



Adsorption and desorption behavior of herbicide diuron on various Chinese cultivated soils

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ABSTRACT

The adsorption–desorption behaviors of diuron were investigated in six cultivated soils of China. The effect of system pH and temperature were also studied. The data fitted the Freundlich equation very well. The adsorption K_F values indicated the adsorption of diuron in the six soils was in the sequence of black soil (D) > yellow earth (F) > paddy soil (B) > yellow-brown soil (C) > yellow-cinnamon soil (A) > lateritic red earth (E). The adsorption K_F and Freundlich exponents n were decreased when temperature was increased from 298 K to 318 K. However, the Gibb's free energy values were found less negative with the increasing temperature. Meanwhile, the extent of diuron adsorption on soil was at rather high level under low pH value conditions and decreased with increasing pH value. In addition, the desorption behavior of diuron in the six soils was in the sequence of lateritic red earth (E) > yellow-cinnamon soil (A) > paddy soil (B) > yellow earth (F) > yellow-brown soil (C) > black soil (D). At the same time, desorption hysteresis of diuron were observed in all of the tested soils. And the soil organic matter content may play an important role in the adsorption–desorption behavior.

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1. Introduction

Environment contamination from pesticides is raising concerns for the public and regulatory agencies. And their fate in soil is of main concern, as they bring a great threat to groundwater. Due to their low solubility and moderate to high hydrophobicity, many pesticides adsorb strongly to soil particles [1,2]. So adsorption of pesticide in soil is the key process that affects their ecotoxicological impact, environmental mobility and rate of degradation. Desorption process of pesticides is also important since it determines the release rate and the potential mobility of pesticides in soil. The adsorption/desorption behavior is especially important for herbicide, because the adsorption quantity and desorption rate decide the negative effect of the adsorbed herbicide on the succession crop.

Diuron (*N*-(3,4-dichlorophenyl)-*N*, *N*-dimethylurea) is a broad-spectrum residual herbicide registered for preemergent and postemergent control of both broadleaf and annual grassy weed. Diuron also has widespread been used in non-agricultural applications, especially industrial uses, where often in combination with other herbicides it provides total vegetation control. Diuron has been used for more than 40 years and it is an approval priority substance by the European Union Water Framework Directive

(Directive 2000/60/EC). The US Environmental Protection Agency (EPA) has classified diuron as a 'known/likely' carcinogen since 1997 based on the results of rats studies [3]. Some environmental behaviors about diuron have been reported, for example, photolysis [4], soil degradation [5], hydrolysis [6,7]. And the results showed diuron was relatively stable in water and not sensitive to light. Most of all, the degradation of diuron in soil was very slow. Dores's research [8] showed that diuron was detected in concentration decreasing until 70 days after application in runoff water and soil, totalizing 13.9% during the whole sampling period. Because of its high persistence, groundwater contamination by diuron has become a serious problem [9]. In Dutch coastal waters a higher level of diuron than the permitted 430 ng L⁻¹ was detected [10]. According to the French Environmental Institute, diuron was detected in surface waters of 34.6% in France where it was the fifth most frequently detected pesticides. It was also found in groundwater samples of 6.4% where it was the seventh most frequently detected pesticide [11]. In the United Kingdom (UK), diuron was consistently one of most frequently found pesticides exceeding the non-statutory Environmental Quality Standard of 0.1 μg L⁻¹ [12].

It is well known that the risk of pesticide to surface water and groundwater mainly comes from the adsorbed pesticide in soil, especially herbicide application on dry fields. Soil adsorption/desorption of pesticide and the influencing factor (organic matter, pH, temperature, etc) had been widely assessed [13–15]. Soil organic matter content appeared to be a predominant factor influencing pesticide retention [15,16]. In addition, the

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temperature and pH had different effect on pesticide adsorption [17]. Moreover, hysteresis phenomena was characteristic of the adsorption–desorption process in soil [18] but still the exact mechanisms of hysteretic adsorption/desorption of anionic, basic and neutral pesticides remained largely unknown [19]. The adsorption behaviors of diuron on soils had been reported by some researchers [20–23]. But the results were different, which may owe to the various soils and experimental conditions. Chaplain et al. [21] thought the soil hydrophobicity was an important factor for the diuron adsorbed. Other researchers [22,24] thought the organic matter was the key adsorbent for diuron. The composted cotton gin trash (CCGT) as a pesticide adsorption medium in remediation of diuron contaminated tailwater was also investigated [20]. The results showed the adsorption failed to reach equilibrium after two days, and the soil organic carbon–water partitioning coefficient (K_{OC}) was 526 mg kg^{-1} . The influence of humic substances [25] and lipids [26] on the adsorption of diuron to soils had been investigated. Forouzangohar et al. even predicted the diuron adsorption coefficient by midinfrared spectroscopy in combination with partial least-squares regression [27].

Most of the researches about diuron adsorption were carried out on the non-cultivated soils and the data could not be used for the risk assessment of diuron because the results were lack of representativeness. The application of diuron in China has been gradually improved in recently years, especially in the south China with a higher groundwater table and rotation system, the environmental risk of the herbicide must be assessed. In this work, the adsorption and desorption behaviors of diuron were investigated on six cultivated soils of China. Kinetics and isotherm adsorption experiments were carried out. Thermodynamic parameters of adsorption for diuron were calculated. Meanwhile, several factors affecting the adsorption efficiency of diuron, such as initial pH of solution and temperature were studied in detail.

2. Materials and methods

2.1. Chemicals

All reagents were of analytical grade unless specified otherwise. Analytical grade diuron, with a purity of 98.5%, was obtained from the National Standards Company (Beijing, China). The chemical structure and physico–chemical properties of diuron are shown in Fig. 1. The properties of diuron came from the Pesticide Properties Database (PPDB), which was developed by the Agriculture and Environment Research Unit (AERU) at the University of Hertfordshire.

2.2. Soil

The adsorption studies were carried out on six cultivated soils, representing different regions with diuron usage in China. In addition, the characteristics of the six soils are diverse and typical, which could be seen in Table 1. The soils were sampled in the surface

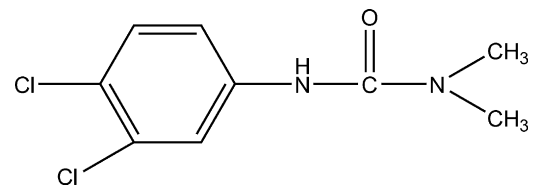


Fig. 1. Chemical structure and physico–chemical properties of diuron. The solubility of diuron in water is 35.6 mg L^{-1} (20°C), and the octanol–water partition coefficient is 741 (pH 7, 20°C).

layer (0–15 cm). These samples were air-dried, gently crumbled and sieved through 2 mm mesh. The methods for soil characteristics analysis were referred to Bao's book [28].

2.3. Adsorption experiments

2.3.1. Equilibrium time studies

Adsorption experiments were carried out using a batch equilibration technique. To minimize changes in ionic strength and to avoid dispersion, 0.01 M CaCl_2 was used as a background solution. Adsorption equilibrium studies were conducted for all the six soils. First, diuron was dissolved by methanol. Then the methanol solution was diluted by 0.01 M CaCl_2 solution to obtain the working solution for test. The concentration of methanol in the working solution was kept in 1%.

Samples of 5 g soil (<2 mm size) were added to 250 mL conical flasks containing 50 mL solution with diuron concentration of 10 mg L^{-1} . These studies were conducted in triplicate. The reaction mixtures were shaken on a horizontal shaker at 200 rpm for a period of 48 h at $25 \pm 1^\circ\text{C}$. From the flasks, 5 mL of supernate was collected (the sample was centrifuged for 5 min, 4000 rpm) at pre-determined time intervals of 0, 1, 2, 3, 6, 12, 18, 24 and 30 h. The diuron concentration was analyzed by HPLC without any cleanup.

2.3.2. Kinetic study

Adsorption kinetic studies were also conducted for all the six soils. The triplicate samples of 5.0 g sieved soils were added to 250 mL conical flasks containing 50 mL diuron solution at concentrations of 1, 2, 5, 10, 20, 50 and 100 mg L^{-1} , respectively. A blank sample was also kept to assess the effects of diuron adsorption onto the tubes and the following possible degradation during the process. After the addition of soil samples, the reaction mixtures were shaken on a horizontal shaker at $25 \pm 1^\circ\text{C}$ until the equilibrium was established. After the desired time (18 h), 5 mL of sample was collected from each flask, centrifuged at 4000 rpm for 5 min and the supernatant was collected for pesticide residue analysis.

2.3.3. Diuron adsorption enthalpy

The adsorption process was performed at two different temperatures (25°C and 35°C). Diuron concentrations were 1, 2, 5, 10, 20, 50 and 100 mg L^{-1} , respectively. Diuron adsorption enthalpy

Table 1
The characteristics of the six cultivated soils.

Soil	pH	Organic matter (%)	Organic carbon (%)	Cation exchange capacity (cmol kg^{-1})	Soil mechanical composition (%)	
					>1 mm	<1 mm
Yellow-cinnamon soil (A)	5.24	1.32	0.77	33	40.91	59.12
Paddy soil (B)	5.82	1.94	1.13	37	41.50	58.51
Yellow-brown soil (C)	6.84	2.08	1.21	27	19.53	80.52
Black soil (D)	8.06	2.62	1.52	46	24.71	75.34
Lateritic red earth (E)	6.43	0.58	0.34	38	32.12	67.90
Yellow earth (F)	6.74	2.92	1.69	34	61.61	38.40

in soils was determined using the batch experiments as described above.

2.3.4. Effect of pH on adsorption

The pH values of the 0.01 M CaCl₂ solutions were adjusted to 5.0, 7.0 and 9.0 with concentrated HCl or NaOH, respectively. The concentration of diuron was 1, 5, 10, 20 and 50 mg L⁻¹. Three typical soils were applied to determine pH effect on diuron adsorption in soils. The experiment was performed in the same way as the kinetic study described above.

2.4. Desorption experiments

Desorption experiments were performed immediately after the adsorption experiments. Supernatant liquid was decanted and the pesticide residue was analyzed. Then the decanted supernatant was replaced with equal volume of fresh CaCl₂ solution (0.01 M). The reaction mixtures were shaken on a horizontal shaker at 200 rpm for 12 h at 25 ± 1 °C. These steps were repeated four times consecutively.

2.5. Quantitative determination of diuron by HPLC

Chromatographic analysis was performed with a HP 1100 (Agilent technologies, Palo Alto, USA) system equipped with a diode array detector. The column was Zorbax Extend C18 (4.6 mm × 245 mm × 5 μm) (Agilent technologies, Palo Alto, USA). The absorbance wavelength was 280 nm. The flow phase was acetonitrile: distilled water (70/30, v/v). The total run time was 10.0 min. The retention time of diuron was 3.6 min. The injection volume was 20 μL.

2.6. Data analysis

Adsorption–desorption data were fitted to the Freundlich model (Eq. (1)) in log format. log K_F and 1/n are the adsorption parameters calculated from the linear regression (Eq. (2)).

$$C_s = K_F C_e^{1/n} \quad (1)$$

$$\log C_s = \log K_F + 1/n \log C_e \quad (2)$$

The Freundlich adsorption isotherm represents the relationship between the corresponding adsorption capacity C_s (mg kg⁻¹) and the concentration of the pesticide in solution at equilibrium C_e (mg L⁻¹). Value n represents the energy distribution of adsorption sites. The adsorption coefficient (K_F) was also calculated as a function of the organic carbon (OC) content and organic matter (OM) of the soil as following equations.

$$K_{OC} = K_F / \%OC \times 100 \quad (3)$$

$$K_{OM} = K_F / \%OM \times 100 \quad (4)$$

And the free energy change (ΔG, cal mol⁻¹) in the adsorption process could be calculated by the following equation [1]:

$$\Delta G = -RT \ln K_{OM} \quad (5)$$

where R is the gas constant (2.0 cal K⁻¹ mol⁻¹), and T is Kelvin temperature.

Hysteresis coefficient, H, is calculated for the adsorption–desorption isotherms according to the following equation:

$$H = (n_{\text{desorption}}) / (n_{\text{adsorption}}) \quad (6)$$

where n_{desorption} and n_{adsorption} are the Freundlich constants obtained for the desorption and adsorption isotherms, respectively.

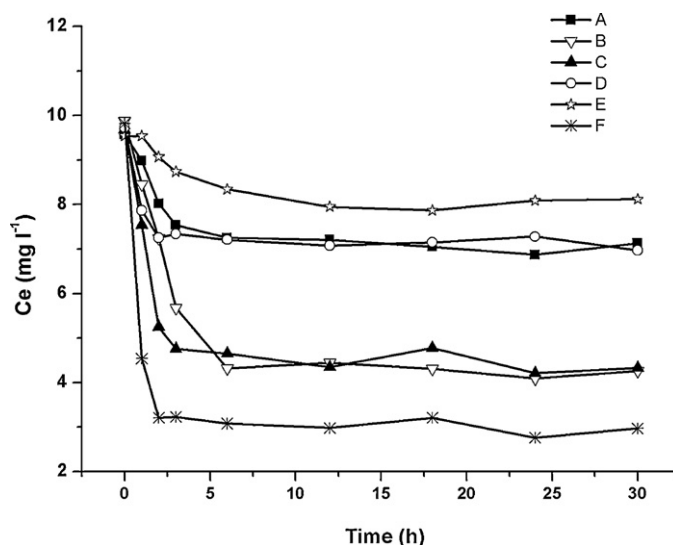


Fig. 2. The adsorption equilibrium time of diuron in six soils.

3. Results and discussion

3.1. The adsorption equilibrium time

A preliminary adsorption experiment was conducted to determine contact times required for adsorption equilibrium attained, and the results are presented in Fig. 2. The adsorption curves are single smooth and continuous leading to saturation. The adsorption kinetics exhibited two distinct stages, a very rapid adsorption in the initial stages (within 3 h) followed by a slow adsorption. This phenomenon was due to the fact that a large number of vacant surface sites were available for adsorption during the initial stage, and then the remaining vacant surface sites were difficult to be occupied due to repulsive forces between the solute molecules on the solid and bulk phases. The equilibrium was reached within 2 h (D and F), 3 h (C), 6 h (B and A), respectively. However, in soil E, the equilibrium was reached within 12 h, which may due to the least organic matter contained in the soil. Then 18 h was selected as the equilibrium time for the six soils in the next study.

3.2. The adsorption isotherm of diuron

In this study, no detectable of diuron degradation was found in the supernatant during the adsorption process. Thus, the reduced herbicide in solution was considered to be responsible only for soil adsorption. The plot of C_s versus C_e for various initial concentrations are shown in Fig. 3. Data were fitted well with Freundlich model (Table 2), as indicated by the high regression coefficients (R² = 0.97–0.98). The matching also indicates that adsorption process is not restricted to one specific class of sites and assumes surface heterogeneity. It has been stated by that magnitude of the exponent 1/n gives an indication of the favor-ability and capacity of the adsorbent/adsorbate system. Values n > 1 represents favorable adsorption conditions, and 1 < n < 10 shows beneficial adsorption. Nonlinear adsorption isotherms (n ≠ 1) were observed in all soils. Obviously, soil A (n = 1.04) showed more linear adsorption toward diuron than other soils. The adsorption isotherm shape provides information concerning the adsorption mechanisms. The soils of A, B, D and F showed a L-type isotherm (n > 1), which indicated that diuron could adsorb to the clay fraction of soil, besides organic matter. These isotherms have been previously reported for diuron adsorption [21,26]. C-type (n < 1) isotherms were found in C and E, which indicated that the adsorption of diuron in the two

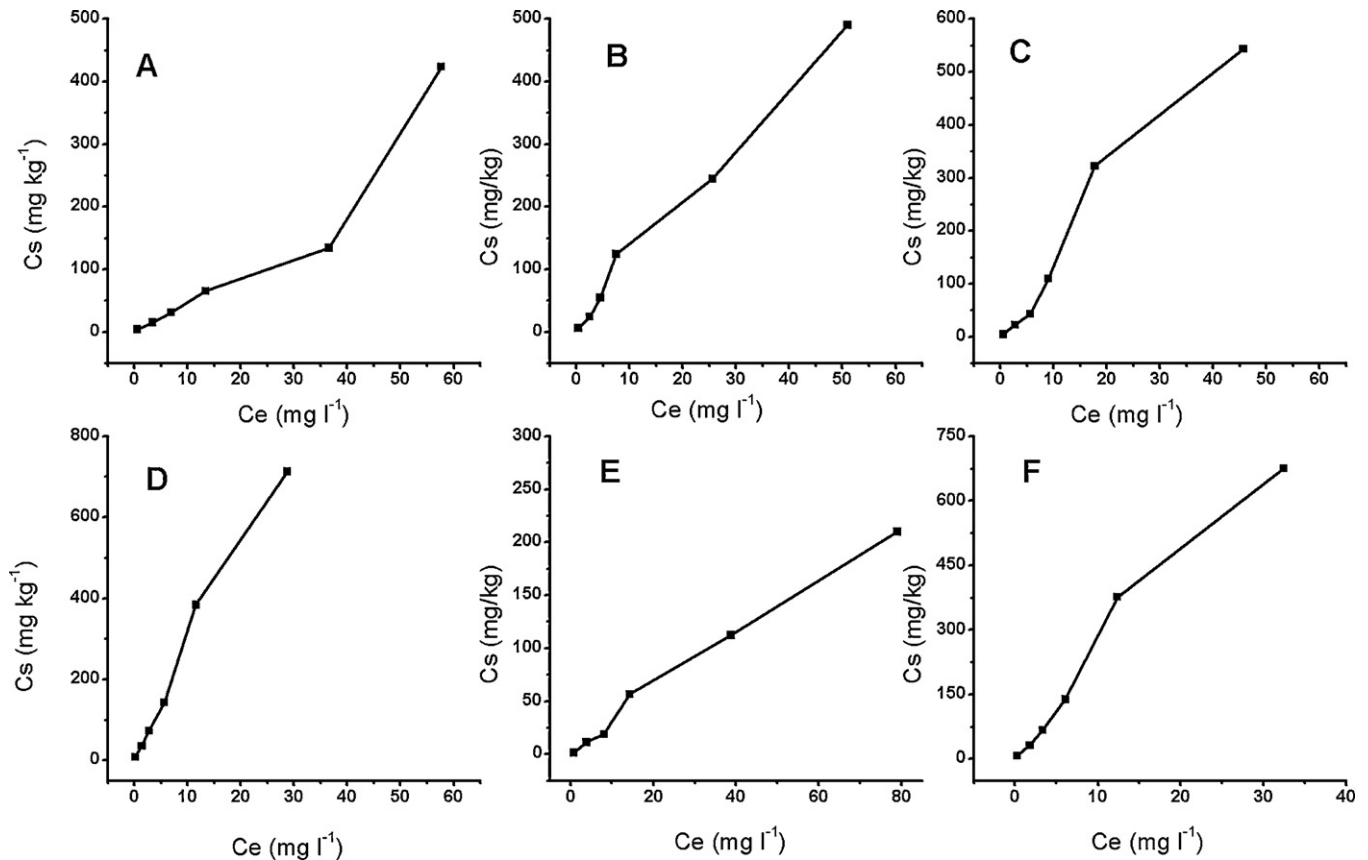


Fig. 3. The adsorption curve of diuron in six soils.

soils was mainly a partitioning mechanism between soil organic matter and soil solution. Soil organic matter plays a main role in adsorption of organic pesticides, which was proved in many researches [15,16]. In our study, 74.53% of the diuron was sorbed in soil D, which had the second highest organic matter content. In addition, 70.27% of the diuron was sorbed in soil F, which had the highest organic matter content. The K_F values suggested the adsorption of diuron in the six soils was in the sequence of $D > F > B > C > A > E$.

3.3. The effect of temperature on diuron adsorption

The effect of temperature on the adsorption of diuron was significant and the results are shown in Table 2 and Fig. 4. The increasing temperature caused a decrease in adsorbed diuron concentration at the same initial concentration, indicating that the process was

exothermic. Temperature may have an effect on the diuron solubility. At higher temperature, diuron solubility increases, showing lower tendency to get adsorbed in the soils. On the other hand, the exothermic adsorption of diuron on soil may be explained by the fact that the adsorbate-adsorbent bonds were weakened with increasing temperature. The K_F values decreased from 14.34 to 4.99 (soil B), 33.57 to 11.16 (soil D), and 25.98 to 18.00 (soil F), respectively. For soil B, the values of the Freundlich exponents n were also decreased when temperature increased from 298 K to 318 K, giving less linear for the adsorption isotherm shape. However, for soils D and F, the n values were firstly decreased then improved with the increasing temperature. Moreover, ΔG values was found less negative from $-4910.94 \text{ kJ mol}^{-1}$ to $-3164.18 \text{ kJ mol}^{-1}$ when the temperature was increased from 298 K to 318 K, indicating that the adsorption process led to an increase in Gibb's free energy and confirming the feasibility of the process and sponta-

Table 2
The adsorption parameters of diuron in soils.

Temperature (K)	Soil	K_F (mg kg^{-1})	n	R^2	Average adsorption rate (%)	K_{OC} (mg kg^{-1})	K_{OM} (mg kg^{-1})
298	A	5.68	1.04	0.97	34.34	737.41	430.16
	B	14.34	1.11	0.98	54.50	1268.59	738.92
	C	8.56	0.89	0.98	51.58	707.64	411.66
	D	33.57	1.12	0.99	74.53	2208.80	1281.44
	E	2.16	0.92	0.98	21.34	634.19	371.77
	F	25.98	1.07	0.98	70.27	1537.49	889.85
308	B	4.99	0.82	0.95	43.45	441.29	257.04
	D	11.16	0.94	0.96	55.20	734.10	425.89
	F	18.00	0.89	0.97	67.73	1064.91	616.34
318	B	1.46	0.60	0.97	22.36	129.16	75.23
	D	7.96	1.09	0.97	32.49	523.43	303.67
	F	4.80	1.01	0.95	41.35	284.19	164.48

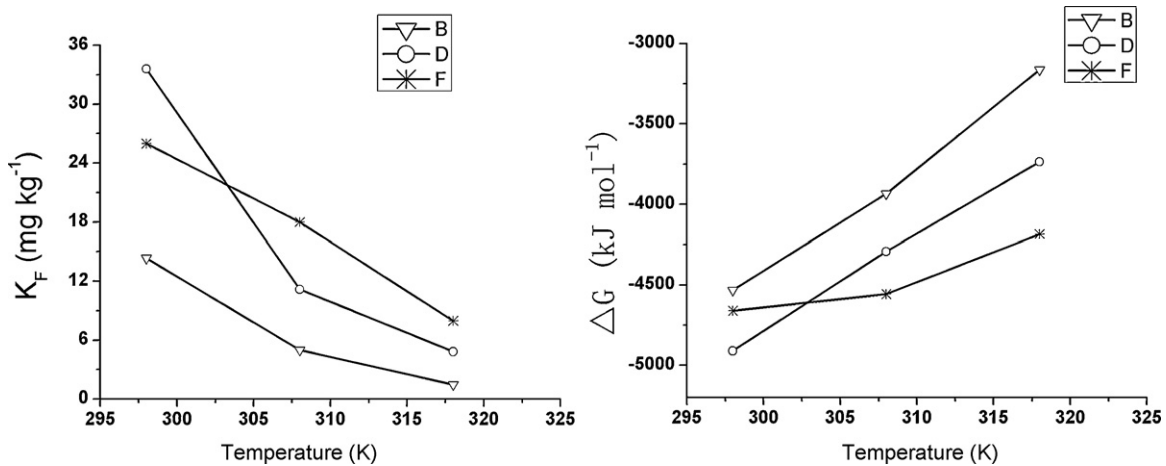


Fig. 4. The effect of temperature on the K_F value and free energy.

neous nature of the adsorption under the experimental condition (Fig. 4).

3.4. The effect of pH on diuron adsorption

The initial pH of the solution significantly affected the adsorption capacity of diuron. As shown in Fig. 5, the adsorption of diuron on the selected soils were rather high at low pH values and decreased with the increasing pH values of the suspension. Though the slopes of the adsorption isotherms (n) for D and F had little change with the increasing pH values, n values came from soil B fluctuated from 1.12 (pH 5) to 0.57 (pH 9). This indicated that the availability of the adsorption sites to diuron molecules reduced as the pH value increased, which accorded with the general trend observed for many pesticides [14,29,30]. The number of protonated surface on the adsorbent increases with decreasing pH and coulombic attraction forces between more positively charged surfaces, which were responsible for increasing adsorption with decreasing pH. For soil B, the K_F value was 17.21 mg kg^{-1} at pH 5, which was 177.3 times higher than those corresponding values at pH 9. But for soils D and F, the K_F values at pH 5 were only 6.4 and 9.0 times higher than the corresponding values at pH 9, respectively. The differences may come from the different concentration of organic matter, D and F with a higher OM% (organic matter content) may have a better buffer capacity for pH change. Because an

important chemical function of organic matter in soil is the source of pH buffering for soil.

3.5. Desorption behavior of diuron on soils

Desorption process of pesticides is also important since it determines the release rate and the potential mobility of pesticides in soil. And pesticide with a lower desorption rate may have a higher risk to the succession crop. In addition, desorption is one of the important factors considered for selecting the treatment options for the contaminated soils. Desorption kinetic studies were conducted to assess the desorption potential of adsorbed diuron and the results are shown in Table 3. Although all the tested soils had a lower desorption rate, the rate of desorption was quite fast in soil E as compared with other five soils. Like as the adsorption, the OM% may play an important role in the desorption process, in soil E which had the least OM%, but had the highest desorption K_F . And the desorption K_F values were 0.05, 0.03, 0.02, 0.01, 0.10, 0.02 mg kg^{-1} for the six soils, respectively. The K_F values suggested the desorption behaviors of diuron in the six soils was in the sequence of $E > A > B > F > C > D$, which looks like in the reverse order of adsorption K_F . Though the three soils, B, F and C were some inconsistent with the reverse performance, they had the similar K_F values. And the inconsistency may have some relationship with the soil mechanical composition. The soil with more loose compo-

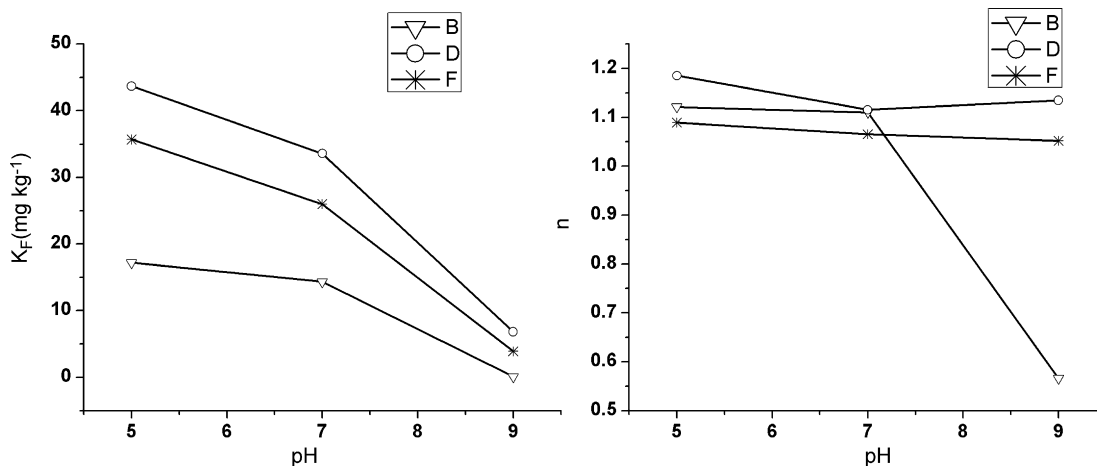


Fig. 5. The effect of pH on the diuron adsorption parameters.

Table 3

The desorption parameters of diuron in soils.

Soil	K_F (mg kg ⁻¹)	n	R^2	Average desorption rate (%)	K_{OC} (mg kg ⁻¹)	K_{OM} (mg kg ⁻¹)	Hysteresis index
A	0.05	1.03	0.99	31.46	6.63	3.87	0.99
B	0.03	0.98	0.99	26.11	2.71	1.58	0.88
C	0.02	1.06	0.99	10.87	1.24	0.72	1.19
D	0.01	0.98	0.99	6.88	0.43	0.25	0.87
E	0.10	1.02	0.99	48.90	29.69	17.40	1.11
F	0.02	0.97	0.99	21.71	1.42	0.82	0.91

sition (>1 mm) may have easier tendency for pesticide desorption. Because more loose composition meant more holes existed in the soil structure, which resulted in more fresh CaCl₂ solution could enter into the holes and wash the adsorbed diuron.

3.6. The comparison of adsorption and desorption

Following the widespread use of pesticides in the natural environment, herbicide behavior in soils greatly depends on adsorption–desorption phenomena and knowledge about these processes is important to predict their mobility in soil. Adsorption influences pesticides leaching in the subsurface and has to be counted when predicting pesticides transport. The adsorption/desorption behavior of diuron on six Chinese cultivated soils were investigated by batch experiments. Freundlich isotherm fitted the adsorption/desorption data well. Adsorption K_F value was highest for soil D, whereas, it was least for soil E. And the desorption K_F values had the reverse order. Multiple linear regressions were made between the adsorption/desorption K_F values and the various soil parameters, and two equations could be obtained:

$$\begin{aligned} \text{Adsorption } K_F &= 11.752 \text{ OM} + 0.964 \text{ CEC} - 41.956 \quad R^2 = 0.992 \quad (p < 0.05) \\ \text{Desorption } K_F &= -0.36 \text{ OM} + 0.106 \quad R^2 = 0.851 \quad (p < 0.05) \end{aligned}$$

From the equations, we could find that soil OM% had a significant positive effect on diuron adsorption, and cation exchange capacity also had some positive effect on the adsorption, though the effect was considerably lower than that of organic matter (from the coefficients could be compared). Meanwhile, the soil OM% was the only factor that had a significant negative effect on diuron desorption. So a decision could be made that the organic matter is the key factor that influences the adsorption/desorption behavior of diuron on Chinese soils. Because the high organic matter content can immobilize the pesticide effectively in the soils. Diuron was strongly adsorbed on soil D (with the second highest OM%). Accordingly, the desorption behavior was more difficult to happen. Moreover, we found an interesting performance, the soil pH (the pH of soil) values had insignificant effect on diuron adsorption, but the system pH (the pH of CaCl₂ solution) had a notable effect on the adsorption. The reason may come from the system pH that influenced the adsorption through adjusting the pesticide properties (e.g. solubility). Apart from this, the system pH also had an effect on the conformation of the organic matter, mainly in the molecules with an amphiphilic character and this also modified the sorption capacity of the soil.

3.7. The hysteresis in adsorption and desorption

Several studies have pointed out the occurrence of hysteresis in the adsorption–desorption reactions in soils [14,15]. Hysteretic adsorption/desorption suggests that adsorption of pesticides occurs with a limited degree of reversibility depending upon both the physico–chemical properties of the molecules and the soils involved. Generally, a value of hysteresis index close to 1 means that desorption process as quickly as sorption does; therefore, hysteresis is absent. On the other hand, a value of hysteresis index

lower than 1 indicates that the rate of desorption is lower than that of sorption, thus hysteresis takes place [16]. In the six soils, the hysteresis index was the highest for soil C and the lowest for soil D (could be seen in Table 3), which showed the soil D had the highest hysteresis effect. The entrapment of pesticide molecules within the condensed soil organic matter makes a significant contribution to adsorption–desorption hysteresis. In soils with high organic matter content, the adsorption–desorption hysteresis was mainly controlled by organic matter. And the soil mechanic composition may also play an important role in the desorption behavior. In view of the K_F values, the adverse effect of diuron on the succession crop should be noticed, especially when diuron was applied on soils with higher organic matter content, for example, soil D. On the other hand, we also should pay attention to the risk on groundwater that come from the diuron application on soils with lower organic matter content

4. Conclusions

Organic matter content had a positive effect on diuron adsorption, and cation exchange capacity also had some positive effect on diuron adsorption, though the effect was considerably lower than that of organic matter. However, the temperature and pH (solution) had a negative effect on diuron adsorption. Meanwhile, organic matter content was the only factor that had a significant negative effect on diuron desorption. Based on the results, we should prevent groundwater from the diuron risk when the application on the soil with lower organic matter, and we also should pay attention to the negative effect, which may result from the adsorbed diuron by the higher organic matter, on succession crop.

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