

Organic Compounds in the Environment

Adsorption and Degradation of the Weak Acid Mesotrione in Soil and Environmental Fate Implications

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ABSTRACT

The ability of soils to adsorb and degrade pesticides strongly influences their environmental fate. This paper examines the adsorption and degradation of a weak acid, a new herbicide mesotrione [2-[4-(methylsulfonyl)-2-nitrobenzoyl]-1,3-cyclohexanedione], in 15 different soils from Europe and the USA. Experiments were conducted to understand the influence of soil properties, covering a wide range of soil textures, soil pH values (4.4 to 7.5), and organic carbon contents (0.6 to 3.35%). Mesotrione adsorption (K_d values ranged from 0.13 to 5.0 L/kg) was primarily related to soil pH, and to a lesser extent by percent organic carbon (%OC). As soil pH rose, mesotrione K_d values got smaller as mesotrione dissociated from the molecular to anionic form. Mesotrione degradation (half-lives ranged from 4.5 to 32 d) was also related to soil pH, getting shorter as soil pH rose. Simple regression of mesotrione adsorption against soil pH and %OC and against degradation provided a close fit to the data. The correlation between mesotrione adsorption and degradation means that K_d and half-life values are only relevant for use in environmental fate assessment if these values are "paired" for the same soil pH and %OC. The implications were as illustrated for leaching, raising important issues about combining pesticide adsorption and degradation behavior in environmental fate assessments.

PESTICIDE ADSORPTION and degradation in soils are key processes determining whether pesticide use will have any effect on environmental quality. For example, simple models and indices of leaching rely heavily on *combining* adsorption and degradation behavior, to assess the likelihood of pesticide leaching to ground water (Rao et al., 1985; Jury et al., 1987; Gustafson, 1989). However, the use of these and other models in assessing environmental behavior is complicated because adsorption and degradation are not fixed properties of pesticides. They are dynamic properties, long-recognized to be influenced by factors such as soil type, use patterns, and weather conditions (Hurle and Walker, 1980). Thus, it is important to characterize adsorption and degradation fully, to increase the precision with which safer pesticide uses and potential issues of concern can be identified.

Nicholls and Evans (1991) outlined why weak acids and other ionizable compounds were affected by soil properties such as soil pH and organic carbon content.

Their dissociation to the anionic form lowers adsorption as soil pH increases. For example, 2,4-D [(2,4-dichlorophenoxy)acetic acid], imazaquin [2-(4-isopropyl-4-methyl-5-oxo-4,5-dihydroimidazol-1*H*-2-yl)-quinoline-3-carboxylic acid], and imazethapyr [5-ethyl-2-(4-isopropyl-4-methyl-5-oxo-4,5-dihydroimidazol-1*H*-2-yl)-nicotinic acid] adsorption decreased as soil pH rose (Wehtje et al., 1987; Renner et al., 1988; Stougaard et al., 1990; Loux and Reese, 1992; Johnson et al., 1995). With sulfonyl ureas, greater adsorption at lower pH has mainly been attributed to adsorption of the molecular forms (Mersie and Foy, 1985; Shea, 1986; Wehtje et al., 1987). Furthermore, the more strongly adsorbed undissociated form adsorbs mainly to organic carbon. Hence, Walker et al. (1989) showed chlorsulfuron [1-(2-chlorophenylsulfonyl)-3-(4-methoxy-6-methyl-1,3,5-triazin-2-yl)urea] and metsulfuron-methyl [methyl 2-[(4-methoxy-6-methyl-1,3,5-triazin-2-yl)carbamoylsulfamoyl]-benzoate] adsorption to be correlated negatively with soil pH and positively with organic carbon content. With flumetsulam [2',6'-difluoro-5-methyl[1,2,4]triazolo-[1,5-*a*]pyrimidine-2-sulfonamide], the influence of soil pH only became clear when adsorption was adjusted for organic carbon (Fontaine et al., 1991).

Similar relationships with soil properties have been found in degradation studies. For example, flumetsulam and linuron [3-(3,4-dichlorophenyl)-1-methoxy-1-methylurea] degradation appeared to be influenced by soil pH and organic carbon (Walker and Thompson, 1977; Walker and Allen, 1984; Lehmann et al., 1992). Walker et al. (1985) also found that napropamide [(*RS*)-*N,N*-diethyl-2-(1-naphthoxy)propanamide] degradation was negatively correlated with the clay content of nine soils, and the correlation was improved by including soil pH as a factor. In field studies on soils adjusted to three soil pH levels for at least 10 years, imazaquin dissipated more rapidly as soil pH rose (Loux and Reese, 1992), as did imazethapyr in one soil (Loux and Reese, 1993).

Furthermore, pesticide adsorption and degradation may be linked. Correlations have been found between adsorption and degradation with some of the above pesticides, including flumetsulam, napropamide, imazaquin, and imazethapyr (Walker et al., 1985; Lehmann et al., 1992; Loux and Reese, 1992). Even though many factors influence degradation in soil, adsorption can have a key effect on the rate of degradation in soil (Scow, 1993), particularly since there is little evidence

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Abbreviations: %OC, percent organic carbon.

to suggest that adsorbed pesticides are directly available to microbes (Guerin and Boyd, 1992).

In the study reported here, the adsorption and degradation of a new triketone herbicide, mesotrione [2-[4-(methylsulfonyl)-2-nitrobenzoyl]-1,3-cyclohexanedione], a weak acid, was examined in 15 soils from Europe and the USA, covering a wide range of soil properties. The overall aim of the study was to understand better the influence of soil properties on the environmental fate of mesotrione, which is primarily for use in controlling broad-leaved weeds in maize. The specific objectives of the study were to (i) investigate and quantify the relationships of mesotrione adsorption to soil pH and organic carbon content, and to degradation; and (ii) evaluate the environmental fate implications of these relationships.

MATERIALS AND METHODS

Physico-Chemical Properties of Mesotrione

Mesotrione is a weak acid with a pK_a value of 3.1 (M. Goodman, personal communication, 1996), which dissociates from the molecular to anionic form as pH rises (see Fig. 1). The dissociation of mesotrione, as with all weak acids, cannot be predicted exactly from measured soil pH, due to pH differences between the soil surface and outer bulk soil solution. Consequently, Nicholls and Evans (1991) noted that a significant fraction of the molecular form of weak acids is likely to be present in soil when the measured soil pH is several pH units above the pK_a . Mesotrione has been shown to be stable to hydrolysis between pH 4 and 9 (P. Miles and S. Powell, personal communication, 1995).

Soil Sampling and Soil Characterization

Soil for the mesotrione adsorption and degradation experiments was sampled from 15 field sites representative of potential mesotrione use areas in Europe and the USA. The samples were taken from topsoil (0–15 cm). Soil pH was measured by electrode in the supernatant of a 1:2 soil to solution ratio (McLean, 1982), with both distilled water and 0.01 M CaCl₂ used as the solution. Organic carbon content was measured using the Walkley–Black procedure (Nelson and Sommers, 1982). Particle size analysis was by wet sieving for sand and by sequential sedimentation and analysis of supernatant for silt and clay, following removal of organic matter with hydrogen peroxide and dispersal of soil particles with sodium hexametaphosphate (Gee and Bauder, 1986). CEC was determined by sodium saturation at pH 7 and flame photometry (Rhoades, 1982). Moisture holding relations at –30 and –1500 kPa were

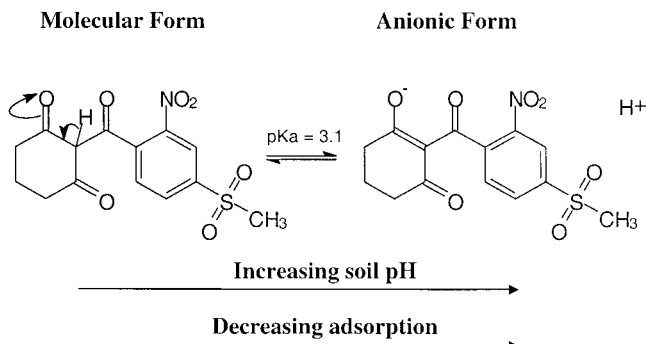


Fig. 1. The dissociation of mesotrione with rising pH.

determined on disturbed samples at field bulk density using the ceramic plate method (Klute, 1986). As shown in Table 1, the soils covered a range of soil textures, soil pH values (4.4 to 7.5 with 0.01 M CaCl₂, and 4.6 to 7.7 with distilled water), and organic carbon contents (0.6 to 3.3%).

Adsorption Experiments

Adsorption coefficients, K_d (L/kg), were determined in duplicate for mesotrione for each soil by applying the equivalent of 0.4 mg (*phenyl-U-¹⁴C*)mesotrione per kg dry soil to soil slurries. The application rate was chosen because it represents a typical concentration in soil following application at the maximum field rate, and because the adsorption isotherm for mesotrione is very close to linear up to this rate. Representative Freundlich $1/n$ values on some of these and other soils were generally around 0.95 (M.C.G. Lane, personal communication, 1998). The soil slurry had a dry soil to liquid ratio of 10 g to 20 mL, comprising air-dried, sieved (2-mm mesh) soil, sterilized by gamma irradiation, and 0.01 M CaCl₂ solution. The use of 0.01 M CaCl₂ solution was chosen to prevent dispersion of the clay colloids and to simulate a typical electrolyte concentration for soil solution (Barber, 1980). The soil slurries were shaken for 24 h at 20°C on an end-over-end shaker before applying mesotrione. The soil slurries were then shaken for a further 24 h to allow adsorption to reach equilibrium. Preliminary experiments showed equilibrium was achieved within 16 h. The soil slurries were then centrifuged (1600 × *g* for 15 min) and the supernatant removed for subsequent analysis (liquid scintillation counting for radiochemical content and thin layer chromatography to determine whether there had been any degradation of mesotrione). The remainder of the soil slurry was then analyzed by soil combustion (for radiochemical content) and by extraction in methanol and 0.5% concentrated HCl 9:1 (v/v) and TLC to determine whether there had been any degradation of mesotrione. Mesotrione was found not to degrade in this experimental system. Hence, the amount of ¹⁴C-mesotrione in the supernatant and remainder of the soil slurry were used to calculate the concentration of mesotrione adsorbed to soil and in soil solution, and hence the K_d values.

Degradation Experiments

The rate of mesotrione degradation, half-life (days), was determined in each soil by applying the equivalent of 0.6 mg (*phenyl-U-¹⁴C*)mesotrione per kg dry soil to freshly sampled field-moist soil. Before application, all soils were moistened to –30 kPa by adding distilled water and pre-incubated at 25°C for approximately 3 d. Application of mesotrione was in 1 mL of solution, comprising 0.05 M potassium phosphate. Six samples of each soil (59 g of moist soil) were dosed with mesotrione. One sample of each soil was extracted immediately. The remaining five samples were incubated at 25°C in separate chambers, purged with humidified air during incubation, and monitored for ¹⁴CO₂ in the efflux to check radiochemical balance. The remaining levels of mesotrione in soil were determined by extracting samples at 0, 7, 14, 21, and 28 d after application. At each sampling time, soil moisture contents were measured and made back up to –30 kPa by adding distilled water, which kept soil moisture contents between 91 to 110% of –30 kPa. Extraction of mesotrione was achieved by shaking the soil for 30 min with 0.05 M NH₄OH and acetone followed by centrifugation. The TLC analysis was then used to determine what fraction was mesotrione. This extraction technique is more complex than that in the adsorption experiments, because it also enabled metabolites to be extracted (not

Table 1. Characterization of the 15 soils studied, plus adsorption coefficients and half-lives.

Location of soil	Soil texture [†]	pH (CaCl ₂)	pH (H ₂ O)	Organic carbon	Sand			CEC [‡]	<i>K_d</i>	<i>K_{oc}</i>	Half-life
					%						
							cmol/kg	L/kg		d	
Osceola, NE	loamy sand	4.4	4.6	0.6	85	9	6	3.9	2.3	390	26
Noblesville, IN	loam	4.7	5.0	1.3	30	46	24	13.2	3.4	250	24
Champaign, IL	silt loam	4.6	5.0	2.0	13	61	26	26.9	5.0	240	32
Valley Springs, SD	silty clay loam	4.8	5.1	2.1	18	51	31	21.1	4.4	210	16
Elk City, KS	loam	5.0	5.3	1.0	41	37	22	10.5	1.1	110	8.0
New Holland, OH	silty clay loam	5.1	5.3	1.8	15	46	39	21.7	1.3	73	19
Breese, IL	silt loam	5.2	5.5	0.9	5	77	18	8.0	1.0	120	17
Danville, IA	silt loam	5.2	5.6	1.6	4	70	26	17.3	1.6	98	11
Martinsville, IN	silt loam	5.6	6.0	0.8	27	59	14	6.7	0.68	90	8.5
Clarence, MO	silty clay loam	6.2	6.4	1.0	8	59	33	17.4	0.63	60	14
Champaign, IL	silty clay loam	7.3	7.5	2.1	10	53	37	29.7	0.61	29	8.2
Richmond, WI	silt loam	5.9	6.1	1.3	16	59	25	10.5	0.67	52	12
Whitakers, NC	sandy loam	6.1	6.4	0.6	73	19	8	2.4	0.34	60	12
Thame, England	clay loam	6.8	7.1	3.3	41	26	33	22.9	1.1	33	4.5
Toulouse, France	loam	7.5	7.7	0.9	44	35	22	8.6	0.13	15	5.9

[†] Classified using the New Jersey system.

[‡] Cation exchange capacity.

part of the reported findings here). Radiochemical balance was between 98.2 to 101.4% throughout the degradation experiments.

RESULTS AND DISCUSSION

Adsorption and Soil Properties

The single-point adsorption experiments resulted in large differences in adsorption between soils (see Table 1). The adsorption coefficient, *K_d*, varied 38-fold; the adsorption coefficient, adjusted for the amount of organic carbon as *K_{oc}*, varied 26-fold. A preliminary analysis showed that mesotrione adsorption was correlated with soil pH and organic carbon. Correlation with soil pH using 0.01 M CaCl₂ was examined since this was used in the adsorption experiment.

The effect of soil pH in all 15 soils was examined initially, to determine how much it influenced mesotrione adsorption, since its dissociation was expected to vary several-fold over the range of soil pH. In common with other acidic pesticides, the decrease in mesotrione adsorption as soil pH increased is due to the lower adsorption potential of the dissociated anionic form compared with the undissociated molecular form (Nicholls and Evans, 1991). Thus, mesotrione adsorption coefficients were log-transformed to evaluate whether they formed a linear pattern against soil pH, to be expected for acid dissociation at pH values around the p*K_a* (Nicholls and Evans, 1991). Figure 2 shows that log₁₀(*K_d*) decreased as soil pH increased, having a regression line of:

$$\log_{10}(K_d) = -0.350\text{pH} + 2.01 \quad [1]$$

with an *r*² value of 0.66, consistent with soil pH having a strong influence on mesotrione adsorption in soil. Other pesticides show similar effects with soil pH. For example, Johnson et al. (1995) showed that adsorption of the phenoxy acid 2,4-D was lower at pH 7 than at pH 5. With the amphoteric compounds, imazaquin and imazethapyr, containing acid and basic functional groups, adsorption decreased as soil pH rose from 3 to 8 (Wehtje et al., 1987; Renner et al., 1988; Stougaard et al., 1990; Loux and Reese, 1992).

Figure 2 also shows that mesotrione adsorption was relatively strong at several soil pH units above its p*K_a* value of 3.1. In the absence of soil, the anionic form of mesotrione would theoretically predominate, since the soil pH values are all above the p*K_a*. It would therefore not be expected to adsorb strongly to soil surfaces, mainly covered by negative charged or neutral sites. However, as noted by Nicholls and Evans (1991), the p*K_a* may appear to be up to approximately 2 pH units higher in soil, because the pH close to soil surfaces is lower than the pH measured in bulk solution. Overall, the results are consistent with soil pH having a major influence over the amount of mesotrione adsorbed, accounting for more than half the variations in mesotrione adsorption to soil, by altering the proportions of molecular and anionic forms of mesotrione in soil. Similarly, Mersie and Foy (1985), Shea (1986), and Wehtje et al. (1987) attributed the higher adsorption of the sulfonyl urea herbicides at lower pH values to adsorption of the molecular forms.

In addition, organic matter can also account for some of the variance in adsorption not accounted for by soil pH, particularly since the molecular form is likely to adsorb to organic matter. Indeed, in 21 soils with a pH ranging from 6 to 8, Fontaine et al. (1991) demonstrated that the relationship between the adsorption of the weak acid flumetsulam and soil pH only became clear when plotted against the adsorption coefficient,

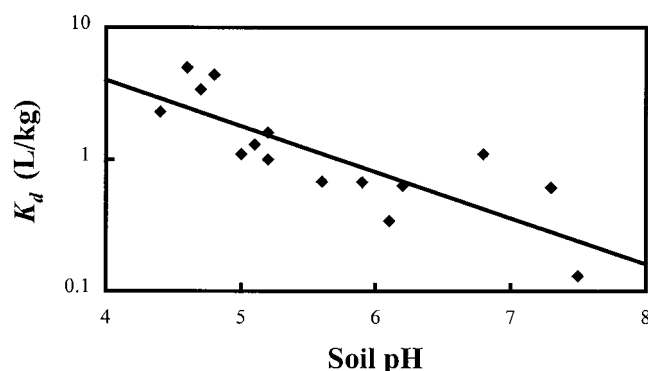


Fig. 2. Plot of mesotrione adsorption (*K_d*) versus soil pH.

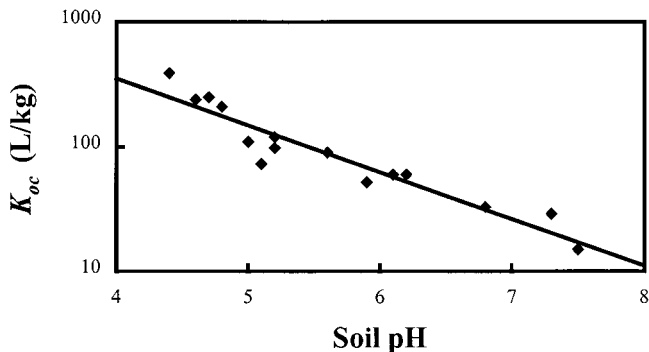


Fig. 3. Plot of mesotrione adsorption (K_{oc}) versus soil pH.

K_{oc} , adjusted for organic carbon content. Thus, mesotrione adsorption was also plotted against soil pH as the log-transformed organic carbon adsorption coefficient, $\log_{10}(K_{oc})$, with K_{oc} calculated as:

$$K_{oc} = (100 \times K_d) / \%OC \quad [2]$$

where %OC is the percent organic carbon in the soil. It is not strictly correct to determine a K_{oc} for mesotrione, because the anionic form is unlikely to adsorb primarily to organic carbon. However, an apparent K_{oc} may characterize the overall adsorption of both molecular and anionic forms of mesotrione with only a few parameters. This contrasts the mechanistic approach of Fontaine et al. (1991), which involves many parameters, including estimating a pK_a to account for the effect of soil surface acidity. Figure 3 shows that $\log_{10}(K_{oc})$ decreased as soil pH increased, with a regression line of:

$$\log_{10}(K_{oc}) = -0.376pH + 4.05 \quad [3]$$

which accounted a further 24% of the variance in mesotrione adsorption than soil pH alone ($r^2 = 0.90$), and gives a close fit to the data with only a couple of parameters. These results confirm that soil pH and organic carbon are the predominant soil properties explaining most of the adsorption behavior of mesotrione in soils. Walker et al. (1989) found similar results in a study on chlorsulfuron and metsulfuron-methyl adsorption in 23 soil samples, in which adsorption was correlated negatively with soil pH and positively with organic carbon.

We conclude that Eq. [2] and [3] provide a sound empirical basis for relating mesotrione adsorption within the measured ranges of soil pH and %OC. This type of relationship is commonly used to relate other types of behavior to soil properties (Webster and Oliver, 1990). In order to test how well this relationship fitted the measured K_d values for mesotrione, fitted K_d values were calculated for the 15 soils by substituting values for soil pH and %OC into Eq. [2] and [3]. The fitted K_d values were all within a factor of two of the measured K_d values, even though the measured K_d values varied 38-fold from 0.13 to 5.0 L/kg. In addition, an F test, described by Dent and Blackie (1979), showed that the fitted K_d values did not differ significantly from a 1:1 correspondence with the measured K_d values at the 1% level of confidence.

Degradation and Soil Properties

The degradation of mesotrione was characterized by fitting the log version of simple first-order kinetics to the decline of mesotrione residue levels during the degradation experiments. Residue declines in all 15 soils were represented reasonably accurately by first-order kinetics (r^2 values were between 0.91 and 0.99, average 0.97), from which half-life values were calculated for mesotrione (see Table 1).

Half-lives for the 15 soils varied sevenfold from 4.5 to 32 d. A preliminary analysis showed that mesotrione degradation was only correlated strongly with soil pH. Other studies have found reasonable correlations with both soil pH and organic carbon. For example, Lehmann et al. (1992) showed that flumetsulam degraded most rapidly in soils at $pH > 7$ and at lower organic carbon contents. Regression analysis demonstrated that linuron degradation was positively correlated with organic carbon contents, with a stronger relationship when soil pH was included (Walker and Thompson, 1977; Walker and Allen, 1984).

Figure 4 shows that mesotrione degradation was primarily correlated with soil pH (that measured using 0.01 M $CaCl_2$), with a regression line of:

$$\log_{10}(\text{half-life}) = -0.192pH + 2.18 \quad [4]$$

giving an r^2 value of 0.63. Regression was with soil pH measured using 0.01 M $CaCl_2$ because only small amounts of distilled water were added in the degradation experiments, so this would not have significantly lowered the concentration of electrolytes in soil solution (on average pH was only 0.3 pH units lower in 0.01 M $CaCl_2$ than in distilled water). Overall, soil pH accounted for more than half the variation in half-life values that got shorter as soil pH increased. The correlation with soil pH is also similar to that for mesotrione adsorption against pH (cf. Fig. 2). As with adsorption, theoretical grounds for explaining how soil pH per se influences degradation need to be examined.

Correlating Degradation and Adsorption

The similar pattern of mesotrione degradation and adsorption with soil pH suggests prima facie that they might be linked. Many factors influence the rate of pesti-

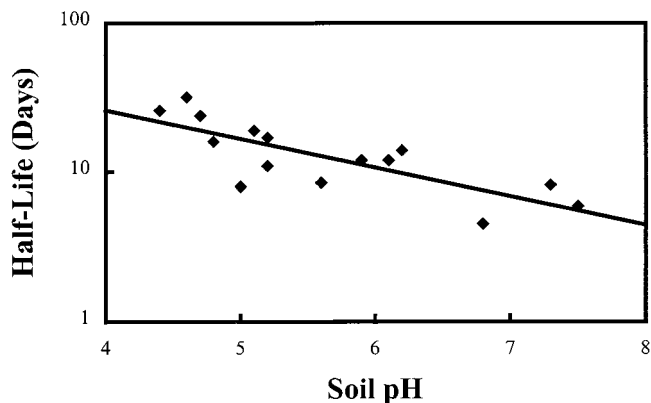


Fig. 4. Plot of half-life versus soil pH.

cide degradation in addition to adsorption, from soil conditions limiting the metabolism of pesticides by microbes, to the rate of pesticide transfer to the degrading microbes by desorption and diffusion processes (Rijnaarts et al., 1990; Mihelcic and Luthy, 1991). Scow (1993), however, acknowledges that adsorption can be a predominant factor influencing the rate at which pesticides are degraded in soil, since it governs the availability of pesticides in soil solution.

In a detailed study, Ogram et al. (1985) demonstrated that 2,4-D was only degraded in soil solution by microbes present both in soil solution and on soil surfaces. And in an experiment to minimize the effects of other factors influencing degradation, Anderson (1981) varied adsorption using charcoal to demonstrate that adsorption directly controlled the degradation rate of diallate and triallate. In a comparable experiment, Cantwell et al. (1989) obtained similar results for imazethapyr. In other studies, correlations have been found between adsorption and degradation for flumetsulam (Lehmann et al., 1992) and napropamide (Walker et al., 1985). For imazaquin and imazethapyr, there is evidence to support the correlations between adsorption and degradation (Basham et al., 1987; Loux et al., 1989a,b; Loux and Reese, 1992).

It is, therefore, not surprising that the degradation of mesotrione was also correlated with its adsorption to soil, as shown in Fig. 5, which had a regression line of:

$$\log_{10}(\text{half-life}) = 0.383 \times \log_{10}(K_d) + 1.09 \quad [5]$$

with an r^2 value of 0.45. This regression line had a lower r^2 value than Eq. [4], for half-life vs. soil pH, probably due to the influence of the lowest data point. Fitted values for half-life were obtained by substituting values for soil pH and %OC into Eq. [2] to [4] for the 15 soils, to test the significance of this regression. These fitted half-lives were almost all within a factor of two of the measured half-lives, even though the measured ones varied sevenfold from 4.5 to 32 d, and an F test, de-

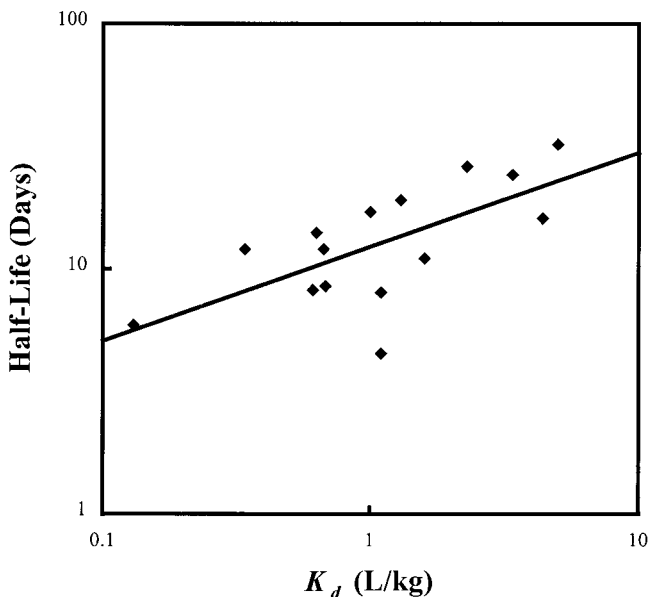


Fig. 5. Plot of half-life versus K_d .

scribed by Dent and Blackie (1979), showed that measured and fitted half-lives did not differ significantly from a 1:1 correspondence at the 1% level of confidence, confirming that degradation was significantly correlated with adsorption.

Environmental Fate Implications

The main implication of these results for assessing the environmental fate of mesotrione is that adsorption and degradation must be regarded as correlated. It is, however, common to ignore this correlation for many kinds of environmental fate assessment. In leaching assessments, for example, the smallest K_d is combined with the longest half-life as a worst-case scenario, forming part of a first-order uncertainty analysis (Loague et al., 1989). For mesotrione, this does not accurately represent the measured data, since this combination refers to high and low pH simultaneously. Where this simple practice results in parameter combinations that have not occurred in the measured data, it will distort assessments. Hence, the use of correlation between degradation and adsorption, where it has been measured, represents a significant advance over this simple practice of random or unrealistic worst-case combinations. Diaz-Diaz et al. (1998), for example, demonstrated how leaching assessment for carbofuran (2,3-dihydro-2,2-dimethylbenzofuran-7-ylmethylcarbamate) and ethoprophos (*O*-ethyl *S,S*-dipropyl phosphorodithioate) changed by accounting for the correlation between degradation and soil pH.

To illustrate environmental fate implications of the correlation between mesotrione adsorption and degradation, values of the Groundwater Ubiquity Score (GUS) index (a simple index of potential leaching) were calculated using measured "paired values" of K_{oc} and half-life from all 15 soils (Gustafson, 1989). The GUS values that were based on these "paired values" show no trend with soil pH in the potential for leaching (Fig. 6). They were all similar despite the large changes in mesotrione adsorption and degradation with soil pH. The GUS values averaged 2.20 and ranged 1.6-fold from 1.64 to 2.57. This characterizes mesotrione as ranging

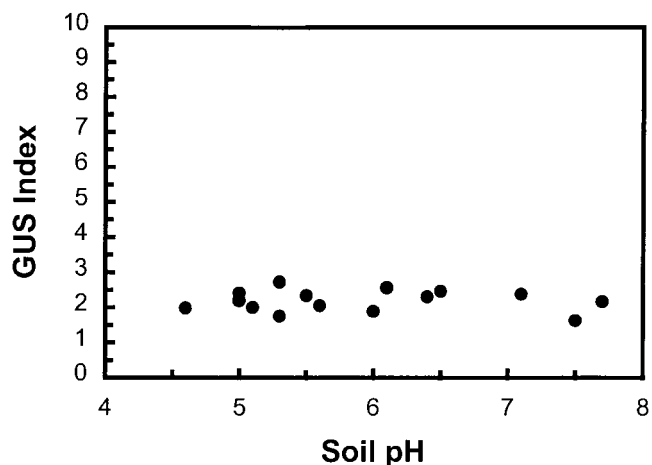


Fig. 6. Spread of measured Groundwater Ubiquity Score (GUS) leaching index values versus soil pH.

from the edge of the improbable leacher region to just inside the transition region between improbable and probable leacher. However, a different view of leaching potential is obtained by ignoring the correlation between adsorption and degradation. This resulted in GUS values ranging from 0.92 to 4.25 with a 4.6-fold variation in mesotrione leaching potential, characterizing it as both clearly in the improbable leacher category and in the probable leacher category.

CONCLUSIONS

The adsorption and degradation of mesotrione was examined to understand the influence of soil properties on its behavior and fate. The adsorption of mesotrione was affected by soil pH and organic carbon. The influence of the variations in mesotrione adsorption accounted for much of the variation in the degradation of mesotrione. The adsorption and degradation of mesotrione were correlated and could also be correlated to soil pH and %OC values. This means that only "paired" K_d and half-lives are relevant for assessing the environmental fate of mesotrione, as illustrated for leaching.

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