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Dissipation dynamic of nicosulfuron in different types of agricultural soils

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Abstract: This work aimed to evaluate the influence of soil characteristics and the applied amount of nicosulfuron on the degradation rate in soil. Soil samples were collected at three localities in Bosnia and Herzegovina – Manjača, Kosjerovo and Tunjice. The experiment was carried out under controlled laboratory conditions. Plant protection product based on nicosulfuron (40 g a.s./L, OD) was applied in concentrations of 0.075, 0.15, and 0.30 mg a.s./kg of soil. Nicosulfuron residues were analysed by the modified QuEChERS method, followed by LC-MS/MS. Soils are classified as silty loams, with variations in mechanical composition and chemical properties. In slightly alkaline soil, the half-life (DT₅₀) of nicosulfuron has increased (43.31 days) compared with DT₅₀ (9.43–16.13 days) in acidic soils. The results indicate that soil characteristics and applied concentration significantly influence nicosulfuron persistence. Hence, it can be considered that nicosulfuron, applied to silty loam soils of Bosnia and Herzegovina, poses a low risk to subsequent crops and the environment.

Keywords: sulfonylurea herbicide; agrochemical; environmental condition; weed control

Intensive and/or inappropriate application of agrochemicals in contemporary agriculture can lead to side effects on the soil, surface, and groundwater, but also on subsequent crops. Therefore, knowledge of soil characteristics and environmental conditions for the application of agrochemicals is required. The use of the herbicide nicosulfuron in the last decades has raised significant concerns about the deteriorated environment and its toxic effect on the living world and biodiversity within the soil. Nicosulfuron (((2-[(4,6-dimethoxypyrimidin-2-yl-carbamoyl)sulfamoyl]-N,N-dimethylnicotinamide)) is an acetolactate synthase (ALS) inhibitor, commonly

employed in maize cultivation to manage a broad spectrum of weed species effectively. The reason for nicosulfuron persistence is chlorimuron-ethyl, which is included in the composition of herbicides from the sulfonylurea group. Thus, nicosulfuron and its residues have the potential to persist in the soil for an extended period, leading to contamination of both crops and the surrounding environment (Soltani et al. 2006).

Depending on the pesticide characteristics and environment, degradation can be relatively fast to slow (Riyaz et al. 2021). However, some pesticides are persistent, meaning that they have a limited ability

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to degrade rapidly. After reaching the soil, pesticides undergo a sorption process, especially in clay soils and soils with a high organic matter content. Their mobility is minimal in such soils, so their runoff into the deeper layers of soil and groundwater generally does not occur. The behavior and fate of pesticides in the environment are influenced by different factors, including soil microorganisms, chemical reactions and sunlight. Due to the complex nature of the environment, numerous pesticides have the potential to become mobile following their application. This mobility can occur through natural processes like runoff, leaching, and erosion (Arias-Estevez et al. 2008). While the ideal scenario for pesticides is to effectively target and reduce specific organisms without causing harm to the environment, it is not uncommon for their residues to have adverse effects on non-target organisms and result in environmental damage, which depends on the nature of the pesticide, the type of formulation, the applied concentration, application frequency, etc.

Pesticide residues are subject to a large number of reactions (hydrolysis, photodegradation, oxidation and reduction), which contribute to their decomposition. Many studies indicate that pesticide residues disrupt plant-microbial interactions in the soil, as well as the functioning of soil enzymes as an indicator of the general state of soil health (Hussain et al. 2009, Ataikiru et al. 2019). Also, the residues of some pesticides are very challenging to decompose in the soil, which can pose risks of poisoning and illness of the human population (Nicolopoulou-Stamati et al. 2016), as well as domestic and wild animals, beneficial insects and pollinators (Sanchez-Bayo and Goka 2016).

Residues of sulfonylurea herbicides are detected in different samples (Wu et al. 2010), most often in soil (Ye et al. 2006), surface water (Polati et al. 2006), and some crops (Sui et al. 2006). Various methods are applied to determine the residues of sulfonylurea herbicides: liquid-liquid extraction, supercritical fluid extraction, solid phase extraction, dispersive liquid-liquid microextraction, microwave extraction, etc. (Grahovac et al. 2017).

The most commonly used instrumental technique for the analysis of polar and thermolabile herbicides such as sulfonylureas is high-performance liquid chromatography (HPLC) with UV detection or diode array detector (DAD). Some authors suggest the use of the enzyme immuno method, gas chromatography, and the HPLC method with tandem mass spectrometry, i.e. HPLC-MS/MS (Grahovac et al. 2017).

Previous research conducted in Bosnia and Herzegovina has shown that in sandy soils with organic matter less than 2% and slightly to moderately alkaline reactions, the adsorption of nicosulfuron is very low, and only a small amount of nicosulfuron will be adsorbed in soil particles (Sunulahpašić et al. 2020).

This research aimed to investigate the dissipation dynamic of nicosulfuron in different agricultural soils rich in organic matter in Bosnia and Herzegovina. The obtained results are used to calculate nicosulfuron half-life (DT_{50}) and evaluate the influence of soil characteristics and applied dosage on the persistence of nicosulfuron.

MATERIAL AND METHODS

Soil samples. Untreated soil samples were collected during the spring of 2020 at three localities with main agricultural production in Bosnia and Herzegovina – Manjača (MAN), Kosjerovo (KOS), and Tunjice (TUN). After sampling, the soil was air-dried, milled, and sieved (2 mm). The analysis was performed in four time intervals, three concentrations, and three replications. Plant protection product (PPP) based on nicosulfuron (40 g a.s./L, OD) was applied in concentrations of 0.075, 0.15, and 0.30 mg a.s./kg of soil, i.e. the amount of 70 mL was applied to 500 g of soil, including the control without herbicide. Samples were stored at room temperature for 0 (zero), 10, 30, and 40 days. Before the analysis, the soil was mixed, and approximately 100 g was taken and milled. For the extraction, a 10 g soil final sample was formed for further analysis.

Extraction of nicosulfuron from soil samples. A modified QuEChERS method was used to extract nicosulfuron. Namely, an amount of 10 g of the soil sample was transferred into a 50 mL polypropylene cuvette, and 3 mL of deionised water and 10 mL of acidified acetonitrile (1% CH_3COOH) were added, shaken for 1 min, and vortex for 1 min. After that, the extraction buffer mixture (QuEChERS Extract Pouches, EN Method Cat. No. 5982/5650 – 4 g $MgSO_4$, 1 g NaCl, 1 g trisodium citrate dihydrate, 0.5 g disodium hydrogen citrate sesquihydrate) was added and again shaken for 1 min and vortexed for 1 min and placed into the ultrasonic bath for 10 min, followed by centrifugation (4 000 rpm for 5 min). Afterwards, the supernatant was filtered (0.45 μm) and transferred into vials for LC analysis.

Determination of nicosulfuron residues in soil samples. Analysis of nicosulfuron residues in the soil

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Table 1. Mechanical composition, texture and chemical properties of soils

Locality	Mechanical composition			Soil texture	Soil type (FAO)	Chemical properties			
	sand	silt	clay			CEC (mmol ₊ /100 g soil)	pH _{H₂O}	pH _{KCl}	OC (%)
	(%)								
MAN	18.60	67.10	14.30	silty loam	Dystric Fluvisol	21.2	5.14	4.24	4.24
KOS	17.80	59.50	22.70	silty loam	Calcaric Fluvisol	28.2	7.70	7.00	3.49
TUN	22.90	62.40	14.70	silty loam	Stagnic Luvisol	22.0	6.50	5.50	3.55

CEC – cation exchange capacity; OC – organic carbon; MAN – Manjača; KOS – Kosjerevo; TUN – Tunjice

was performed by LC-MS/MS (Agilent Technologies 1260 Infinity II with Triple Quadrupole sistemom 6420) chromatography and InfinityLab Poroshell 120 EC-C18 (2.1 × 150 mm, 4 microns) column. The mobile phase is composed of water, formic acid, ammonium formate (A), and methanol, formic acid, and ammonium formate (B).

Statistical analysis. An analysis of variance (two-way ANOVA), followed by the Tukey post hoc test, was used for the statistical analysis in this study (Statistica 14.0.0.15 version, Tibco Software Inc., Palo Alto, USA, 2021).

RESULTS AND DISCUSSION

Mechanical composition and chemical properties of soils. Soils used in this study are classified as silty loams. The soil is rich in organic carbon. However, the obtained results indicate variations in the mechanical composition and chemical properties. The pH value was in the range of 5.14 to 7.70 (in H₂O) and 4.24 to 7.0 (in KCl), acidic to weakly alkaline and acidic to neutral reactions, respectively (Table 1).

Dissipation dynamic of nicosulfuron in different soils. The dissipation dynamic of nicosulfuron in soil was evaluated through its residues using a validated method. Validation of the method for the analysis of nicosulfuron in soil followed standards for this type of analysis (SANTE/2020/12830, Rev. 1 24 February 2021); the linearity of detector response, repeatability, the accuracy of the method, the limit of detection and quantification are within the limit values (Table 2).

The results of the analysis of nicosulfuron residues in soil samples are shown in Figure 1. The initial amounts of nicosulfuron in the soils from the locality Manjača and Tunjice, which are similar due to their mechanical composition and chemical properties, were at the level of the applied concentrations. During the next 40 days, in these acidic soils, nicosulfuron dissipated gradually, with the final amount below 0.05 mg/kg for all applied concentrations. With increased pH, degradation was slower; thus, in neutral to slightly alkaline soil from the locality Kosjerevo, the initial amount of nicosulfuron was below the applied concentration, followed by slow dissipation due to R².

The dissipation curve of nicosulfuron in the soil from the locality of Manjača, Kosjerevo, and Tunjice is represented by the first-order kinetic model and the decomposition constant (Figure 1).

A two-way ANOVA (Table 3) showed a significant effect of soil type and applied dosage on the dissipation dynamic of nicosulfuron in soil. To determine which soil type there is a statistically significant difference, a post-hoc analysis was conducted using the Tukey HSD (honestly significant difference) test (Table 4). Based on the obtained results, it can be observed that there is a statistically significant difference ($P < 0.05$) between all three soil types at all applied concentrations of nicosulfuron.

The obtained results expressed the variation of the nicosulfuron content in all soils, depending on the main factors, applied doses, and time (Table 5). The half-life of nicosulfuron in the soil was calculated using $t_{1/2} = \ln 2/k$, since in this study the dissipation of nicosulfuron followed the first-order kinetics equa-

Table 2. Validation parameters

Herbicide	Linearity (R ²)	Repeatability (%)	Accuracy	Limit of detection	Limit of quantification (mg/kg)
Nicosulfuron	0.9982	2.34	91.1–102.7	0.01	0.03

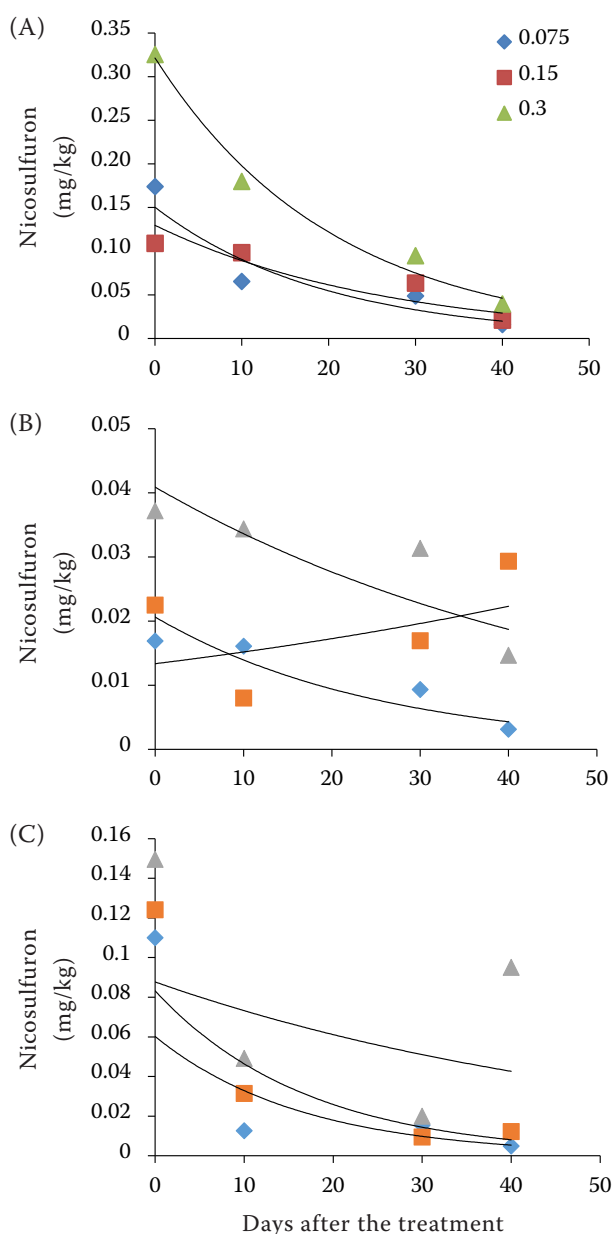


Figure 1. Nicosulfuron concentration in samples of soil from (A) Manjača locality; (B) Kosjerevo locality and (C) Tunjice locality

tion $C_t = C_0 e^{kt}$, where C_t represents the concentration of pesticide residues during time t , C_0 is the initial concentration after application and k represents the degradation constant (days).

The highest degradation constant was in the soil at the Tunjice locality, while the lowest was in the soil from the locality Kosjerovo. The half-life (DT_{50}) was the highest in the soil from the Kosjerevo locality ($DT_{50} = 43.31$ days), then in the soil from the Manjača locality (16.13 days), while the shortest half-life was in the soil sample from the locality Tunjice

Table 3. Results of two-way ANOVA

Source	75 mg/kg					150 mg/kg					300 mg/kg				
	Type III SS	df	MS	F	sig.	Type III SS	df	MS	F	sig.	Type III SS	df	MS	F	sig.
Corrected model	0.086	11	0.008	178.778	0.000	0.060	11	0.005	114.625	0.000	0.274	11	0.025	191.194	0.000
Intercept	0.068	1	0.068	1 560.472	0.000	0.075	1	0.075	1 584.718	0.000	0.287	1	0.287	2 205.440	0.000
Day	0.041	3	0.014	309.626	0.000	0.023	3	0.008	159.794	0.000	0.089	3	0.030	226.773	0.000
Soil	0.021	2	0.010	236.659	0.000	0.018	2	0.009	187.658	0.000	0.104	2	0.052	401.595	0.000
Day × soil	0.025	6	0.004	94.060	0.000	0.019	6	0.003	67.696	0.000	0.081	6	0.013	103.271	0.000
Error	0.001	24	4.379E-005			0.001	24	4.737E-005			0.003	24	0.000		
Total	0.156	36				0.136	36				0.564	36			
Corrected total	0.087	35				0.061	35				0.277	35			

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Table 4. Tukey *HSD* (honestly significant difference) test results of soil type and nicosulfuron concentration

(I) soil	(J) soil	75 mg/kg			150 mg/kg			300 mg/kg		
		MD (I–J)	SE	sig.	MD (I–J)	SE	sig.	MD (I–J)	SE	sig.
MAN	KOS	0.0168*		0.000	0.0251*		0.000	0.0491*		0.000
	TUN	–0.0404*		0.000	–0.0293*		0.000	–0.0816*		0.000
KOS	MAN	–0.0168*	0.00270	0.000	–0.0251*	0.00281	0.000	–0.0491*	0.00466	0.000
	TUN	–0.0572*		0.000	–0.0544*		0.000	–0.1306*		0.000
TUN	MAN	0.0404*		0.000	0.0293*		0.000	0.0816*		0.000
	KOS	0.0572*		0.000	0.0544*		0.000	0.1306*		0.000

MD – mean difference; SE – standard error; MAN – Manjača; KOS – Kosjerovo; TUN – Tunjice

(9.43 days). In terms of persistence, pesticides could be classified as non-persistent (half-life < 30 days), moderately persistent (half-life between 30 and 100 days), and persistent (half-life > 100 days) (Gavrilescu 2005). Thus, nicosulfuron could be classified as non-persistent in weakly acid silty loam soil, while in alkaline soil, nicosulfuron is moderately persistent due to slower degradation in this soil type.

Besides, values of Pearson's coefficient (*r*) show the correlation between soils' mechanical composition, texture and chemical properties and the dissipation dynamic of nicosulfuron (Table 6). Increased clay content, pH value, and cation exchange capacity lead to an increase in the half-life of nicosulfuron in soil.

The degradation potential of pesticides is strongly affected by environmental conditions and the depth of the soil profile (Dechesne et al. 2014). The horizontal distribution of pesticides depends on the type of pesticide, sampling design, and site characteristics. In surface soil layers, pesticide degradation potential is characterised by a low variation coefficient, which can be associated with low (Vinther et al. 2008) or intense pesticide degradation (Fredslund et al. 2008). Variations of the degradation constant in the presented research are not high. According to Mulla and McBratney (1991), the variability of degradation parameters is less pronounced concerning the physical characteristics of the soil, which was confirmed in this study.

Table 5. Half-life (DT_{50}) and degradation parameters of nicosulfuron in the three soil type

The initial amount of nicosulfuron (mg/kg)	Regression equation	Degradation constant	R^2	DT_{50} (day)
MAN				
0.30	$y = 0.325e^{-0.049x}$	0.049	0.9638	14.14
0.15	$y = 0.109e^{-0.032x}$	0.032	0.7912	21.65
0.075	$y = 0.174e^{-0.055x}$	0.055	0.8755	12.60
\bar{x} average value				16.13
KOS				
0.30	$y = 0.037e^{-0.016x}$	0.016	0.6611	43.31
0.15	–	–	–	–
0.075	$y = 0.016e^{-0.016x}$	0.016	0.7987	43.31
\bar{x} average value				43.31
TUN				
0.30	$y = 0.149e^{-0.071x}$	0.071	0.9139	9.76
0.15	$y = 0.124e^{-0.071x}$	0.071	0.7767	9.76
0.075	$y = 0.11e^{-0.079x}$	0.079	0.6024	8.77
\bar{x} average value				9.43

MAN – Manjača; KOS – Kosjerovo; TUN – Tunjice

Table 6. Correlation matrix (Pearson correlation coefficient, r) for half-life (DT_{50}) of nicosulfuron and mechanical composition, texture and chemical properties of soils

	pH _{H₂O}	pH _{KCl}	CEC (mmol ₊ /100 g soil)	Sand 2.0–0.06 mm	Silt 0.06–0.002 mm	Clay < 0.002 mm	Humus
				(%)			
r	0.733	0.789	0.957	–0.756	–0.662	0.974	–0.406
P	0.476	0.421	0.186	0.454	0.539	0.146	0.734

CEC – cation exchange capacity; r – Pearson correlation coefficient; P – P -value

Numerous research studies show the simultaneous influence of different factors on degradation rather than deal with the precise identification of these factors (Dechesne et al. 2014). Some physicochemical and biological factors are often cross-correlated, making it impossible to identify the real factors, so they are not determined during research. This is especially present during the analysis of the degradation of different compounds, where the measurable parameters of the soil characteristics vary with the depth of the soil profile, thus creating a "false" correlation with the degradation process. Moreover, other methodological aspects can be limiting in terms of identifying the "true" factors of degradation potential (Gonod et al. 2006, Vos et al. 2013).

Although the physicochemical parameters of soil have a different influence on pesticide degradation, a regular soil characteristic in many studies is the pH value. It is considered that the pH is a good indicator of the degradation of pesticides, such as bentazon and phenyl ureas herbicides (Rodriguez-Cruz et al. 2006), and one of the key factors in soil microbial activity (Lauber et al. 2009), with a significant impact on the fate of pesticides (Bending et al. 2001, Franco et al. 2009). In the warm, moist soil with a pH of 5.7, half-lives of nicosulfuron and rimsulfuron were less than 6 days under field conditions, while in the laboratory using the same soil, half-life for both sulfonylurea herbicides were < 3 days (Poppell et al. 2002). In the study conducted by Hultgren et al. (2002), DT_{50} of sulfonylurea herbicide prosulfuron positively correlated with soil pH, varying from 6.5 days at pH 5.4 to 122.9 days at pH 7.9. After the application at the recommended dosage, as well as at a higher dosage level (one and half the recommended dosage), the half-life of nicosulfuron in soil under corn production was 15–17 days (Wu et al. 2010).

Research conducted in this study shows that soil characteristics and applied concentration significantly influence nicosulfuron persistence. In slightly alkaline

soil, the half-life of nicosulfuron has increased, with DT_{50} of 43.31 days, compared with DT_{50} (9.43–16.13 days) in acidic soils. Hence, it can be considered that nicosulfuron, applied to silty loam soils of Bosnia and Herzegovina, poses a low risk to subsequent crops and the environment.

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