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Adsorption–desorption behaviour of clomazone in Regosol and Chernozem agricultural soils

RADA D. ĐUROVIĆ-PEJČEV^{1*}, SVJETLANA B. RADMANOVIĆ², ZORICA P. TOMIĆ²,
LAZAR M. KALUĐEROVIĆ², VOJISLAVA P. BURSIĆ³ and LJILJANA R. ŠANTRIĆ¹

¹*Institute of Pesticides and Environmental Protection, Banatska 31b, 11080 Belgrade, Serbia,*

²*University of Belgrade, Faculty of Agriculture, Nemanjina 6, 11080 Zemun-Belgrade, Serbia*

and ³*University of Novi Sad, Faculty of Agriculture, Trg D. Obradovića 8,
21000 Novi Sad, Serbia*

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Abstract: Studies of adsorption and desorption of pesticides by soils are important for understanding and predicting their fate and transmission in the environment. Considering the agricultural and environmental relevance of clomazone, its sorption–desorption behaviour was studied in two widespread Serbian agricultural soil types named Regosol and Chernozem. Both phenomena are well-described by the Freundlich equation, which shows that clomazone is generally sorbed more to organic matter than to the mineral soil fractions. Chernozem, a soil containing more of both organic matter and clay, was found to bind more, and desorb less herbicide, than Regosol. Higher desorption hysteresis obtained for Chernozem could be attributed to its larger number of high-energy sorption sites, compared to Regosol. In both soils, the hysteresis effect increases with the rise of initial clomazone concentration in the soil-water system, while the percentage of desorbed amount during successive desorption cycles decreases. The presented adsorption–desorption study shows that soil composition plays an important role in clomazone behaviour and fate in the environment, and a significantly reduced probability of contamination of both the deeper soil layers and groundwater may be expected when this herbicide is found in humus-rich soils.

Keywords: pesticides; sorption; soil; modelling.

INTRODUCTION

Clomazone (IUPAC: 2-(2-chlorobenzyl)-4,4-dimethyl-1,2-oxazolidin-3-one) is an isoxazolidinone herbicide used to control annual grasses and broadleaf weeds in many crops, including soybean, rice, tobacco, cotton, pea, maize, oil-seed rape, sugar cane, cassava and pumpkin crops.¹ In Serbia, clomazone has found significant use in soybean, tobacco and oil seed rape weed control pro-

* Corresponding author. E-mail: rada.djurovic@pesting.org.rs; rajcica76@gmail.com
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grammes. Six products based on this active ingredient, containing 360 or 480 g a.i. L⁻¹, are registered in domestic market here in Serbia.² Its physicochemical properties indicate that this herbicide has relatively high solubility in water (1100 mg dm⁻³), and is not rapidly degraded by environmental fate processes such as photolysis or hydrolysis under a wide range of pHs.¹ Also, its properties, such as moderate mobility (K_{OC} : 150–562 dm³ kg⁻¹) and persistence in soil (DT_{50} : 30–135 days),¹ as well as potentially harmful effects on cultivated plants after crop rotation,³ make clomazone as a possible environmental contaminant of agricultural relevance. Additionally, clomazone could be classified^{4–6} as a pesticide of concern due to its possible impact on aquatic systems because its solubility in water is ≥ 30 mg dm⁻³, organic carbon normalized sorption coefficient (K_{OC}) is 300–500 dm³ kg⁻¹, sorption distribution coefficient (K_d) is < 5 dm³ kg⁻¹, photolytic half-life ($t_{1/2}$) is > 3 days, and microbial degradation $t_{1/2}$ is > 2 –3 weeks.

However, when clomazone molecules get into the soil, their further behaviour will depend on many factors, including the type and characteristics of the soil, frequency and quantity of precipitation and irrigation, temperature, *etc.* Among all fate processes in which clomazone may participate, sorption-desorption are the most important because they determine clomazone amounts in soil solution, and consequently could affect its mobility, degradation and plant uptake, since these processes involve only the free (non-sorbed) molecules fraction.

Considering that soils are a very heterogeneous mixture, consisting of mineral and organic components with variable composition and surface activity which participate in the binding of pesticides, knowing their adsorption-desorption behaviour in various soil types is very important for understanding and predicting of sorption mechanisms, persistence, mobility and other fate processes in soil environments.

Available literature data indicate that clomazone sorption to soil occurs either through organic carbon (OC)^{7,8} or both OC and clay fractions of soil.⁹ Additionally, some researchers have reported that soils collected from sites with a history of burned vegetation events had higher clomazone retention.^{7,10} Desorption of pesticides is also a very important process because it determines the rate and amount of pesticides that could be released from the soil, and their potential mobility in soil.^{11,12} However, there is only one report to our knowledge that has focused on the desorption behaviour of clomazone in this medium.⁷ Moreover, that study and all other previously mentioned references have only revealed the adsorption behaviour of clomazone in soils collected from rice fields in the USA or China.

Considering the agricultural importance and potential environmental impact of clomazone, the aim of this study was to determine its adsorption-desorption behaviour in two Serbian widespread types of agricultural soils described in the WRB 2015 soil classification¹³ as Regosol and Chernozem reference soil groups.

Since the selected soils differ in OC and clay contents as possible sorption fractions of soils, the objective of this study was also to determine whether and to what extent they participate in the retention and release of clomazone on/from this medium, as well as to determine to what extent different clomazone concentrations affect its adsorption–desorption behaviour.

EXPERIMENTAL

Reagents and materials

Clomazone (99.5 % purity) was purchased from Dr Ehrenstorfer, Germany. Its stock solution (1.5 g dm⁻³) was prepared in acetonitrile (J. T. Baker, Holland) and kept at -18 °C.

Calcium chloride was purchased from Merck (Germany), while highly purified deionized water (Purelab Option – R7, Elga, UK) was used for the preparation of 0.01 M CaCl₂ solution.

Working standard solutions of clomazone, in concentrations of 0.5, 1.5, 3, 6, 9, 12 and 15 mg dm⁻³, were prepared by diluting its stock solution with 0.01 M CaCl₂ solution.

Samples of two agricultural soils, namely Regosol and Chernozem, were collected in October 2017 from the top 25 cm soil layer in fields in which soybean had been grown in the same year. Regosol was sampled in Pirot (south–east Serbia), and Chernozem in Novi Banovci (Vojvodina region, Serbia). Clomazone had not been applied to the fields for at least 3 years previously. Soil samples were air-dried and ground to pass a 2-mm sieve. Soil texture, OC and CaCO₃ contents, as well as pH were determined by standard methods.¹⁴ General soil properties are shown in Table I.

TABLE I. General soil composition and properties

Soil	pH (H ₂ O)	Content of soil components, %					
		Coarse fragments	Sand	Silt	Clay	CaCO ₃	OC
Regosol	7.98	18.05	53.2	23.4	23.4	14.5	1.15
Chernozem	7.77	0	35.4	35.7	28.9	2.5	1.93

Regosol is a sandy clay loam containing 18 % coarse fragments, calcareous, moderately alkaline, with only 1.15 % OC, clay consisting primarily of kaolinite. Chernozem is a clay loam, calcareous, slightly alkaline, with 1.93 % OC, clay considered primarily of illite.

Adsorption–desorption studies

Adsorption and desorption isotherms of clomazone were obtained by the batch equilibration method.¹² Duplicate samples of 2 g of each soil and 10 cm³ of 0.01 M CaCl₂ solution with initial clomazone concentrations ranging from 0.5 to 15 mg dm⁻³ were mixed in 50 cm³ polypropylene centrifuge tubes. The tubes were shaken at 20±1 °C on a rotary shaker for 24 h, and then centrifuged at 3000 rpm for 5 min. A previous kinetics study had indicated that 24 h was adequate to attain the equilibrium redistribution of clomazone in the soil/water system. The supernatants were filtered through 0.22 µm membranes before further analysis. The amounts of clomazone adsorbed by the soils were calculated from the difference between the initials and supernatant concentrations in each sample.

Clomazone desorption studies were carried out for its initial concentrations of 1.5, 6 and 15 mg dm⁻³, immediately after the adsorption process has been completed, by replacing 5 cm³ of the obtained supernatants with 5 cm³ of clomazone-free 0.01 M CaCl₂ solution. The tubes were again equilibrated for 24 h on the rotary shaker, which was followed by centrifugation at

3000 rpm for 5 min, filtration of the obtained supernatants and chromatographic analyses. For all concentrations, three successive desorption steps were examined.

All sorption and desorption measurements were repeated 3 times.

Chromatographic analysis

Clomazone concentrations in the obtained adsorption–desorption supernatants were determined by the Shimadzu Prominence high performance liquid chromatography (HPLC) system, equipped with a pump model LC-20AD and diode array detector (DAD) model SPD-M20A. The following conditions were used: Eclipse XDB-C18 column (4.6 mm×150 mm, 3.5 μm), using acetonitrile and water (70:30 volume ratio) as the mobile phase at the flow rate of 1.0 cm³ min⁻¹, in an oven at 40 °C, 40 mm³ injection volume, and quantification at 214 nm. The detection and quantification limits were 0.0009 and 0.003 mg dm⁻³, respectively.

Data analysis

Adsorption-desorption coefficients (K_d / dm³ kg⁻¹) were calculated as the relation of the herbicide concentration in soil after the completion of adsorption-desorption experiments (c_s / mg kg⁻¹) and equilibrium concentration in the solution (c_e / mg dm⁻³) according to Eq. (1):

$$K_d = \frac{c_s}{c_e} \quad (1)$$

The K_{OC} coefficients were calculated by normalizing K_d coefficients with the OC content of the soils investigated according to the equation:

$$K_{OC} = \frac{100K_d}{\text{Content of OC}} \quad (2)$$

Clomazone adsorption and desorption isotherms were described by the linear form of the Freundlich equation:

$$\log c_s = \log K_f + \frac{1}{n} \log c_e \quad (3)$$

where c_s and c_e have the same meaning as before, and K_f and n_f are the Freundlich constants characterizing the adsorption-desorption capacity and intensity, respectively.

The desorption percentage (D / %) for all desorption steps were calculated by Eqs. (4) and (5):

$$D = 100 \frac{c_{s(\text{ads})} - c_{s(\text{des},1)}}{c_{s(\text{ads})}} \quad (4)$$

$$D = 100 \frac{c_{s(\text{des},i)} - c_{s(\text{des},i+1)}}{c_{s(\text{des},i)}} \quad (5)$$

where $c_{s(\text{ads})}$ and $c_{s(\text{des},i)}$ are clomazone concentrations in the soil after the completion of adsorption and desorption steps, respectively, and i is the number of desorption steps (i : 1–3).

For both soils and all concentrations tested, the hysteresis coefficients (H) were calculated for the adsorption–desorption isotherms according to the Eq. (6):

$$H = \frac{1/n_{\text{des}}}{1/n_{\text{ads}}} \quad (6)$$

where $1/n_{\text{ads}}$ and $1/n_{\text{des}}$ are the Freundlich slopes obtained for the adsorption and desorption isotherms, respectively.

RESULTS AND DISCUSSION

Adsorption studies

The adsorption isotherms of clomazone for the two agricultural soils are presented in Fig. 1, and the Freundlich parameters obtained by applying Eq. (3) to experimental data are presented in Table II.

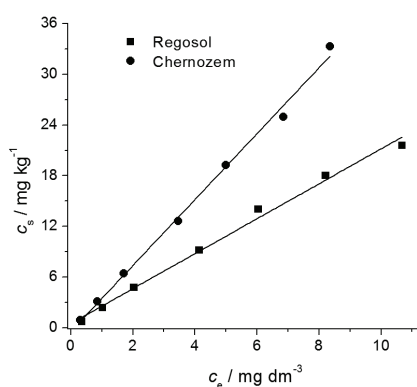


Fig. 1. Adsorption isotherms of clomazone in two Serbian agricultural soils.

TABLE II. Freundlich parameters and distribution coefficients ($K_{\text{d(ads)}}$ and $K_{\text{OC(ads)}}$) for clomazone sorption on two agricultural soils

Parameter	Soil	
	Regosol	Chernozem
$K_{\text{f(ads)}} / \text{mg}^{(n-1)/n} \text{ dm}^{3/n} \text{ kg}^{-1}$	2.21 (± 0.01) ^a	3.43 (± 0.01)
$1/n_{\text{ads}}$	1.00 (± 0.02)	1.07 (± 0.02)
r^2	0.997	0.998
$K_{\text{d(ads)}} / \text{dm}^3 \text{ kg}^{-1}$	2.14 (± 0.05)	3.83 (± 0.06)
r^2	0.991	0.995
$K_{\text{OC(ads)}} / \text{dm}^3 \text{ kg}^{-1}$	1.86	1.99

^aValue in parentheses is the 95 % confidence interval

The data indicate that clomazone sorption behaviour could be well described by Freundlich model with correlation coefficients (r^2) of 0.997 and 0.998 for Regosol and Chernozem, respectively. The obtained isotherms are C-type according to Giles classification,¹⁵ which is also indicated by $1/n_{\text{ads}}$ parameters, whose values are close to 1 for both soils under study (Table II). The C-type of the isotherms is characterised by a slope that is independent of the herbicide initial concentration in the solution, *i.e.*, the partition of clomazone molecules between the soil and the liquid phase is constant. However, over the entire initial clomazone concentration range studied (0.5–15 mg dm^{-3}), adsorption in Chernozem was higher than it was in Regosol (Fig. 1), which was also confirmed by the

obtained Freundlich parameters $K_{f(ads)}$ (2.21 and 3.43 for Regosol and Chernozem, respectively, Table II). These relations of $K_{f(ads)}$ values correspond well with the obtained relation for $K_{d(ads)}$ values (Table II), which is not surprising considering the linear C-type of adsorption isotherms obtained for both soils. Considering the composition of the tested soils, and the well-known fact that the pesticide sorption in soil occurs either through the mineral or organic component, or both, the obtained results are not surprising. Generally, in soils that have higher levels of organic matter (>5 %), pesticide adsorption may be assumed to depend on organic matter content, while the nature of organic matter has little influence on adsorption processes.^{16–18} Conversely, in soils with low organic matter contents, the adsorption of pesticides often depends on active inorganic fraction components, which is predominantly the clay fraction. Therefore, higher OC and clay contents in Chernozem than in Regosol clearly indicate that higher adsorption could be expected on Chernozem soil particles. However, if we assume that OC is the main soil parameter responsible for clomazone sorption, the values of $K_{OC(ads)}$ coefficients calculated for both soils should be very close to each other. Indeed, comparing the $K_{OC(ads)}$ constants presented in Table II, it becomes evident that the sorption of clomazone molecules in both tested soils takes place primarily at the active sorption centres of organic matter. However, slight differences in the $K_{OC(ads)}$ constants obtained for Regosol and Chernozem indicate that clay takes a certain part in sorption, too. At first, higher clay content in Chernozem (28.9 %), compared to 23.4 % in Regosol (Table I), was consistent with a somewhat higher $K_{OC(ads)}$ value obtained for Chernozem. Additionally, the differences in soil mineral composition, and the type of phyllosilicates structure in those soil minerals, indicate their different participation in clomazone sorption. Generally, it should be assumed that the layered structure of the 2:1 phyllosilicates type present in illite (Chernozem) has a greater internal and external porosity, and a larger specific surface area, so that the interlayer space of this mineral provides a greater possibility for clomazone molecules movement, in contrast to kaolinite as the 1:1 type of phyllosilicates, which is dominant in Regosol.

A comparison of our results with literature data revealed considerable consistency. Unfortunately, the reference data mainly relate to soils in rice fields, and originating either from the USA or China,^{7–10,19} so that some discrepancies between our present and published data are not surprising. Thus, for example, the L-type of clomazone sorption isotherm has been reported in most studies,^{7,9,10,19} while in some of them the obtained K_d values were higher than our findings.^{7,9} However, all studies agreed that organic matter is the soil fraction that predominantly participates in clomazone adsorption.

Desorption studies

Desorption isotherms of clomazone for the two agricultural soils were presented in a log–log scale (Fig. 2), while the obtained Freundlich desorption parameters were listed in Tables III and IV.

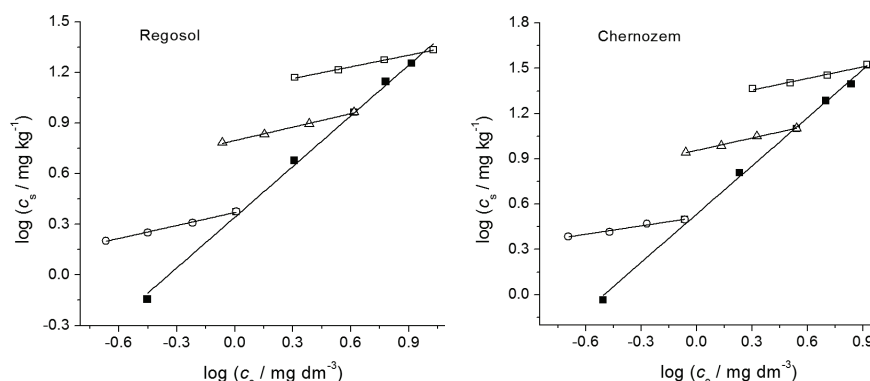


Fig. 2. Adsorption–desorption isotherms of clomazone in two Serbian agricultural soils: ■) adsorption; ○) desorption for 1.5 mg dm⁻³; △) desorption for 6 mg dm⁻³; □) desorption for 15 mg dm⁻³.

TABLE III. Freundlich parameters, hysteresis coefficients (H) and desorption percentages (D) for clomazone desorption in Regosol

Parameter	Initial concentration, mg dm ⁻³		
	1.5	6	15
$K_{f(\text{des})} / \text{mg}^{(n-1)/n} \text{dm}^{3/n} \text{kg}^{-1}$	2.34 (± 0.004) ^a	6.26 (± 0.004)	12.46 (± 0.01)
$1/n_{\text{des}}$	0.26 (± 0.01)	0.27 (± 0.01)	0.23 (± 0.01)
r^2	0.995	0.995	0.997
H	0.277	0.260	0.241
D (I) ^b / %	17.60	14.70	14.52
D (II) / %	12.44	13.39	12.59
D (III) / %	10.70	10.84	9.89
Total D (I–III) / %	35.58	34.13	32.67

^aValue in parentheses is the 95 % confidence interval; ^bnumber of desorption cycles

Eq. (3) evidently well described the experimental desorption data for all three initial concentration levels of clomazone, with r^2 ranging from 0.995 to 0.997, and 0.970 to 0.994 for Regosol and Chernozem, respectively.

The obtained $1/n_{\text{ads}}$ (Table II) values were much higher than $1/n_{\text{des}}$ (Tables III and IV), regardless both of the initial clomazone concentration and soil types studied, which indicates that a significant amount of clomazone adsorbed is not easily desorbed and that desorption cannot be predicted on the basis of adsorption isotherms. The finding that the sorption of clomazone to soil is not a reversible process was reported by Gunasekara et al.⁷ for four American soils, too.

TABLE IV. Freundlich parameters, hysteresis coefficients (H) and desorption percentages (D) for clomazone desorption in Chernozem

Parameter	Initial concentration, mg dm ⁻³		
	1.5	6	15
$K_{f(\text{des})} / \text{mg}^{(n-1)/n} \text{ dm}^{3/n} \text{ kg}^{-1}$	3.24 (± 0.01) ^a	9.03 (± 0.004)	18.95 (± 0.01)
$1/n_{\text{des}}$	0.19 (± 0.02)	0.27 (± 0.01)	0.26 (± 0.02)
r^2	0.970	0.994	0.975
H	0.258	0.256	0.227
D (I) ^b / %	18.05	12.42	13.05
D (II) / %	11.69	13.44	11.35
D (III) / %	6.40	9.80	8.04
Total D (I-III) / %	32.26	31.62	29.12

^aValue in parentheses is the 95 % confidence interval; ^bnumber of desorption cycles

The magnitude of the hysteresis effect was evaluated (Eq. (6)), and the values spanned a range of 0.241–0.277 and 0.227–0.258 for Regosol and Chernozem, respectively (Tables III and IV). These values were somewhat higher than those reported for clomazone adsorption–desorption behaviour in four American soils,⁷ but considering their different origin and history of their uses, as well as different management practices, the detected inconsistency was expectable. However, for each of the initial clomazone concentrations in the study, the H value obtained for Regosol was higher than one obtained for Chernozem. The foregoing results indicate that Chernozem, with its higher adsorption capacity than Regosol, showed fewer tendencies to desorb clomazone. Lower clomazone desorption from Chernozem obtained for all initial pesticide concentrations could also be related to the higher $K_{f(\text{des})}$ values obtained for Chernozem (Table IV), compared to Regosol (Table III).

Concerning the adsorption process, it seems that the content of organic matter in the soil, as the dominant promoter of clomazone sorption to the soil, is negatively correlated to the desorbed clomazone amount in the desorption process. Sorption hysteresis in soils has been reported for a large number of pesticides,^{20–23} and several possible causes of this phenomenon have been proposed, such as chemical or biological transformation, nonequilibrium conditions, and high-energy bonding. However, during desorption experiments under this presented study, there was no evidence of clomazone degradation in the obtained chromatograms. Furthermore, a preliminary kinetics study had indicated that 24 h of equilibration was adequate to attain the equilibrium redistribution of clomazone in the soil/water system, so that a nonequilibrium hypothesis was excluded too. A high-energy bonding hypothesis obviously may be the only one that could explain the obtained data.

As Tables III and IV show, the irreversibility of the adsorption process in both soils increases (H coefficient decreases) with growing initial clomazone concentrations, suggesting that desorption is easier at lower concentration levels

of the herbicide. This finding could indicate that high-energy sorption sites in both soils are filled only at higher clomazone concentrations, wherein desorption from that type of centres is much harder when compared to low-energy binding centres. This phenomenon is particularly visible in Chernozem, which is not surprising considering the higher contents of both OC and clay in that soil, compared to Regosol.

The desorption percentage ($D / \%$) for all three successive desorption steps and the total percentage of desorbed herbicide during all desorption cycles were calculated by Eqs. (4) and (5), and presented in Tables III and IV. Regardless of the initial concentration of clomazone in the adsorption-desorption study, the amount desorbed evidently decreased with the subsequent desorption cycles. These results seem to confirm the hypothesis of bound residues, specifying that some clomazone residues remained strongly bound to soil after adsorption. Only the fraction of herbicide that was weakly bound to soil could be easily released during the desorption process. Considering the total amount of clomazone desorbed during all three consecutive desorption cycles in the study (D (I-III)), it seems that this fraction is highly dependent on the initial clomazone concentration. Decrease in D (I-III) values with the increasing initial clomazone concentration in the soil-solution system indicate that high-energy binding sites are limited, and the progressive saturation of those sites occurs as the solute concentration increases. The mentioned strong adsorption resulted in the hysteresis effect. In general, for each concentration examined in the desorption study, the D (I-III) values obtained for Regosol (32.67–35.58 %) were higher than for Chernozem (29.12–32.26 %). These results were expectable, considering both the higher adsorption capacity (Table II) and the lower desorption tendency of Chernozem (Table IV) compared to Regosol (Table III).

CONCLUSION

The study conducted on two typical agricultural soils in Serbia broadened our knowledge of the sorption-desorption behaviour of clomazone which considering its intensive use, relatively high solubility in water, moderate mobility and persistence in soil, as well as the possibility of causing harmful effects on cultivated plants after crop rotation, could be classified as a possible environmental contaminant of agricultural relevance. Clomazone was found to sorb more to organic matter than to the mineral fraction of soil. Although Chernozem has been shown to have a higher adsorption capacity, at the same time it showed less tendency to desorb herbicide molecules than Regosol. The higher hysteresis effect (lower H values) observed in Chernozem could be interpreted as supporting the high-energy bonding hypothesis, which implies more high-energy sorption sites in that soil than in Regosol. Although it has been shown that clomazone is potentially a greater contaminant of deeper soil layers, and consequently of

groundwater, when it is found in soil with lower organic matter and clay contents, further studies are necessary to clarify the nature of the binding forces acting on clomazone sorption in soils.

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ИЗВОД

АДСОРПЦИОНО–ДЕСОРПЦИОНО ПОНАШАЊЕ КЛОМАЗОНА У ПОЉОПРИВРЕДНИМ ЗЕМЉИШТИМА ТИПА РЕГОСОЛ И ЧЕРНОЗЕМ

РАДА Д. БУРОВИЋ-ПЕЈЧЕВ¹, СВЈЕТЛАНА Б. РАДМАНОВИЋ², ЗОРИЦА П. ТОМИЋ², ЛАЗАР М. КАЛУЂЕРОВИЋ², ВОЈИСЛАВА П. БУРСИЋ³ И ЉИЉАНА Р. ШАНТРИЋ¹

¹Институт за истраживање и заштити животне средине, Банатска 31б, 11080 Београд, ²Пољопривредни факултет, Универзитет у Београду, Немањина 6, 11080 Београд и ³Пољопривредни факултет, Универзитет у Новом Саду, Три Д. Обрадовића 8, 21000 Нови Сад

Испитивање адсорпционог и десорпционог понашања пестицида у земљишту је веома важно за разумевање и предвиђање њиховог кретања и даље судбине у животној средини. Узимајући у обзир значај кломазона са становишта пољопривреде и потенцијалне штетне ефекте које може имати на животну средину, адсорпционо-десорпционо понашање овог хербицида је проучавано у Регосолу и Чернозему, као два широко заступљена типа пољопривредног земљишта у Србији. Оба процеса се добро могу описати Фројдлинговом једначином, а резултати указују да се кломазон много више везује за органску материју него за минералну фракцију земљишта. Чернозем који карактерише већи садржај органске материје и глине, везује више али и десорбује мање овог хербицида у односу на Регосол. Већи десорпциони хистерезис добијен за Чернозем се може објаснити већим бројем високо-енергетских сорпционих центара присутних у овом земљишту у односу на Регосол. Ефекат хистерезиса расте за оба земљишта са повећањем концентрације кломазона у систему земљиште-земљишни раствор, при чему се проценат десорбоване количине пестицида у сваком наредном десорпционом циклусу смањује. Резултати адсорпционо–десорпционих експеримената указују на то да понашање и судбина кломазона у животној средини у великој мери зависе од састава земљишта, при чему је за очекивати да је вероватноћа контаминације дубљих слојева земљишта па и подземних вода значајно мања када се овај хербицид нађе у земљиштима богатим хумусом.

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