.* (1960). The S-shape of the curve is usually explained as due to significant competition, for substrate sites, from the molecules of the solvent. As is usual in pesticide adsorption studies, the empirical Freundlich relationship was used to evaluate the results ( $r^2 \geq 0.94$ ). The values of  $K_f$  and  $1/n$  are given in Table 3. Because the values of  $1/n$  deviated considerably from unity in several samples, it was preferred to use the distribution coefficient  $K_d$  as a measure of the adsorption capacity. Both the  $K_f$  and  $K_d$  values substantiate that the adsorption of triclopyr on clays decreases in the order  $\text{Fe}^{3+} > \text{Al}^{3+} > \text{Cu}^{2+} > \text{Ca}^{2+} > \text{K}^+ > \text{Na}^+$ , which is also the order of decreasing acidity of both interlayer and external solutions (4.2–7.2). Such a behavior suggests that the magnitude of adsorption is influenced by the nature of the saturating cation and particularly by its polarizing power on the coordinated water molecules. The acidic dissociation constant of triclopyr, as reported in the literature, is 2.68 (Worthing and Hance, 1991). Potentiometric titration showed that the herbicide behaves as a diprotic acid with the two protonation sites on the pyridine cyclic nitrogen and the carboxylate group (Figure 2). Both groups are considerably acidic with  $\text{p}K_a$  values of 2.28 and 3.35, respectively. Thus, depending on pH, triclopyr can exist in cationic, neutral, and anionic forms. Although the percentage of protonated and neutral herbicide species in solution would be insignificant in the pH range 4.2–7.2 of the clay aqueous suspension (Table 3) the effective acidity at colloid surfaces may be 2–3 pH units lower than solution pH (Mortland and Raman, 1968; Bohn *et al.*, 1985). This would account for the greater adsorption observed on  $\text{Fe}^{3+}$ - and  $\text{Al}^{3+}$ -montmorillonite samples at pH levels much

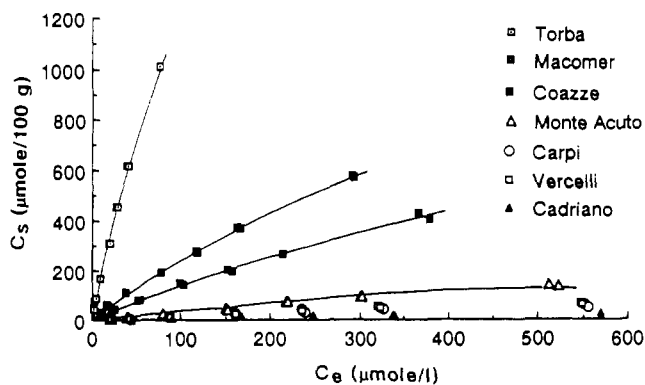


Figure 3. Adsorption isotherms of triclopyr on seven soils.

Table 4. Freundlich Constants ( $K_f$  and  $1/n$ ) and Determination Coefficient ( $r^2$ ) for the Adsorption of Triclopyr on Soil

sample	$K_f^a$	$1/n^a$	$r^2$	$K_{om}$
Torba	26.710 ( $\pm 0.051$ )	0.848 ( $\pm 0.026$ )	0.998	1.043
Macomer	5.746 ( $\pm 0.014$ )	0.813 ( $\pm 0.007$ )	0.998	0.359
Coazze	3.295 ( $\pm 0.016$ )	0.818 ( $\pm 0.009$ )	0.997	0.598
Monte Acuto	0.446 ( $\pm 0.020$ )	0.933 ( $\pm 0.080$ )	0.997	0.186
Carpi	0.224 ( $\pm 0.065$ )	0.915 ( $\pm 0.037$ )	0.980	0.133
Vercelli	0.169 ( $\pm 0.062$ )	0.979 ( $\pm 0.036$ )	0.984	0.117
Cadriano	0.064 ( $\pm 0.096$ )	0.978 ( $\pm 0.056$ )	0.962	0.045

<sup>a</sup> Values within parentheses are 95% confidence intervals.

higher than  $pK_a$  values for the herbicide. Thus, physical forces, hydrogen bonding, or cationic binding between the protonated pyridine moiety of the herbicide molecule and the clay surface could account for the greater extent of adsorption observed on these clays. Instead, as solution pH increases (e.g., with  $K^+$ - and  $Na^+$ -montmorillonite), the herbicide would exist predominantly in the anionic form, leading to a repulsion interaction with the negatively charged clay surfaces.

H-humic acid adsorbed more triclopyr than Ca-humate and much more than Ca-clay (Table 2), suggesting that once again the nature of exchange cation or the pH of the colloidal suspension (3.8 for H-humic acid and 4.9 for Ca-humate), has a great influence on the adsorption mechanism of triclopyr. The adsorption was also tested on mixtures of Ca-montmorillonite and H-humic acid. It is interesting to note that the comparison of experimental  $K_d$  values with those expected by assuming an independent adsorptive behavior for clay and humic acid indicates that the interaction between these two components reduces the surface available for herbicide adsorption. Similar observations were made for metolachlor on mixtures of clay and organic matter (Pusino *et al.*, 1992). These findings agree with soil experiments which generally show that the contribution of the clay fraction to the adsorption is much lower than studies with the pure minerals would indicate (Hance, 1969).

**Adsorption on Soil.** The adsorption isotherms were generally of the L-type (Giles *et al.*, 1960) (Figure 3), indicating a minor competition, for the adsorbing sites, between solute and solvent molecules, as compared to the herbicide-clay system. The empirical Freundlich equation ( $r^2 \geq 0.96$ ) described closely the adsorption on the soil. The constants  $K_f$  and  $1/n$  determined on our system, together with the coefficients ( $r^2$ ) for the linear fit, are given in Table 4. Inspection of the data listed in Table 4 indicates that  $K_f$  decreases in the order Torba > Macomer > Coazze > Monte Acuto > Carpi > Vercelli > Cadriano, which is also the order of decreasing amount of organic matter content in the soils. This trend is in accord with results obtained by studies on triclopyr movement

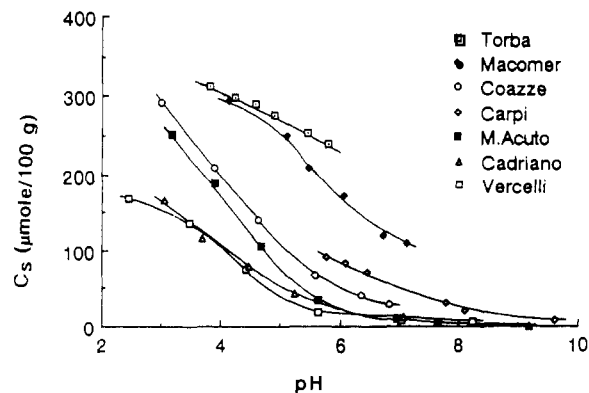


Figure 4. Effect of pH on the adsorption of triclopyr on seven soils.

Table 5. Linear Regression Analyses for Triclopyr Adsorption ( $K_f$ ) and Selected Properties of Soils ( $x$ )<sup>a</sup>

soil parameter	correl coeff	probability level	intercept	slope
$x$	$r$	$P$	$a$	$b$
OM (%)	0.930	0.002	-2.14	0.95
clay (%)	0.235	0.611	2.28	0.15
pH	0.606	0.149	32.94	-4.63
CEC	0.903	0.005	-5.16	0.54

<sup>a</sup> For the equation  $K_f = a + bx$ .

(Stephenson *et al.*, 1990) which showed that, at all sampling times, 90% or more of triclopyr was recovered in the soil organic layer.

However, the dependence of triclopyr adsorption on soil solution pH was also investigated (Figure 4). It was found that the herbicide adsorption increased as the pH of soil solution decreased, according to the general trend for the interaction of ionizable molecules with organic matter (Nicholls and Evans, 1991). To check this hypothesis, linear regression analyses between  $K_f$  and selected soil parameters were performed (Table 5). As can be seen, the correlation between  $K_f$  and OM content is highly significant. Thus, the organic matter content may explain 86% ( $r^2$ ) of the total variance shown in triclopyr adsorption. The next best correlated soil constituent was the CEC, as would be expected due to the close dependence of CEC on organic matter and clay contents of soils. Soil pH was weakly correlated to triclopyr adsorption, although pH was previously shown to affect triclopyr adsorption on selected soils where pH was adjusted. The weak correlation of herbicide adsorption by the seven soils with pH may be due to the limited pH range (4.43–7.72) characteristic of natural soils as compared to that explored with adjusted soils. Furthermore, all of the pH values were substantially greater than  $pK_a$  values of triclopyr. However, a high correlation does not give always a valid picture of the adsorption, as soil properties are frequently interrelated (Burchill *et al.*, 1981). In fact, the values of  $K_f$  for the seven soils, as normalized by referring them to 100% organic matter,  $K_{om}$ , are not very close to each other (Table 4), as would be expected if organic matter is the major soil parameter responsible for adsorption. By using a multiple regression referred to two soil parameters, CEC and soil pH, to describe triclopyr adsorption by the seven soils, a much higher correlation coefficient of 0.989, significant at the 0.0005 probability level, was obtained for the fit. Thus, the CEC and soil pH in soil may account for 98% of the total variance. The multiple-regression equation takes the form

$$k_f = 14.942 + 0.477\text{CEC} - 3.174\text{pH}$$

These results are not in contrast to previous observations

about the role of organic matter in triclopyr adsorption by soil, as the soil CEC is closely related to the amount of organic matter. Most likely, the better fit obtained by use of CEC instead of organic matter content takes into account the reduction in adsorptive capacity of organic matter due to interactions with other soil components. On the other hand, the adsorption depends also on soil pH because this is responsible for the dissociation or protonation processes of both the herbicide molecules and the adsorbent surfaces. Therefore, the combination of a high OM content and a low pH value may be responsible for the  $K_f$  value of Torba soil being significantly different from the others.

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