

Chlorsulfuron Adsorption and Degradation in Soil¹KAUSALYA THIRUNARAYANAN, ROBERT L. ZIMDAHL, and DARRYL E. SMIKA²

Abstract. Adsorption of chlorsulfuron {2-chloro-*N*-[[[4-methoxy-6-methyl-1,3,5-triazin-2-yl)amino]carbonyl]benzenesulfonamide} on four soils having different pH values (6.2 to 8.1) was low. Log/log Freundlich isotherms for all soils at 8 and 30 C were linear, the extent of adsorption being greater at 8 than at 30 C. There was an inverse relationship between pH and degradation rate with a half-life of 88.5 days at pH 6.2 and 144 days at pH 8.1 at 20 C. Rapid disappearance was noted during the first 15 days after application. With decreasing moisture and temperature, chlorsulfuron degraded more slowly. The half-life ranged from 229 days at 10 C and moisture content of 75% field capacity to 62.5 days at 40 C and 75% field capacity at pH 7.7.

Additional index words. Soil moisture, soil temperature, soil pH.

INTRODUCTION

Chlorsulfuron shows promise for broadleaf weed control in small grains (21). An important characteristic is its high herbicidal activity at application rates as low as 10 to 40 g ai/ha (19, 21). Because of its high activity in soil, its persistence has implications for following crops. It has been shown that corn (*Zea mays* L.), sugarbeets (*Beta vulgaris* L.), and sunflowers (*Helianthus annuus* L.) should not be planted the year following chlorsulfuron use at rates greater than 18 g/ha (13).

Chlorsulfuron half-life has been reported to be between 1 and 2 months in the field (21, 30) and its persistence is influenced by soil temperature (30). Han and Rapisarda³, using field, greenhouse, and laboratory conditions with soils from five different locations, found an average 2-month half-life. In a subsequent investigation, they reported a 2-week half-life in nonsterile soil. Walker and Brown (30) reported a similar half-life in a sandy loam soil.

The disappearance of any organic chemical in soil is governed by edaphic factors such as soil moisture and temperature (14), and kinetic rate laws are used to describe this phenomenon (7). The available data on chlorsulfuron and most kinetic studies do not describe the effect of pH on the rate of degradation. Soil pH can affect soil adsorption

of herbicides and activity of microorganisms, both of which may affect herbicide degradation. The influence of pH has been determined for dalapon (2,2-dichloropropionic acid) (2), four acidic herbicides (3), and thiocarbamates (20, 25). Dalapon was degraded most rapidly at pH 6.5 and the degradation rate decreased at pH 7.5 or 5.3 (2). Corbin et al. (3) reported the most rapid degradation of 2,4-D [(2,4-dichlorophenoxy)acetic acid] and dicamba (3,6-dichloro-*o*-anisic acid) at pH 5.3 and the slowest rate at pH 7.5. There was no effect of the same pH range on chloramben (3-amino-2,5-dichlorobenzoic acid) and picloram (4-amino-3,5,6-trichloropicolinic acid). Working with thiocarbamates, Smith and Fitzpatrick (25) reported that EPTC (*S*-ethyl dipropylthiocarbamate), pebulate (*S*-propyl butylethylthiocarbamate), and vernolate (*S*-propyl dipropylthiocarbamate) were not affected by treatment with 10 N sodium hydroxide solution at 95 C for 1 h, whereas diallate [*S*-(2,3-dichloroallyl)diisopropylthiocarbamate] was degraded in alkali under much milder conditions. The results of Lode and Skuterud (20), however, indicated an increased degradation of EPTC as pH increased from 5 to 7.

The objectives of these experiments were to determine: a) the influence of soil pH on chlorsulfuron adsorption, b) the effect of pH on the rate of degradation of chlorsulfuron in four soils, and c) the effect of soil moisture and temperature on degradation rate in one soil.

EXPERIMENTAL METHODS

The four soils used in the following experiments had similar texture and pH values ranging from 6.1 to 8.2. They were collected at four Colorado locations in 1982 (Table 1) and were air dried and stored dry until use.

Adsorption study. To determine equilibration time, 5 g of soil and 50 ml of a 0.05 ppmw ¹⁴C-chlorsulfuron solution were equilibrated in 150-ml erlenmeyer flasks placed in a gently shaking water bath at 30 ± 1 C. The chlorsulfuron (uniform labeled in the phenyl ring) had a chemical purity of 98%, a radiochemical purity of 99%, and a specific activity of 6.01 μCi/mg (2.15 mCi/mole). Three replicates were analyzed for each soil type. A 3-ml sample was removed from each flask at 1, 4, 8, 12, 24, 36, and 72 h and centrifuged for 30 min at 3330 × g. A 0.5-ml sample of the supernatant was placed in a 20-ml glass scintillation vial containing 15 ml of a water-accepting scintillation cocktail⁴ and 5 ml of distilled water. The sample was shaken well and analyzed in a liquid spectrometer using ¹⁴C-toluene internal quench correction standards. The decrease in volume of the ¹⁴C-chlorsulfuron/soil solution at each time interval was accounted for in the subsequent calculation.

To determine adsorption, 1 g of each soil was placed in a 25-ml glass centrifuge tube and 10 ml of the appropriate concentration of ¹⁴C-chlorsulfuron, i.e., 0.025

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³Han, Jerry C-Y and C. Rapisarda. 1982. Personal communication. E. I. DuPont de Nemours and Company, Wilmington, DE 19898.

⁴New England Nuclear, Boston, MA 02118. Trade names are used solely to provide specific information and do not constitute a guarantee or endorsement by the U.S. Dep. Agric.

Table 1. Characteristics of soils utilized in studies.

Soil	Classification	pH	Sand	Clay			Organic matter	Water added to obtain 75% field capacity	Cation exchange capacity
				(%)	(%)	(%)			
Platner loam	Aridic Paleustoll, fine, montmorillonitic, mesic	6.2	40	32	28	1.4	19.0	10	
Weld clay loam	Aridic Paleustoll, fine, montmorillonitic, mesic	7.1	30	38	32	1.3	19.6	25	
Rago silty clay loam	Pachic Argiustoll, fine, montmorillonitic, mesic	7.7	30	42	42	1.3	20.2	10	
Longmont clay	Aeric Halaquept, fine, montmorillonitic (calcareous), mesic	8.2	28	28	38	1.5	27.3	35	

ppm, 0.028 ppm, 0.033 ppm, 0.05 ppm, and 0.066 ppmw, was added. Four replicate samples of each soil were capped, placed in reciprocating shaker water baths at temperatures of 8 or 30 ± 1 C and gently shaken for 24 h. The samples were then centrifuged at 1200 × g for 30 min, and 1 ml of the supernatant was pipetted into a glass scintillation vial. The results are expressed as a distribution coefficient (K_d), which is the ratio of the amount of ¹⁴C-chlorsulfuron adsorbed to the amount in the equilibrium solution (28).

Adsorption was described by the Freundlich equation: $X = KC/C^n$ where X = amount adsorbed (μg/g soil), K, n = constants, and C = equilibrium concentration (μg/ml).

The replicated adsorption data were converted to their logarithm (base₁₀) and analyzed by linear regression (26). The slopes and intercepts of the adsorption isotherms for the different soil-temperature combinations were analyzed for variance (26).

pH study. All soils were air dried and screened through a 2-mm sieve. For each 4.5 kg of soil, 45 ml of 6.6 ppmw (w/v) ¹⁴C-chlorsulfuron in methylene chloride was pipetted onto a mound of soil to obtain a concentration of 6.4 ± 0.35 ppm. This concentration contained 3.8 × 10⁻² μCi/mg at time zero. At the conclusion of the experiment (212 days) the concentration of chlorsulfuron in the Platner soil was 0.99 μg/mg, which was equivalent to 5.9 × 10⁻³ Ci/mg of soil. Therefore, we extracted 0.321 Ci/mg of ¹⁴C-chlorsulfuron or 84.5% of the amount applied. The balance (15.5%) remained in the soil. This recovery is representative of all soil when one takes into account the lower degradation rate in the other soils. This equaled a field application rate of 142 g ai/ha which was approximately three times the labeled use rate, but this was required because of the low specific activity. After the methylene chloride had evaporated, the soil was mixed in a twin-shell blender for 1 h. Treated soil (100 g) was placed in 250-ml glass jars, moistened to 75% field capacity (FC), and stirred, after which the jars were loosely capped to allow for air exchange. The same procedure was followed to prepare three replicates of each of the four soils that were incubated at 20 C. The samples were removed at time zero, 15 days, and every 30 days thereafter for 240 days.

Temperature and moisture study. Three replicates of the Rago silty clay loam (pH 7.7) were incubated at 10, 20, 30, or 40 C and 75% FC. Chlorsulfuron was applied in the same way and at the same rate as reported for the previous study. For the moisture study, soil was treated at 142 g ai/ha as previously described; water was added to give three replicates of 25, 50, or 75% FC and these were incubated at 20 C.

Soil extraction and analysis. Soil (100 g) was weighed into an erlenmeyer flask, and 200 ml of aqueous 0.1 M Na₂CO₃ + 0.1 M NaHCO₃ (pH 10) was added. The mixture was shaken vigorously on a wrist action shaker for 1 h. After settling for 1/2 h, the supernatant was transferred to a 250-ml polypropylene centrifuge bottle and centrifuged at 3000 rpm for 20 min. The clear supernatant was transferred to a 500-ml separatory funnel. The remaining soil was re-extracted with 100 ml of extractant and centrifuged, and the supernatants were combined. The aqueous solution was shaken gently for 1 min, three times, with 75-ml portions of chloroform which were discarded. Since the pK_a of chlorsulfuron is about 3.8 (31), the compound remained in the anionic form in the aqueous solution. The aqueous solution was drained into a 400-ml beaker and the pH was adjusted to 3 to 4 by slowly adding 10% HCl. In this pH range, chlorsulfuron is nonionic and can be extracted with various organic solvents. The pH adjustment was done carefully to avoid foaming. The solution was transferred immediately to a 500-ml separatory funnel with a 5-ml distilled water rinse. It was extracted three times by shaking vigorously for 1 min with 100-ml portions of ethyl acetate. The emulsion, which formed only after the first extraction, was broken by centrifuging at 2130 × g for 30 min. The ethyl acetate containing the chlorsulfuron was separated from the aqueous phase and the three portions were combined in a 500-ml round-bottom flask. The combined extracts were passed through a pad of sodium sulfate to remove traces of water. Glacial acetic acid (1 ml) was added to the ethyl acetate extract and the solution was evaporated to dryness on a rotary evaporator using a 45-C water bath. The residue was quantitatively transferred, with ethyl acetate, to a 2-ml volumetric flask.

Table 2. Freundlich constants, determination coefficients, and Kd values for adsorption of chlorsulfuron in four soils at two temperatures.

Soil pH	30 C				8 C			
	Kd ^a	K	1/n	R ²	Kd ^a	K	1/n	R ²
6.2	0.123 a	0.24	1.20	91.0	0.140 a	0.22	1.14	96.0
7.1	0.072 b	0.18	1.27	88.1	0.114 b	0.12	1.02	88.0
7.7	0.054 c	0.11	1.21	82.2	0.086 c	0.16	1.19	94.5
8.1	0.055 c	0.08	1.15	74.8	0.104 c	0.28	1.30	95.6

^aValues in each column followed by the same letter are not significantly different at P = 0.01 according to an analysis of covariance with equilibrium concentration as the covariant.

Thin-layer chromatographs of extracts were made on 5- by 20-cm silica gel plates⁵. Each plate was divided into two lanes, and 50 μ l of extract was applied in 10- μ l portions to one lane 1.5 cm from the bottom of the plate. Chlorsulfuron was cochromatographed in the other lane. Plates were developed in acetone:toluene (1:1, v/v) for 15 min. Each lane of the chromatogram was divided into 1-cm increments, and the gel scraped from each increment was placed in separate 20-ml scintillation vials. Water-accepting scintillation cocktail⁴ (15 ml) and distilled water (5 ml) were added to each vial. The vials were tightly capped and shaken to form a gel. Radioactivity was determined with a scintillation spectrometer. The counting efficiency was 75% and the time zero recovery of chlorsulfuron was 72.4 \pm 3.7%. The 1-cm division that cochromatographed with chlorsulfuron (i.e., had the same R_f) was identified as chlorsulfuron.

The data from the pH and temperature and moisture study were analyzed using regression methods, and the slopes (rate constants) were compared for parallelism by the *t*-test (18).

RESULTS AND DISCUSSION

Adsorption study. The equilibrium time for adsorption of ¹⁴C-chlorsulfuron in soils was 24 h, with almost no additional adsorption occurring in another 48 h (data not shown), which agrees with the data of Shea (24). Adsorption was low on all four soils (data not shown). Adsorption increased with chlorsulfuron concentration, which is typical of the behavior of acidic organic molecules such as picloram (1, 5, 6, 9). Maximum adsorption of chlorsulfuron did not exceed 14% of the amount applied in any soil. While adsorption was higher at low pH, a definite relationship was not apparent (Table 2). An inverse relationship was expected because between pH 6.2 and 8.1 anionic chlorsulfuron would be the dominant species (pK_a 3.8).

Values of 1/n for chlorsulfuron were greater than unity, indicative of low adsorption (Table 2). Similar 1/n values have been reported for prometryn [2,4-bis(isopropylamino)-

6-(methylthio)-s-triazine] (12) and fenuron (1,1-dimethyl-3-phenylurea) (10), but the soils studied had high organic carbon contents (45.7 and 12%, respectively). K-values for chlorsulfuron were low and ranged from 0.08 to 0.28 (Table 2). Similar values have been reported for fenuron (10) which also exhibits a low percent adsorption. Anionic herbicides like picloram are known to preferentially adsorb on activated charcoal and anion-exchange resins but do not adsorb extensively on clay minerals and in natural soils (6); chlorsulfuron appears to behave similarly.

As expected, chlorsulfuron's K_d values were higher at 8 than at 30 C. Low K_d values and low adsorption have also been reported for picloram (6) and for chloramben (28). Shea (24) reported higher adsorption of chlorsulfuron on aluminum oxide anionotropic resins, which indicated its potential for interaction with soil aluminum or iron oxides. He also reported a negative adsorption of chlorsulfuron anions at negatively charged surfaces of montmorillonite, illite, and kaolinite.

The slopes of the linear relationship between log of amount adsorbed and log of equilibrium concentration were not different. K_d values, however, increased with a decrease in temperature, indicating that adsorption follows this trend and is most likely an exothermic process. Decreased adsorption with increasing temperature has also been observed with 1,3,5-triazines (28), alachlor [2-chloro-2',6'-diethyl-N-(methoxymethyl)acetanilide] (23), picloram (4), and several dinitroanilines (11). This phenomenon can be explained on the basis of surface-solute and water-solute interactions. With increasing temperature, the kinetic energy of the adsorbed molecules and molecules approaching the adsorbent surface increases, hence the electrostatic attraction decreases, resulting in decreased adsorption. The difference in the amount of chlorsulfuron adsorbed for all soil-temperature combinations was tested by covariance analysis with equilibrium concentration as the covariate (analysis not shown). Temperature did affect the amount adsorbed with the least effect at the lowest pH.

pH study. There is a clear inverse relationship between pH and degradation rate (Figure 1). A rapid disappearance of the chemical was noted during the first 15 days, followed by a constant and slower rate in all soils. Therefore, half-lives were calculated, including and excluding time-zero data. Hamaker and Goring's (8) two-compartment model

⁵G25UV-254 TLC plates, Brinkmann Instruments, Inc., Westbury, NY 11590.

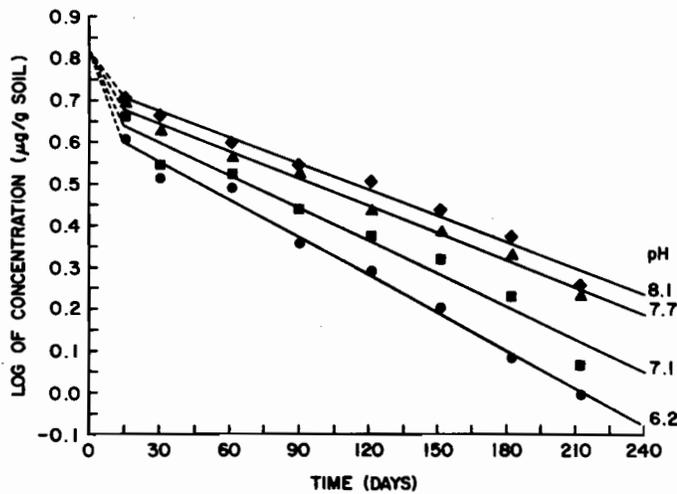


Figure 1. The effect of soil pH on chlorsulfuron degradation.

may explain this phenomenon. Turnover of pesticides is a complex process in which a pesticide and its metabolites coexist in labile and unavailable pools. Thus, when a herbicide is added to soil, a large percentage of it is available for rapid degradation, which has been shown in some cases (15, 34). With time, a greater percentage of the herbicide remaining is less available and degradation slows, eventually reaching a steady state and proceeding at a rate determined by the labile pool. Based on our results this time may be as short as 2 weeks.

In the present study, there was rapid degradation from a t_0 level of 6.48 ± 0.28 ppmw during the first 15 days, which masked the subsequent slower reaction and a simultaneous buildup of the less available pool. A slow, constant release from this pool is consistent with the degradation patterns observed after 15 days. The rapid early degradation could, of course, have been caused by our addition of chlorsulfuron to dry soil followed by wetting and the consequent flush of microbial activity. Thus, this could have been an unavoidable creation of our method. However, Jenkinson and Powlson (16) discussed the effects of mechanical disturbance and air drying on microbial respiration. They suggest that the expression of these actions may

be similar and can be stimulating or depressing. Air drying renders some nonbiomass carbon decomposable (stimulation) and kills an appreciable fraction of biomass (depressing). An increase in microbial activity was observed by Stevenson in remoistened air-dried soil (27).

The half-lives of chlorsulfuron were 38.1, 60.2, 82.0, and 99.0 days for soils at pH 6.2, 7.1, 7.7, and 8.1, respectively, when the calculation included time-zero (Table 3). Half-lives calculated from slopes of first-order plots beginning at day 15 were 88.8, 105.0, 135.8, and 143.3 days for the pH 6.2, 7.1, 7.7, and 8.1 soils, respectively.

Field studies by Zimdahl and Fithian (32) indicated that application rates as low as 18 g/ha affected the dry-weight yield of sugarbeet and corn after 25 months (approximately 14.25 months of this was a period when moisture and temperature were conducive to degradation). Using a 30-day half-life for chlorsulfuron (for a pH 7.1 soil at 20 C, 12% moisture) (30) or our 61.2 days (including time-zero, Table 3) and assuming degradation occurs over 14.25 months, we find a concentration (0.17 and 1.5 g, respectively) that should not injure sugarbeet or corn. However, assuming a $t_{1/2}$ of 104.2 days (Table 3) we find a residual concentration of greater than 4.4 g/ha that was injurious to sugarbeet and sunflower (32). This combination of field data with our laboratory data lends credence to our conclusion that chlorsulfuron has a longer half-life in soil than has been reported and to our exclusion of time-zero data in calculating the half-life.

Effect of soil moisture and temperature on degradation of chlorsulfuron. When t_0 data (mean 6.36 ± 0.35 ppmw) were excluded, half-lives varied from 231.7 days at 10 C to 63.0 days at 40 C. Degradation rate increased directly with temperature and moisture content (Table 4). As in the pH study, herbicide concentration dropped sharply after 15 days, and subsequently the rate was constant and followed the first-order kinetic model (data not shown). Hamaker and Goring's (8) two-compartment model is applicable. A major route of chlorsulfuron degradation in soil is nonenzymatic hydrolysis, so it can be assumed that increasing soil moisture content will increase degradation. Increasing moisture from 25 to 50% of field capacity increased degradation rate 46% and another 16% when moisture was further increased to 75% of field capacity. This is because chemical hydrolysis and microbial degradation were favored.

Table 3. Calculated half-lives from plots including and excluding time-zero (t_0) and determination coefficients (R^2).

Soil	pH	Graphically determined	First-order rate constant	R^2	Calculated half-life	First-order rate constant	R^2
		half-life including t_0	including t_0^a		excluding t_0	excluding t_0^a	
		(days)	(days ⁻¹)		(days)	(days ⁻¹)	
Platner loam	6.2	38.1	1.8×10^{-2} a	0.95	88.8	0.8×10^{-2} a	0.98
Weld clay loam	7.1	60.2	1.2×10^{-2} b	0.89	105.0	0.7×10^{-2} b	0.95
Rago silty clay loam	7.7	82.0	0.9×10^{-2} c	0.91	136.6	0.51×10^{-2} c	0.97
Longmont clay	8.1	99.0	0.7×10^{-2} c	0.92	143.3	0.6×10^{-2} c	0.97

^aValues in columns followed by the same letter are not significantly different at $P = 0.05$ according to a t -test for parallelism of slopes (16).

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Table 4. First-order rate constants (K), half-lives ($T_{1/2}$), Arrhenius activation energy (E_a), and determination coefficients (R^2) under different storage conditions for chlorsulfuron in Rago silty clay loam at pH 7.7 when t_0 data were excluded.

Storage conditions		First-order rate constant (K) ^a	$T_{1/2}$	E_a	R^2
Temperature	Soil moisture				
(C)	(% field capacity)	(day ⁻¹)	(days)	(kcal/mol)	
10	75	0.30 × 10 ⁻³ a	231.7	8.1	0.956
20	75	0.51 × 10 ⁻³ c	136.6	9.4	0.976
30	75	0.85 × 10 ⁻³ d	81.3	4.9	0.965
40	75	0.11 × 10 ⁻³ e	63.0	...	0.980
20	25	0.30 × 10 ⁻³ a	231.7	...	0.967
20	50	0.44 × 10 ⁻³ b	158.3	...	0.978

^aValues in columns followed by the same letter are not significantly different at $P = 0.05$ according to a t -test for parallelism of slope (16).

Shorter half-lives were observed at higher temperatures than at lower temperatures, indicating the temperature dependence of the reaction(s). Increasing temperature increased degradation rate 2 to 3 times during the first 15 days but the effect of temperature decreased with time. Variations in temperature and moisture have similar effects on pesticide degradation in soil (7, 14, 22, 29, 30, 33). Our results agree with those reported by Walker and Brown (30) on the effect of moisture and temperature on chlorsulfuron degradation in soil.

The dependence of degradation on temperature can be characterized by the Arrhenius equation:

$$\log H_1 - \log H_2 = \frac{E_a}{4.576} \left[\frac{T_2 - T_1}{T_1 \cdot T_2} \right]$$

where H_1 and H_2 are half-lives at absolute temperatures T_1 and T_2 , and E_a is the Arrhenius activation energy. The Arrhenius activation energy was 4.9 to 9.4 kcal/mol (Table 4). When E_a was calculated using half-lives that included the time-zero data, higher values were obtained which suggest the temperature dependence of degradation. However, when half-lives that excluded the time-zero data were used, E_a was lower. This may mean that in the first 15 days degradation is predominantly by chemical hydrolysis in acidic soil and that enzymatic processes assume a greater role with time, especially in alkaline soils (17). The E_a values are similar to those reported by Hyzak and Zimdahl (15) for metribuzin [4-amino-6-*tert*-butyl-3-(methylthio)-*as*-triazin-5(4*H*)-one] and two analogs.

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