

DEGRADATION, REMEDIATION, AND CONSERVATION OF SOILS

Adsorption of the Herbicide Acetochlor by Different Soils Types

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Abstract—Adsorption of the herbicide acetochlor on eleven soils of different soil-geographical zones was studied. Distribution coefficients of acetochlor, K_d and K_{OC} , were calculated from the isotherms obtained. It was shown that the adsorption capacity of soils for acetochlor increases with increasing content of organic carbon and the specific surface. A hydrophobic mechanism was suggested for the adsorption of acetochlor by soils.

INTRODUCTION

Acetochlor (2-chloro-2-methyl-6-ethyl-*N*-ethoxy-methylacetanilide) is a relatively new and poorly studied chloroacetanilide herbicide used as a selective herbicide of systemic action. It is recommended for the treatment of corn, sunflower, and soybean plantations against annual grasses and some dicotyledonous weeds. The competent and safe application of acetochlor requires the study of its behavior in the soil. The wide diversity of soil types in Russia determines the topicality of studying the behavior of new herbicides in soils of different zones. In Russia, the acetochlor preparations Harness and Trophy are registered and authorized for use [17].

Acetochlor is an efficient herbicide, but it can also suppress crops planted subsequently. The half-life of acetochlor in soil (T_{50}) depends on the application rate and can vary from 8 [15] to 879 days [19]. Acetochlor is a persistent herbicide, and adsorption is the main process determining its further behavior in the soil [7]. Acetochlor-derived herbicides have been used in Russia since not very long ago; therefore, the behavior of acetochlor in soils of different soil-geographical zones, including its adsorption, is studied insufficiently. Most studies of acetochlor behavior were performed by foreign authors on soils untypical for Russia [9, 21, 23–25, 27, 28]. Russian works studies were carried out with no more than three soil types, which is insufficient for revealing the regularities in the behavior of acetochlor [6, 19]. Therefore, the study of the adsorption behavior of acetochlor on a representative sample set is a topical problem.

The aim of this work was to study the adsorption of acetochlor on soils from different soil-geographical zones and to determine its relationship with soil properties.

EXPERIMENTAL

The adsorption capacity of soils for acetochlor was studied on a continuous series of zonal soils: soddy-podzolic soils (3 samples), gray forest soils (3 samples), and chernozems (3 samples). The set of soils was expanded with samples of meadow-chernozemic and alluvial meadow soils (Table 1).

Soil samples were taken from the 0- to 10-cm layer of the humus-accumulative horizon, air-dried, and passed through a 2-mm sieve. A mixed sample was composed of the soil thus prepared by the quartering method and used for the characterization of the soil and the adsorption experiments.

The soils studied were characterized by physico-chemical parameters that best described their adsorption capacity for herbicides [8]. For this purpose, the pH_{water} , calcium and magnesium in water extract, organic carbon (Nikitin's modification of the Tyurin' method), and the C_{ha}/C_{fa} ratio were determined in the selected soil samples [1, 3, 14]. The particle-size analysis of the soils was performed using the pyrophosphate method according to the Dolgov and Lichmanova procedure [12]. The specific surface (S) was determined by the water vapor sorption method (Kutilek) [24].

Studying the adsorption capacity of soils for acetochlor. The acetochlor-binding capacity of the soils was studied by the adsorption isotherm method. For this purpose, 5-g samples of prepared soil were placed in the centrifugal tubes with screw caps, and 25 ml of acetochlor solutions in water with increasing concentrations (0.05, 0.5, 2.3, 4.5, 6.8, 22.8, and 45.5 mmol/l) were added. The ionic strength of the herbicide solutions was maintained constant by adding 0.1 M KCl; the solution pH was adjusted to 5.5 with 0.1 KOH or HCl.

Table 1. The main physicochemical parameters of the soils studied

Soil, index	C_{org} , %	C_{ha}/C_{fa}	pH_{water}	Ca^{2+}	Mg^{2+}	S , m^2/g
				meq/100 g soil		
Loamy soddy-podzolic soil (Moscow oblast):						
virgin, P_v^s	4.11	0.4	5.5	nd	nd	119
cultivated, P_c^s	1.22	0.9	7.6	7.1	1.4	67
anthricultural, P_a^s	4.92	0.3	7.4	9.2	2.1	125
Virgin sandy loamy gray forest soil (Tula oblast), GF_v	1.61	1.1	6.6	1.8	1.3	88
Arable clay loamy gray forests soil (Vladimir oblast), GF_{ar}	1.35	1.4	6.1	1.7	1.1	99
Virgin loamy dark-gray forest soil (Tula oblast), GF^d	4.53	1.0	7.1	4.1	2.2	88
Deep clay loamy typical chernozem (Voronezh oblast), CH_d^1	8.33	2.1	6.8	2.9	1.1	259
Clay loamy typical chernozem (Lipetsk oblast), Ch^1	4.67	1.5	5.9	2.8	1.4	192
Clay loamy ordinary chernozem (Kursk oblast), Ch^o	3.57	1.6	6.9	1.66	1.3	154
Clay loamy meadow-chernozemic soil (Voronezh oblast), Chl	3.44	1.6	7.9	3.5	2.8	190
Loamy sandy alluvial meadow soil (Tula oblast), Al	2.66	1.5	7.2	3.7	0.7	114

Note: Here and below, (S) is the specific surface.

After addition of solutions, the tubes were closed and shaken at room temperature for 24 h. This exposure time was sufficient for the attainment of the equilibrium in the system, because it takes 6 h to attain equilibrium, according to our preliminary data. The variance analysis of the data on the kinetics of the acetochlor adsorption by the three soddy-podzolic soils showed no significant changes in the equilibrium herbicide concentration after 6, 24, and 48 h of soil-herbicide interaction.

After 24 h of interaction, the tubes were centrifuged at 5000 rpm for 15 min. The equilibrium concentration of acetochlor was determined in the supernatant. The content of adsorbed herbicide was calculated from the difference between the initial and equilibrium concentrations. The concentrations of acetochlor were determined by fluorescence polarization immunoassay [23] using AMDA-EDF Rf 0.88 tracer (acetochlor labeled with fluoresceinthiocarbamyl ethylenediamine) and polyclonal antibodies (rabbit antiacetochlor BSA lot 6 13.07.99) specific to acetochlor [22]. The determination of 10 samples took 7 min. The fluorescence polarization was measured on a TDx analyzer (Abbott, U.S.A.).

This method was used for the determination of acetochlor in salt extracts from soils for the first time; therefore, we preliminary studied its potential under selected conditions. It was found that the acetochlor concentration determined in a 0.1 KCl extract from soil exceeds its application rate by 30–50% depending on the soil studied. This effect is apparently due to the presence of dissolved organic substances, whose inter-

actions with the antigen result in overestimating the results of the analysis of the soil extract. Therefore, herbicide calibration solutions were prepared using a 0.1 M KCl extract from the corresponding soil rather than distilled water. This allowed us to eliminate the interfering effect of dissolved organic matter.

RESULTS AND DISCUSSION

The physicochemical properties of the soils studied are given in Table 1.

In the studied series of soils, the content of organic matter varied from 1.2 (P_c^s) to 8.3% (Ch_{thd}^1), which agrees well with literature data [13]. The relatively high content of organic carbon in the cultural soddy-podzolic soil (4.9%) is explained by the regular application of organic fertilizers on the experimental plot of the Chashnikovo Training-Experimental Soil-Ecological Center, where the soil samples were taken.

The C_{ha}/C_{fa} ratio regularly increased from soddy-podzolic soils to chernozems. The lowest C_{ha}/C_{fa} value (0.3) was observed in the P_a^s soil; the highest value (2.1) was typical for Ch_d^1 . The humus type varied in the soil series from the fulvate (P_a^s and P_v^s) and humate-fulvate (P_c^s) to fulvate-humate (GF_v , GF_{ar} , GF_d , Ch_t , Ch_o , Chl , and Al) and humate (Ch_d^1) types. The C_{ha}/C_{fa} ratios in all the soils, except for the cultural soddy-podzolic soil, corresponded to the typical ranges of corre-

Table 2. Particle-size composition of the soils studied

Soil	Content of fractions, %, with particle size, mm						
	2–0.25	0.25–0.5	0.05–0.01	0.01–0.005	0.005–0.001	<0.001	<0.01
P _v ^s	5.9	21.2	44.6	7.1	7.7	13.5	28.2
P _c ^s	4.0	17.6	57.4	8.3	7.8	16.9	28.9
P _a ^s	9.5	22.4	42.9	3.3	13.2	8.6	25.1
GF _v	0.5	22.4	50.3	8.8	8.5	9.5	26.8
GF _{ar}	0.1	6.2	48.3	13.0	11.3	21.1	45.4
GF ^d	0.7	8.9	59.2	9.2	9.0	13.0	31.3
CH _d ^t	0.2	20.6	25.2	6.7	25.2	22.1	54.0
Ch ^t	0.4	3.0	40.3	7.2	15.1	33.9	56.2
Ch ^o	0.4	2.3	42.3	8.1	15.4	31.5	55.0
Chl	1.2	19.1	23.1	12.3	7.3	37.0	56.6
Al	6.2	53.0	23.2	2.0	4.2	11.4	17.6

sponding soil types [10]. The low C_{ha}/C_{fa} ratio for the P_a^s (0.3) was probably due to the regular application of organic fertilizers.

The lowest pH value (5.5) was typical for the P_v^s soil, and the highest value (7.9) for the Chl soil, which agrees with literature data [13]. Unusually high pH values were observed for the P_a^s and P_c^s soils (7.6 and 7.4, respectively), which can be explained by regular liming. This supposition is confirmed by the high content of calcium in the water extracts from both soils (7.1 and 9.2 meq/100 g, respectively).

The specific surface of the soils under study varied from 67 (P_c^s) to 259 m²/g (Ch_d^t). The highest values were observed for chernozems, and the lowest values for the arable soddy-podzolic soil. The obtained values agree with the literature data [2, 4, 20]. According to Zubkova and Karpachevskii [4], the specific surface in the humus-accumulative horizons of zonal soils varied from 30 m²/g in soddy-podzolic soils to 145 m²/g in chernozems.

The particle-size composition of soil largely determines the behavior of herbicides [8], and the content of physical clay (particles <0.01 mm) is of classification significance; therefore, a complete particle-size analysis was conducted for all the soils (Table 2).

The content of physical clay (particles <0.01 mm) in the soil samples under study varied from 17.6 (alluvial meadow soil) to 56.6% (meadow-chernozemic soil); the content of the clay fraction (<0.001 mm) varied

from 8.6 (P_a^s) to 37.0% (Chl). The obtained data agree well with the results reported in the literature [13] and allow the soils to be classified by their texture [12]. The alluvial meadow soil was graded as loamy sandy; all the soddy-podzolic varieties and the virgin gray forest soil were classified among sandy loamy soils; the dark gray forest, arable gray forest, and all the chernozemic soils were classified as clay loamy soils.

The isotherms of acetochlor adsorption by the soils of different types as derived from the adsorption experiments are given in Fig. 1.

It is seen that the adsorption isotherms of acetochlor in all the studied soils were characterized by a gentle initial slope (in the equilibrium concentration range of acetochlore from 0.03 to 0.08 mmol/l) followed by an abrupt increase. The shape of the isotherms indicates that, when the equilibrium concentration of acetochlor in the solution is higher than 0.08 mmol/l, a complete saturation of the soil adsorption sites takes place and, hence, multilayer adsorption begins. This type of adsorption is characterized by an exponential increase in the amount of adsorbed substance [11]. This supposition was confirmed by the fact that, for soils with relatively low specific surfaces, the exponential increase was observed at lower equilibrium acetochlor concentrations compared to soils with higher specific surfaces. Thus, an abrupt increase was observed at acetochlor concentrations of 0.03–0.04 and 0.06–0.08 mmol/l for gray forest soils (*S* = 88–99 m²/g) and chernozems (*S* = 154–259 m²/g), respectively.

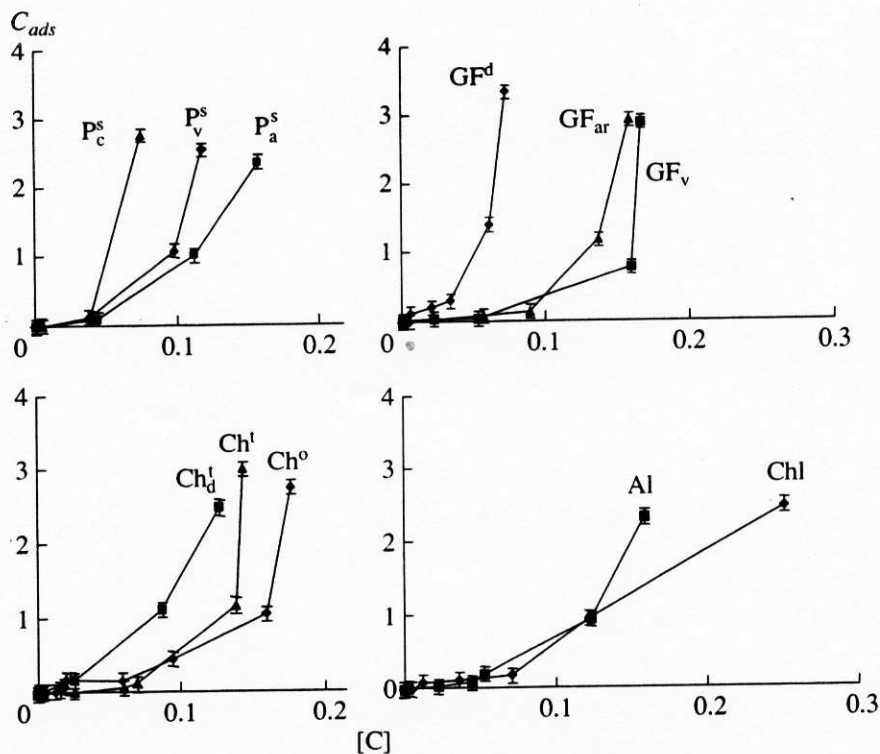


Fig. 1. Adsorption isotherms of acetochlor on soils of different soil-geographical zones; here and below: (C_{ads}) is the adsorbed acetochlor, mmol/kg; ($[C]$) is the acetochlor equilibrium concentration, mmol/l.

To determine the type of isotherms of acetochlor adsorption by soils, the initial region of an isotherm was studied in more detail (Fig. 2). The analysis of the initial isotherm region showed that it is the S-type according to the classifications by Giles [11]. This isotherm type indicates that the interaction force between the adsorbed herbicide molecules is higher than that between the dissolved herbicide and the soil; the adsorbate molecules are arranged on the adsorbent surface in chains and clusters, which is typical for the hydrophobic adsorption mechanism.

For the quantitative evaluation of the adsorption capacity of the soils, we used the distribution factor of acetochlor between the volume and the surface phases

$$K_d = \frac{C_{ads}}{[C]}, \quad (1)$$

where K_d is the factor of distribution; C_{ads} is the content of acetochlor adsorbed by the soil, mol/kg; and $[C]$ is the equilibrium concentration of acetochlor, mol/l.

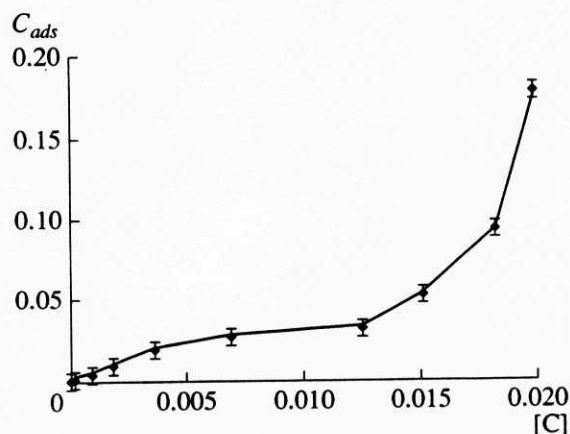


Fig. 2. Initial adsorption isotherm of acetochlor by the ordinary chernozem.

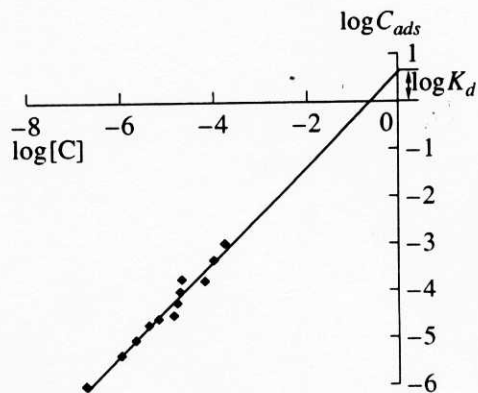


Fig. 3. Determination of K_d from the linearized adsorption isotherm of acetochlor by the ordinary chernozem.

To determine the numerical values of K_d , the linearization of isotherms was performed by taking the logarithm of Eq. (1) according to [5]:

$$\log C_{ads} = \log [C] + \log K_d. \quad (2)$$

In the plot of the linearized isotherm in the coordinates $\log [C] - \log C_{ads}$ (Fig. 3), a straight line drawn through it at an angle of 45° cuts off a segment of the ordinate axis, which is equal to $\log K_d$. The content of adsorbed herbicide was expressed in mol/kg, and its equilibrium concentration was expressed in mol/l; hence, the K_d value is expressed in l/kg.

It is notable that the factor K_d mainly characterizes the initial region of the isotherm and does not account for multilayer adsorption. This method of calculation describes the distribution of acetochlor in the range of concentrations typical for real agroecosystems: according to application rates, the initial herbicide concentration in the soil cannot be higher than 13.0–20.0 mmol/l at the total rate of 500 l/ha [17]. Along with the parameter K_d describing the distribution of acetochlor between the solution and the soil, we calculated K_{OC} , the distribution coefficient normalized to the content of organic carbon in the soil, using the following formula:

$$K_{OC} = K_d / C_{org} \times 100\%, \quad (3)$$

where C_{org} is the content of organic carbon in the soil, %.

The parameter K_{OC} is widely used for assessing the environmental hazard of pesticides [16]. Being normalized to the C_{org} content, this parameter characterizes the affinity of soil organic matter for the herbicide.

The values of K_d and K_{OC} found for acetochlor are given in Table 3. The K_d range for the soils studied was 2.1–18.6 l/kg. For comparing our results with the literature data, K_d values were calculated from the reported isotherms of acetochlor adsorption by soils [27, 32]. The values found values were in the range from 0.1 to 10.6 l/kg, which agrees well with our data.

The lowest K_d value (2.1 l/kg) was observed for the arable gray forest soil; the highest value (8.6 l/kg) was found for the deep typical chernozem. By the K_d values, the soils studied formed the following sequence:

$$GF_{ar} < GF_v < Ch^o \leq Al < P_c^s < Chl < Ch^t < GF^d < P_v^s \leq P_a^s < Ch_d^t.$$

The results obtained are not sufficient for revealing a correlation between the soil type and its adsorption capacity for acetochlor. However, a clear dependence of the K_d value on the content of organic carbon is observed within the soil types. For the soddy-podzolic soils, the following sequence was obtained for K_d and organic carbon:

$$P_c^s < P_v^s \leq P_a^s.$$

Table 3. Distribution factors (K_d and K_{OC}) of acetochlor for different soil types

Soil	K_d , l/kg	K_{OC} , l/kg C_{org}
P_v^s	10.2 ± 0.5	249 ± 12
P_c^s	5.4 ± 0.3	441 ± 22
P_a^s	10.3 ± 0.5	210 ± 11
GF_v	3.5 ± 0.2	217 ± 11
GF_{ar}	2.1 ± 0.1	156 ± 8
GF^d	9.1 ± 0.5	200 ± 10
Ch_d^t	18.6 ± 0.9	224 ± 11
Ch^t	8.6 ± 0.4	185 ± 9
Ch^o	4.7 ± 0.5	133 ± 7
Chl	6.6 ± 0.3	192 ± 10
Al	4.8 ± 0.2	181 ± 9

Note: (±) confidence interval calculated for $P = 0.95$, $n = 3$.

The gray forest soils and chernozems formed similar sequences:

$$GF_{ar} < GF_v < GF^d,$$

$$Ch^o < Ch^t < Ch_d^t.$$

From the data presented, a conclusion may be drawn that the content of organic matter is the main factor determining the acetochlor adsorption capacity of soils that are similar in their mineralogy and humus quality.

According to the K_{OC} values, the soils under study formed the following sequence:

$$Ch^o < GF_{ar} < Al \leq Ch^t \leq Chl < GF^d \leq P_a^s \leq GF_v < Ch_d^t < P_v^s \ll P_c^s.$$

The range of obtained K_{OC} values was 133–441 l/kg C_{org} . These values are close to those reported in the literature (118–311 l/kg C_{org}) [16, 25]. An anomalously high K_{OC} value for the cultivated soddy-podzolic soil (441 l/kg C_{org}) is notable, which exceeds its values for all other soils (133–249 l/kg C_{org}) by almost two times. This can be explained by the low C_{org} content in this soil (1.22%, which is the lowest value among all the soils studied). The low C_{org} content apparently results in the incomplete blocking of mineral adsorption sites by organic matter and, hence, increases the role of the mineral soil component in the adsorption of acetochlor by the soil. Taking into account that the mineral component significantly contributes to the adsorption capacity of this soil, it is clear that the normalization of the distribution factor to the C_{org} content results in an overestimation of the K_{OC} value. Therefore, the data for the

Table 4. Correlation between the acetochlor adsorption capacities of the soils studied and the soil properties

Parameter	C_{org} , %	C_{ha}/C_{fa}	pH_{water}	Ca ²⁺	Mg ²⁺	S , m ² /g	Fraction content, %	
				meq/100 g			<0.001 mm	<0.01 mm
K_d	0.94	0.12	-0.06	0.12	-0.03	0.68	-0.01	0.20
K_{OC}	-0.22	-0.35	0.24	0.44	-0.08	-0.34	-0.28	-0.35

Note: The significant Spirman correlation coefficients ($\alpha = 0.05$, $n = 11$, $r \geq 0.61$) are given in bold type.

cultivated soddy-podzolic soil were not considered when the K_d and K_{OC} values were compared.

To compare the K_d and K_{OC} values and estimate their variabilities, coefficients of variation (v) were calculated for both data sets. The coefficients of variation were 60.3 and 17.3% for the K_d and K_{OC} values, respectively.

The lower variability of K_{OC} compared to K_d indicates that the adsorption of the herbicide in the soil is related to its binding with organic matter [18]. The differences in the K_{OC} values among the soils are determined by the different affinities of organic matter for acetochlor in these soils.

From the data obtained, the ecological-agrochemical assessment of the acetochlor hazard in different soils was carried out using a combined scale of pesticide hazard, which accounts for the octanol-water coefficient, solubility, and K_{OC} of a pesticide and characterizes its mobility [16]. According to this approach, the pesticide is assigned to a specific class of hazard from the value of each parameter and is defined by a number of points. These numbers are then summed up, and the total index of ecological-agrochemical hazard is determined.

The found acetochlor K_{OC} values were in the range 75–499 l/kg C_{org} , which characterized it as a medium-mobile herbicide (3 points) in all of the soils studied,

according to the classification used. However, some authors studying the migration of acetochlor in the soil profile classify it as a weakly mobile herbicide [9, 15, 19, 21]. Acetochlor belongs to low-mobile herbicides (2 points) by the octanol-water coefficient logarithm ($\log K_{ow} = 3.03$) and to medium-mobile herbicides (2 points) by the solubility (23 mg/l). Thus, the total index of the ecological-agrochemical hazard of acetochlor is 7 in all the soils studied. For comparing, a similar classification analysis was performed for herbicides of other classes. The data reported by Spiridonov *et al.* [16] were used in the calculations. According to the results obtained, herbicides may be arranged in the following decreasing sequence according to their ecological-agrochemical hazard:

dicamba (14 points) > chlorsulfuron (13) > triasulfuron (12) > imazetapir (11) > pyrazosulfuron-ethyl (10) \approx picloram (10) > amidosulfuron (9) > metolachlor (8) > atrazine (7) \approx acetochlor (7).

Thus, acetochlor occupies the last position in the considered herbicide sequence, which characterizes it as the least hazardous herbicide.

To reveal the relationships between the physico-chemical properties of soils and their adsorption capacity for acetochlor, correlation analysis between the obtained distribution coefficients of acetochlor (K_d and K_{OC}) and soil properties was performed (Table 4).

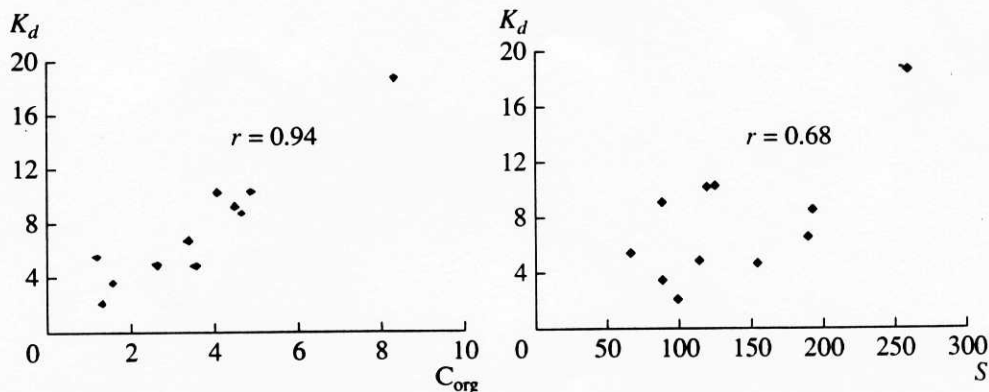


Fig. 4. Plots of K_d vs. the organic carbon and the specific surface of soils.

On comparing the acetochlor K_d values and the physicochemical parameters of soils (Table 4), significant correlations were found with the content of organic carbon ($r = 0.94$) and the specific surface ($r = 0.68$) (Fig. 4).

Taking into account that the specific surface in the humus-accumulative horizons of soils is largely determined by the content of organic matter [2, 20], the revealed relationships can indicate the predominant role of soil organic matter in the development of the adsorption capacity for acetochlor. The high octanol-water coefficient of acetochlor ($\log K_{OW} = 3.03$) and the shapes of the adsorption isotherms also indicate the higher affinity of the herbicide to hydrophobic adsorption sites on soil organic matter [32]. The additional proof of the leading role of organic matter is provided by the lower variability of K_{OC} compared to K_d , the similar tendencies in the changes of K_d and organic carbon in soils, and no correlation between the K_{OC} values and all of the considered soil properties.

Thus, the comparison of the adsorption capacity of soils for acetochlor and their physicochemical properties attests to the leading role of soil organic matter in the adsorption of acetochlor.

CONCLUSIONS

(1) The adsorption capacity of different soils for acetochlor is characterized by the K_d range 2.1–18.6 l/kg and the K_{OC} range 133–441 l/kg C_{org} , which qualify acetochlor as a moderately mobile herbicide.

(2) It was shown that the adsorption capacity of soils for acetochlor depends on the content of organic matter and the value of the specific surface. The leading role of organic matter in the development of the adsorption capacity of soils for acetochlor was revealed.

(3) The shape of the isotherms of acetochlor adsorption by soils and the correlation between the acetochlor K_d values and the content of organic carbon in the soil suggest the hydrophobic mechanism of its adsorption by soil.

(4) The ecological-agrochemical parameters show that the environmental hazard of acetochlor is lower than that of other herbicides such as dicamba, chlorsulfuron, triasulfuron, imazetapir, pyrazosulfuron-ethyl, picloram, amidosulfuron, and metolachlor.

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