

## Influence of Organic Matter and pH on Bentazone Sorption in Soils

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Bentazone (3-isopropyl-1*H*-2,1,3-benzonhiadiazain-(4)3*H*-one 2,2-dioxide) is a postemergence herbicide which is used extensively worldwide, especially in China. The sorption of bentazone in various types of soils and extracted humic acids was investigated using a batch equilibration technique. Significant linearity was observed in sorption isotherms in five different types of soil, with distribution coefficients ( $K_d$ ) that varied between 0.140 and 0.321 mL g<sup>-1</sup>. The distribution coefficient was determined to be a function of organic matter and pH in the soil. A model based on distribution coefficients was developed to predict bentazone sorption in soils. The organic matter-normalized partition coefficients for the neutral and anionic forms of bentazone were 370.3 and 2.40 mL g<sup>-1</sup>, respectively. Hence, more attention should be given to the potential leaching problem when bentazone is applied in soils containing low organic matter and high pH.

**KEYWORDS:** Sorption; bentazone; soil; humic acids; partition coefficients

### INTRODUCTION

Bentazone is a widely used selective herbicide for controlling weeds in corn fields, paddy fields, and orchards. Although bentazone is mainly applied postemergence, much of it eventually contaminates the soil and water systems (1). The investigation of sorption–desorption phenomena of herbicides in soil is of great interest in both environmental and ecological research. Sorption of organic contaminants in soil is an important process affecting environmental fate (transport, degradation, volatilization, and bioaccumulation) (2).

Most of the available reports state that bentazone exhibits little sorption in soil and has relatively high mobility. Abernathy and Wax found that bentazone was weakly adsorbed in 12 Illinois soils (3), as evidenced by its movement with a solvent in thin-layer soil plates and soil column studies. Bergstrom and Jarvis reported a distribution coefficient  $K_d = 2.9$  mL g<sup>-1</sup> for bentazone sorption in a Mellby sand (5.9% organic matter) (4). Donigian and Carsel estimated a  $K_d$  ranging from 0.26 to 6.6 mL g<sup>-1</sup> (5). Gaston et al. showed that bentazone sorption in six Dundee slit loam soils could be described with  $K_d = 0.032 \pm 0.002$  mL g<sup>-1</sup> ( $r^2 = 0.94$ ) (6). It was also reported that the sorption of bentazone in Sharkey clay obeyed the Freundlich isotherm, and the approximate linear distribution coefficient,

$K_{oc}$ , was  $6.7 \pm 0.2$  mL g<sub>oc</sub><sup>-1</sup> (7). Nevertheless, several field lysimeter studies on bentazone mobility have reported minor leaching (4). Abernathy and Wax attributed their results to repulsion between ionized bentazone and soil colloids (3). Gaston et al. found greater sorption of bentazone in the Sharkey soil due to finer texture and higher organic carbon (6, 7). However, little systematic investigation has been attempted to find how dominant factors affecting bentazone sorption in soil are quantitatively related to sorption.

Soil properties, such as organic matter content, clay and oxide content, dissolved organic matter, pH, particle size, and cation-exchange capacity, have been found to be important factors affecting herbicide sorption (8–10). However, most studies on bentazone were conducted without extensive investigation of soil properties or the effects of sorbents/sorbates and physical/chemical properties on sorption (10–12). The objective of this study is to develop a model explaining the sorption of bentazone in six soils with different physicochemical properties.

### MATERIALS AND METHODS

**Soils.** Five types of soil were utilized for the studies described herein. Surface soils (0–15 cm) were collected from Yuhang, Linan, Anji, and Deqing, in the north of Zhejiang Province, China. Large lumps of the soil were manually broken apart and air-dried for 48 h. The soil was then homogenized by passing through a sieve with 2-mm openings, put into plastic bags, and stored at 4 °C until use.

Physical and chemical soil characteristics are given in Table 1. Soil pH was measured in a mixture of soil + water (1:1 by weight) with a glass pH electrode. Particle size distributions were evaluated using the

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**Table 1.** Physical and Chemical Properties of Soils Collected from 0 to 15 cm Depths in Zhejiang Province, China

no.	site of soil	type of soil	OM <sup>a</sup> (g kg <sup>-1</sup> )	pH (soil/H <sub>2</sub> O, 1:1)	clay (g kg <sup>-1</sup> )	sand (g kg <sup>-1</sup> )	silt (g kg <sup>-1</sup> )	CEC <sup>b</sup> (cmol/kg)
1	Yuhang	loam, sand	40	7.67	378	61	482	30.0
2	Linan	clay, loam	6.4	4.14	493	156	310	15.7
3	Anji	sand, loam	43	7.36	151	344	442	17.8
4	Anji	loam, sand	28	4.79	254	293	408	6.4
5	Deqing	loam, sand	40	4.9	289	113	535	12.4

<sup>a</sup> Soil organic matter content. <sup>b</sup> Cation exchange capacity.

pipet method (13), and the organic matter content (OM) was measured by a colorimetric method using chromic acid (14). The cation exchange capacity (CEC) was determined by following the procedure reported by Hendershot and Duquette (15).

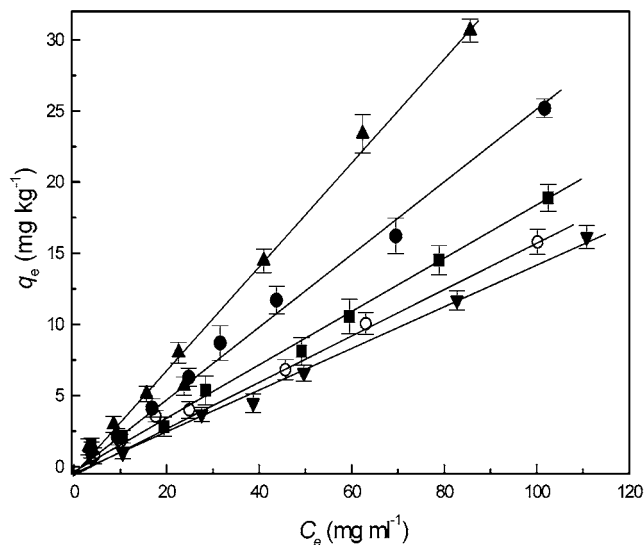
**Humic Substances.** Humic substances were extracted by the procedure outlined by Stevenson (16) and Piccolo (17). First, a 50-g soil sample was washed with 0.1 M HCl to decompose free carbonates and remove alkali earth metals; it was then washed again with distilled water until free of Cl<sup>-</sup> ions. The acid-washed samples were placed in centrifuge bottles with 0.5 M NaOH (1:5 soil/extractant) under a N<sub>2</sub> atmosphere and shaken for 12 h. Suspensions were then centrifuged, and the supernatants were filtered through acid-washed glass wool. Residues were further extracted with the same extracting solution until colorless extracts were obtained. Once filtered, each extract was immediately brought to pH = 1 by adjusting with concentrated HCl. The fractions were combined, and the humic acids were allowed to settle for 24 h at ambient temperature (20 ± 1 °C). Suspensions were centrifuged at 3600 rpm. The precipitated humic acids were washed three times with 0.1 M HCl, purified by shaking in a polyethylene bottle under N<sub>2</sub> for 36 h with 0.5% HCl–HF solution, washed again to remove residual Cl<sup>-</sup>, and then dried in a vacuum desiccator at ambient temperature.

**Chemicals.** Bentazone (≥97%) was obtained from BASF (Ludwigshafen, Germany). The water solubility was 500 mg L<sup>-1</sup>, and pK<sub>a</sub> = 3.2.

**Sorption in Soils and Humic Acids (HA).** A batch equilibration technique was used. A series of 10.00- and 7.00-mL 0.01 M calcium chloride solutions containing initial concentration of bentazone ranging from 3.0 to 111.0 mg L<sup>-1</sup> were added to 10.0-g soil samples (dry weight basis) in 25-mL screw-cap glass centrifuge tubes with Teflon-lined septa. Mercury chloride (200 mg L<sup>-1</sup>) was added as a biocide. The samples were shaken at 120 strokes per minute on a reciprocating shaker for 24 h in darkness at 25 ± 2 °C. The obtained suspended fraction was then centrifuged at 4800 rpm (3600g) for 30 min. Three milliliters of supernatant was removed and filtered through a 0.25-μm PTFE membrane (syringe filter). The first 2.5 mL of filtrate was transferred and used to measure pH. The last 0.5 mL was collected and stored in a vial for HPLC analysis. Experiments were carried out in triplicate with a blank as a control.

The experimental procedure for the determination of the sorption of bentazone on HA was followed as described above, except using 50.0 mg of HA with 8.00 mL of solution, and a 10-mL capacity centrifuge tube instead. The pH of the solution was adjusted to 2.5 with 1.0 M HCl before sorption experiments were initiated.

**Simulation of the Effect of pH on the Octanol–Water Partition Coefficient.** Reversed-phase HPLC was used to mimic the partitioning of bentazone between organic matter and water under different pH values. A YGW C<sub>18</sub> column (Dalian Yilite, China), 250 mm × 2.5 mm i.d. and a methanol–water mobile phase were used. As reported by Braumann et al. (18), the retention time obtained using only this mobile-phase system is strongly related to log *P*, the logarithm value of the partition coefficient of a compound between *n*-octanol and water. Phosphoric acid was added to the mobile phase to adjust the pH. Other instrumental conditions were the same as described in the analytical method below, except for the flow rate, which was maintained at 0.8 mL min<sup>-1</sup>, and the use of various ratios of methanol–water. KNO<sub>3</sub> was used to determine the dead time.



**Figure 1.** Linear sorption isotherms of bentazone on different soils: Anji paddy soil (▼), Linan red soil (○), Yuhang paddy soil (■), Anji paddy soil (●), and Deqing paddy soil (▲). Points are observed values, while the line is the fit to the linear regression model.

**Analysis of Bentazone.** Equilibrium concentrations (*C<sub>e</sub>*) were analyzed on an LC-10ADvp high-performance liquid chromatograph (Shimadzu, Kyoto, Japan) equipped with an SPD-10Avp photodiode array detector, an SIL-10ADvp automatic injector, two HPLC pumps, and a CTO-10Avp oven and Class-vp data system. The HPLC conditions were as follows: Waters μBondapak C<sub>18</sub> column, 250 mm × 2.5 mm i.d.; 1 mL min<sup>-1</sup> flow rate; 55:45 methanol/water (pH = 2.8, adjusted with H<sub>3</sub>PO<sub>4</sub>) as mobile phase; detection wavelength, 218 nm; injection volume, 20 μL; and oven temperature, 30 °C.

## RESULTS AND DISCUSSION

**Sorption in Soils and Humic Acids.** The sorption behavior of bentazone in five selected soils was evaluated by both the modified linear isotherm model (1) and the Freundlich model (2),

$$q = K_d C_e \quad (1)$$

$$q = K_f C_e^{1/n} \quad (2)$$

where *q* is the sorbed amount (mg kg<sup>-1</sup>), *C<sub>e</sub>* is an equilibrium concentration (mg L<sup>-1</sup>), *K<sub>d</sub>* is the linear distribution coefficient, *K<sub>f</sub>* is the Freundlich empirical constant, and 1/*n* is an exponent that expresses the degree of isotherm nonlinearity.

The linear and the Freundlich models were fit to the isotherm data with linear regression. The isotherm data and the best-fit linear isotherms are shown in **Figure 1**. Best-fit regression coefficients are listed in **Table 2**.

As shown in **Table 2**, the experimental data of bentazone in five tested soils fit well to both linear and Freundlich models

**Table 2.** Fitting Results of Sorption Isotherms Using Linear Model and Freundlich Models

no.	pH <sup>a</sup>	linear isotherm			Freundlich isotherm			
		range (mg L <sup>-1</sup> )	K <sub>d</sub> (mL g <sup>-1</sup> )	K <sub>oc</sub> (mL g <sup>-1</sup> )	R	K <sub>f</sub> (mg <sup>1-n</sup> L <sup>n</sup> kg <sup>-1</sup> )	1/n	R
1	7.63	3–64	0.206	8.86	0.95	0.369	0.780	0.91
2	4.15	4–64	0.162	43.5	0.99	0.189	0.976	0.99
3	7.28	3–93	0.140	5.60	0.99	0.147	0.985	0.97
4	4.84	3–93	0.233	14.3	0.99	0.350	0.931	0.98
5	4.86	3–93	0.321	13.8	0.99	0.476	0.925	0.99

<sup>a</sup> Measured in equilibrium suspension.

with  $R > 0.91$ . The values of all correlation coefficients ( $R$ ) of linear models were greater than those from the Freundlich model, and the exponents,  $1/n$ , in the Freundlich model were close to unity. Accordingly, the linear model better described the sorption behavior of bentazone in soils than in the Freundlich model.

Linear isotherms are characteristic of herbicide partitioning between soil organic matter and water (19). Near unity values of  $1/n$  also support sorption due to partitioning (20). Hence, it is also possible to determine the affinity of soils to bentazone by comparing  $K_d$  values. The  $K_d$  values indicate that bentazone sorption in each soil, ordered from greatest sorption to least sorption, is soil 5 > soil 4 > soil 1 > soil 2 > soil 3. The values of  $K_d$  ranged from 0.140 to 0.321 mL kg<sup>-1</sup> for these five soils, in accordance with results reported by Donigian (5) and Gaston (7).

Shan et al. (21) proposed to classify pesticides with  $K_{oc}$  (organic carbon-normalized  $K_d$ ) values less than 50 as a very high mobility group and those having a  $K_{oc}$  value between 50 and 150 as a high mobility group. All values of  $K_{oc}$  in this study were less than 50 (Table 2). This suggests that bentazone has very high mobility with a possible leaching problem. These findings bring to attention possible water pollution caused by bentazone application, especially in the event that heavy rains transport the bentazone.

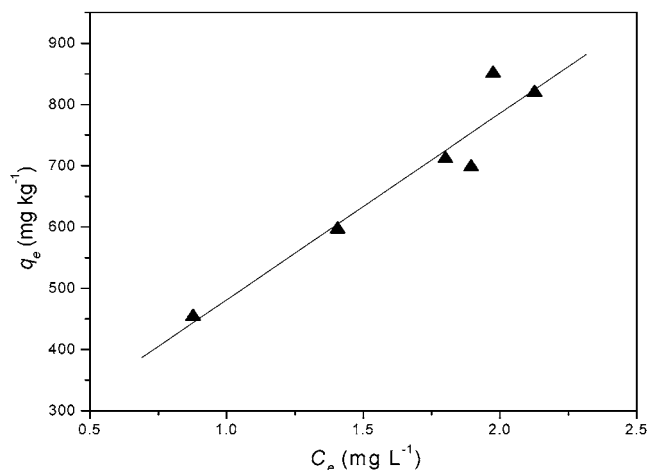
Organic matter, especially humic substances, plays a very important role in the fate and movements of herbicide in soils with strong sorption behavior (22, 23). Soil organic matter has been treated as a solid-phase partitioning medium for nonionic organic compounds (8).

The linear model fit experimental results for sorption of bentazone on HA over all tested concentrations, as shown in Figure 2. The sorption pattern of bentazone on HA was similar to that for all soils (Figure 1). The linear isotherm model indicates that a constant partitioning of herbicide occurred between solution and adsorbent, namely, sorption is directly proportional to the concentration solution phase over the whole concentration range investigated.

**Comparison of the Effect of pH on  $K_{ow}$  and  $K_{om}$ .** The aqueous solubility and the logarithm of the octanol–water partition coefficient (i.e.,  $\log K_{ow}$ ) are two important properties correlated with the sorption behavior of pesticides. For the dependence of  $\log K_{ow}$  on the  $pK_a$  and pH for ionic pesticides, Hu et al. (24) suggested a simple relation,

$$K_{ow} = K_{ow n} / (1 + 10^{(pH - pK_a)}) + K_{ow i} (1 - 1 / (1 + 10^{(pH - pK_a)}))$$

where the subscripts “o” and “w” refer to octanol and water phases, respectively, and  $K_{ow n}$  and  $K_{ow i}$  are  $[HA]_o/[HA]_w$  and  $[A^-]_o/[A^-]_w$ , respectively ( $[HA]$  and  $[A^-]$  are the concentrations of the neutral and ionic species of the pesticide, respectively). Chromatographic techniques have been widely used to calculate



**Figure 2.** Sorption isotherms for bentazone on a humic acid extracted from a paddy soil. ▲, experimental points; —, linear model regression. CaCl<sub>2</sub>, 0.013 M; pH = 2.83;  $R = 0.98$ ;  $K_d = 304.69$ .

$K_{ow}$ . HPLC retention times reflect the partitioning behavior between the nonpolar stationary and the relatively polar mobile phases. Braumann et al. reported that  $\log k_w$  could be used instead of  $\log P_{ow}$  as a hydrophobic parameter for herbicides (18).

The capacity factor,  $k$ , is defined as

$$k = (t_R - t_o) / t_o \quad (3)$$

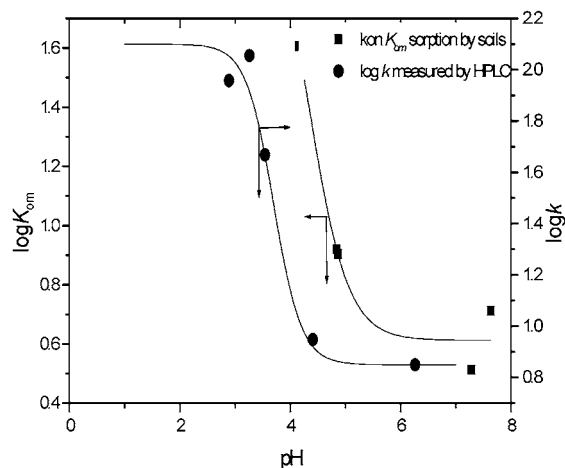
where  $t_R$  and  $t_o$  are the retention times of bentazone and KNO<sub>3</sub>, respectively.  $\log k_w$  was obtained by extrapolation to zero according to

$$\log k = \log k_w + S\varphi \quad (4)$$

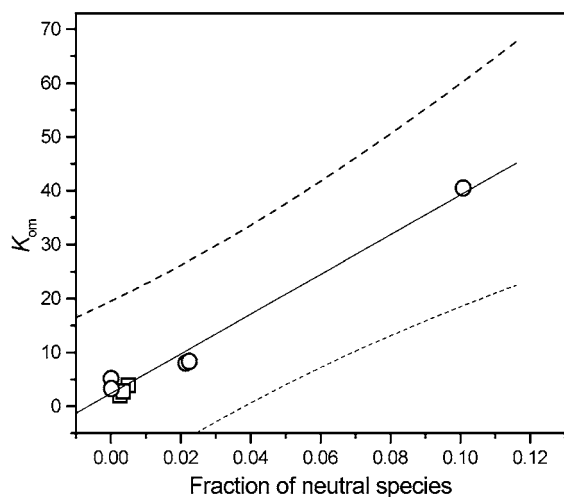
where  $\varphi$  is the volume fraction of organic solvent in the water–organic solvent mixture,  $k_w$  represents the capacity factor of a solute with pure water as the mobile phase, and  $S$  is related to the solvent strength of the pure organic solvent.

The dependencies of  $\log k_w$  and  $\log k_{om}$  on pH are shown in Figure 3. It was found that the  $\log k_w$  of bentazone is almost constant when the pH was less than 3.0, but decreased dramatically when the pH was increased from 3.0 to 4.4. Further, decreases were small at higher pH. The  $\log k_{om}$  relationship to pH was similar. The similarity between  $\log k_{om}$  and  $\log k_w$  versus pH suggests that the same mechanism may govern the effect of pH on  $k_{om}$ , i.e., partitioning of bentazone between aqueous solution and organic matter.

**Correlation of  $K_d$  and Soil Properties.** Correlations between sorption of nonionic compounds and soil organic carbon contents have been extensively studied (8, 9, 23). However, if the organic matter is less than 0.1%, clay and silt play an important role in the sorption of pesticides (25). Zachara et al. (26, 27) reported



**Figure 3.** Comparisons of the effect of pH on organic matter-normalized partition coefficient and on capacity factor. Arrows mean the curve corresponding to the coordinate.



**Figure 4.** Construction of a model to predict bentazone sorption in soils and confirmation. □, data extracted from Gaston (6, 7); ○, experimental points; —, linear fit; ···, 99% confidence intervals.

that ionized compounds, such as N-heterocyclic base, were sorbed preferentially as the cation. These findings support that the sorption was a function of sorbent CEC and solution of pH. Therefore, it is worthwhile to examine whether the  $K_d$  values measured in this study correlated with the soils' properties, including OM, CEC, slit, sand, and clay contents and suspension of pH. However, the respective correlation coefficients were 0.297,  $-0.323$ ,  $-0.510$ , 0.406, 0.083, and  $-0.605$ , indicating no apparent correlation between  $K_d$  and each of soils' properties.

As shown in the linear isotherms (Figures 1 and 2) and by the similarity of  $K_{om}$  and  $k$  in relation to pH (Figure 3), the sorption of bentazone in soils was primarily controlled by partitioning of neutral species on soil organic matter. The total sorption,  $q_{total}$ , however, is given by

$$q_{total} = q_n + q_{ion} \quad (5)$$

where  $q_{total}$  is the total amount of bentazone adsorbed,  $q_n$  is the amount of neutral species adsorbed, and  $q_{ion}$  is the amount of ionic species adsorbed. If sorption of bentazone in soils is due to partitioning of both neutral and anionic forms in soil organic matter, the effect of pH on sorption can be explained via its control on bentazone ionization. Linear sorption equations for total, neutral, and anionic bentazone are expressed as follows:

$$q_{total} = K_d C_e \quad (6)$$

$$q_n = K_d^n C_e^n \quad (7)$$

$$q_{ion} = K_d^{ion} C_e^{ion} \quad (8)$$

$$\alpha = 1/(1 + 10^{(pH-pK_a)}) \quad (9)$$

where  $\alpha$  is the fraction of neutral species. Substituting eqs 6–9 into eq 5 yields

$$K_d C_e = \alpha K_d^n C_e^n + (1 - \alpha) K_d^{ion} C_e^{ion} \quad (10)$$

Eliminating  $C_e$ , eq 10 takes the form

$$K_d = \alpha K_d^n + (1 - \alpha) K_d^{ion} \quad (11)$$

When divided by organic matter content of soil, eq 11 becomes

$$K_{om} = \alpha K_d^n / f_{om} + (1 - \alpha) K_d^{ion} / f_{om} \quad (12)$$

Rewriting eq 12, we obtain

$$K_{om} = \alpha(K_d^n - K_d^{ion}) / f_{om} + K_d^{ion} / f_{om} \quad (13)$$

If the partition coefficients normalized by organic matter for both neutral and anionic species of bentazone in different soils are constant, then a linear relationship is expected between  $K_{om}$  and  $\alpha$ , as shown in Figure 4. On the basis of laboratory data, the regression equation is

$$K_{om} = 368\alpha + 2.40 \quad (R = 0.99) \quad (14)$$

Equation 14 implies that both values of the pH of the soil solution and the organic matter contents in soil control bentazone sorption. Clearly, however, the neutral species is more adsorbed than the anionic form.

From the sorption coefficient  $K_d$ , measured for sorption bentazone on humic acids (HA) at pH 2.83, as shown in Figure 2, it was found that  $K_{om}^n = 434.65$ , very close to the calculated value (370.3) obtained using eq 14. This agreement also confirms that the sorption of bentazone in soils is primarily controlled by partitioning of neutral species on soil organic matter.

This model was also tested using results reported by Gaston et al. (6, 7) (Figure 4). The reported experimental values were closely fitted to the line by eq 14, which implies that the model can be applicable to predict the sorption of bentazone in soil containing even a limited difference of soil properties.

**Conclusion.** The sorption behavior of bentazone in extracted humic acids and soils having different physicochemical properties was best described by a linear model that suggests the partitioning of neutral soils on organic matter was the dominant mechanism. Experimental results also indicate that bentazone is highly mobile. A study of  $K_{om}$  and  $k$  in relation to pH showed that the distribution coefficient was a function of organic matter content and pH. These results led to a model for predicting bentazone sorption on the basis of organic matter content and pH.

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