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Residue and dissipation dynamic of spinetoram insecticide in pear fruits

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Abstract: *Cacopsylla pyri* (Linnaeus, 1758) is the most significant and widespread pear pest. It attacks the pear only, causing direct and indirect damages. This study was conducted in order to evaluate the efficacy of insecticide spinetoram in the control of *C. pyri*. The experiment was set up according to EPPO methods, at the locality Kula (Republic of Serbia) in a pear orchard (Williams variety). Spinetoram (250 g/kg a.i., WG) was foliar applied in the amount of 0.3 kg/ha, during overlapping pest generations, when pear was on BBCH 75 scale. The efficacy was evaluated through the number of larvae aged L_1–L_3 and L_4–L_5. Spinetoram has shown satisfying efficacy, especially in the control of L_1–L_3 larvae (81.6%). In a dissipation study, spinetoram residues in pear fruits were determined using QuEChERS method followed by HPLC-DAD. The method was validated to fulfill SANTE/12682/2019 criteria. Three days after the application, spinetoram residues in pear fruits were below the MRL (0.2 mg/kg). The results have shown that spinetoram dissipation pattern followed the first-order kinetics (R^2 = 0.979) with a half-life of 2.17 days, in pear fruits. This study suggests that spinetoram could be safely used in pear, and it could take an important place in insecticide resistance management.

Keywords: spinosyne; pear psylla; DT_{50}; pre-harvest interval; PHI

Spinetoram is a novel semi-syntetic insecticide, a multicomponent tetracyclic macrolide, from the chemical group of spinosyns. It is a chemically modified product of the soil actinomycete *Saccharopolyspora spinosa* (Mertz & Yao 1990), contained of two closely related components, XDE-175-J and XDE-175-L (Figure 1) (Yamada 2008), present at a ratio of approximately 3 : 1 (Malhat & Abdallah 2019). Although not fully studied, it is known that spinosyns act as allosteric modulators of nicotine acetylcholine receptors (nAChR) (IRAC 2020). However, they also act on γ-aminobutyric acid (GABA) receptors, which is why their mechanism of action is unique (Orr et al. 2009). Compared to the other insecticides, spinosyns show higher selectivity, sparing beneficial insects, mammals, birds and aquatic organisms (Yamada 2008; Malhat 2013), and at the same time, the efficacy in the control of target organisms, with relatively long persistence (Kkadan et al. 2020).

Spinetoram has shown to be effective in the control of Lepidoptera, Thysanoptera and Coleoptera (Andrić et al. 2019), including *Cacopsylla pyri* (Linnaeus, 1758) (Boselli & Sccannavini 2014). *C. pyri* (pear psylla) is the most significant pear pest,

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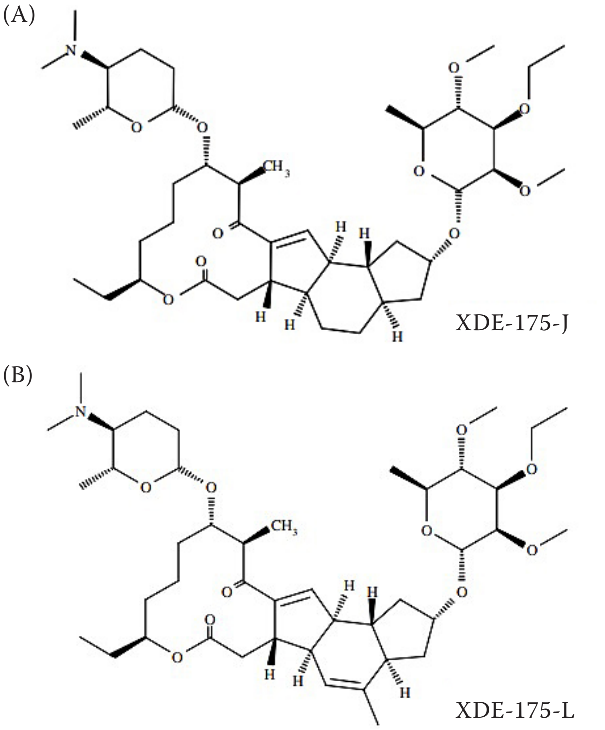

Figure 1. Structural formula of spinetoram (A) J (XDE-175-J) and (B) spinetoram L (XDE-175-L) (Yamada 2008)

causes damages only to pear (Civolani 2012). Due to the narrow trophic and reproductive species orientation, it accompanies the pear at the temperate-continental Euro-Asian mainland. The pear psylla has several generations per year (4–6), and they overlap in a certain part of the vegetation. The damages are caused by larvae and adults, and as a result of their activity during a stronger attack, leaves curl, the shoots lag in growth and often dry out. In addition to these, there are also significant indirect damages psylla causes as a vector of some dangerous pear pathogens, such as pear decline phytoplasma and abundant secretion of "honeydew" as a suitable substrate for the development of the soot fungus (Civolani 2012). The most commonly applied insecticides for the control of the pear psylla were chlorpyrifos, chlorantraniliprole, fenitrothion, endosulfan, tetradifon (Bicchi et al. 2003), thiacloprid, phosalone, flufenoxuron, abamectin, α-cypermethrin, diflubenzuron (Kocourek & Stará 2006). Nowadays, some of the listed active substances are still in use, but with an increased risk of residue accumulation, phytotoxicity, the resistance of target species and persistence. To avoid or minimize the consequences, more and more attention is paid to developing new, more eco-friendly active substances, such as spinetoram.

However, it is still necessary to evaluate its effect on treated plants and residues in agricultural products in different agroecological conditions. Besides active substances, the behavior of pesticides after the application can be affected by plant species, climate, photodegradation, the chemical structure of pesticides, and its formulation (Garau et al. 2002). In order to understand these processes, it is necessary to conduct the dissipation dynamic studies in different agroecological regions.

Due to the lack of data on the use of spinetoram, as well as its residue analysis in pear fruits, the aim of this study was the determination of the efficacy of spinetoram in control of *C. pyri* and the presence of its residues in this fruit, in order to evaluate dissipation dynamic and calculate the half-life and pre-harvest interval (PHI) of spinetoram. Based on the available data, this study provided the first results of the dissipation dynamics of spinetoram in pear fruits.

MATERIAL AND METHODS

Chemicals. Plant protection product (PPP) based on spinetoram (250 g/kg a.i., WG) was used. Analytical standard of spinetoram 98% was purchased from Dr. Ehrenstorfer, Germany. Acetonitrile (HPLC purity) and NH_4COOH were from J.T.Baker (Germany). For the extraction and clean-up, QuEChеRS Extraction Kit (Cat. No. 5982-5650) and QuEChеRS Dispersive Kit (Cat. No. 5982-5356) (Agilent Technologies Inc., USA) were used. Ultra-pure water was obtained by the TKI system.

Field trial. The experiment was conducted at the locality Kula, Republic of Serbia (45°38'36.5"N, 19°30'24.1"E), in an 11-year-old pear orchard (Williams variety), with a distance between the rows of 4 m, and between the trees in a row 2.5 m. Experiment was set up in four replications, as a randomized block design in an open field, according to standard EPPO methods (2015).

For the control of *C. pyri* larvae, during overlapping generations, PPP was foliar applied in the amount of 0.3 kg/ha, with water consumption of 1 000 L/ha, while pears were in the BBCH 75 phenophase. The number of larvae was monitored visually, in the control variant and in the treatments, immediately before the application of spinetoram and 7 days after. The number of larvae aged L_1–L_3 and L_4–L_5 on ten marked shoots 20–25 cm long, on the outside of the treetop exposed to the sun, at a height of 1–2 m, was monitored. Under the magnifying glass, in the

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field conditions, the counting and identification of larval ages were performed.

During the experiment, meteorological conditions were also monitored. Average daily temperatures were between 16.8–21.1 °C, while precipitation ranged from 0.1–4.8 mm.

Residue analysis. For the analysis of spinetoram residues, pear fruit samples (500–1 000 g) were randomly collected 1 h after the application, and each day during one week. Fruit samples were packed in plastic bags, delivered to the laboratory and stored (–20 °C) until the analysis. In addition, for the method validation, untreated fruits were sampled.

The extraction and clean-up of spinetoram from pear fruits were performed with acetonitrile and the QuEChERS method (EN 15662) (Anastassiades et al. 2003). Spinetoram residues were analyzed using high-performance liquid chromatography with a diode array detector (HPLC-DAD) (Agilent Technologies, USA).

RESULTS

Efficacy assay. Monitoring of the common pear psylla larvae in the pear orchard, as well as their number, were performed through visual observation of marked young shoots. The first visual examination was performed immediately before the insecticide application (July 10). Then, the number of pear psylla larvae aged L_1–L_3 ranged on average 1–11.25 per variant, and in L_4–L_5 5.25–9.25 per variant (Table 1). Seven days after the treatment (July 17) with spinetoram, the number of larvae aged L_1–L_3 decreased significantly, compared to the control, with an achieved

Method validation. Optimal conditions for the determination and separation of spinetoram residues were achieved by high-performance liquid chromatography with a diode-array detection (HPLC-DAD) and Zorbax Eclipse XDB-C18 (Agilent Technologies, USA) column (50 × 4.6 mm, 1.8 μm). The mobile phase consists of 10 mM NH_4COOH and acetonitrile (8/92, v/v), under isocratic elution (0.75 mL/min), the column was kept at room temperature, while injected volume was 20 μL. Spinetoram was detected at wavelength of 250 nm. Under these conditions, both spinetoram components, XDE-175-J and XDE-175-L, were separated and detected (Figure 2).

The method for the determination of spinetoram residues in pear fruits has been developed and validated through the linearity, accuracy and precision of the method, limit of detection (LOD) and quantification (LOQ), and matrix effect (ME; %), according to the SANTE (2020) (Table 3).

The linearity of the detector response was determined at five concentration levels (0.03–2.0 μg/mL). In the range of tested mass concentrations, good linearity was achieved (R^2 = 0.999). The precision was evaluated through the repeatability, i.e. 20 μL of spinetoram standard solution, prepared in a pear matrix (1.0 μg/mL) was injected five times. The relative standard deviation (RSD) of 0.55% indicates high reproducibility of spinetoram determination using this method (RSD ≤ 20%).

Table 1. Average number of larvae (L_1–L_3) *Cacopsylla pyri* and efficacy of spinetoram

Insecticide	Before application			After application			
	Σ	$\bar{x}$	SD	Σ	$\bar{x}$	SD	E (%)
Spinetoram (0.3 kg/ha)	4	1.00	1.00	7	1.75^b	1.09	81.6
Control	45	11.25	3.96	38	9.5^a	2.87	–
LSD (0.05)		–			3.26		

Letters indicate significant differences between groups (P < 0.05); $\bar{x}$ – average values ; E – efficacy ; LSD – least significant difference

Table 2. Average number of larvae (L_4–L_5) *Cacopsylla pyri* and efficacy of spinetoram

Insecticide	Before application			After application			
	Σ	$\bar{x}$	SD	Σ	$\bar{x}$	SD	E (%)
Spinetoram (0.3 kg/ha)	21	5.25	0.43	18	4.5^b	1.12	64.0
Control	37	9.25	1.29	50	12.5^a	2.96	–
LSD (0.05)		–			3.16		

Letters indicate significant differences between groups (P < 0.05); $\bar{x}$ – average values ; E – efficacy ; LSD – least significant difference

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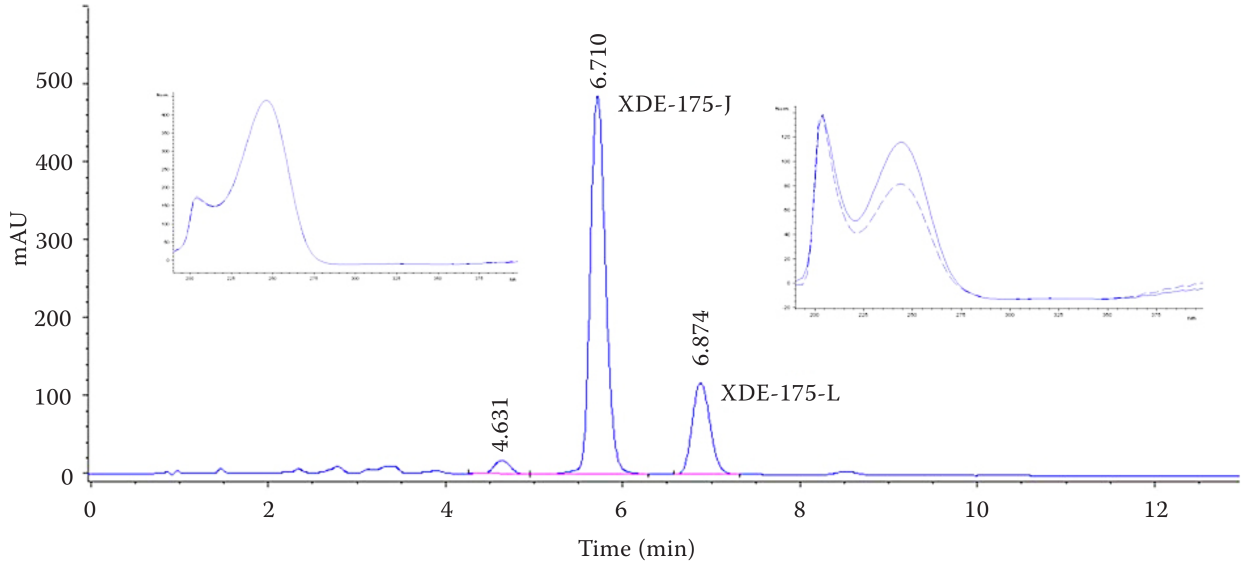

Figure 2. Chromatogram of standard solution of spinetoram in acetonitrile and overlapped spectra of spinetoram from acetonitrile and pear sample

Table 3. Validation parameters

Insecticide	*LOD* (mg/kg)	*LOQ* (mg/kg)	R^2 (0.02–2.0 µg/mL)	Precision (%)	Recovery (%)	*ME* (%)
Spinetoram	0.01	0.03	0.999	0.55	87.3–102.7	99.0

LOD – the limit of detection; *LOQ* – limit of quantitation ; R^2 – the coefficient of determination; *ME* – matrix effect

For the accuracy of the method, blank samples were spiked with a spinetoram standard solution at three levels (0.03, 0.5 and 1.0 mg/kg). The average value of the recovery, calculated by the matrix-matched calibration curve, was 87.3–102.7%. Obtained results were completely in accordance with the criteria for the determination of pesticide residues in food (70–120%, SANTE 12682/2019).

The calculated limit of detection (*LOD*) (the lowest concentration at which the value of the extraction yield is acceptable) determined as signal-to-noise of 3 and limit of quantitation (*LOQ*) determined by considering a signal-to-noise ratio of 10 by using matrix-matched calibration curves. The obtained *LOD* of spinetoram using the described method was 0.01 mg/kg, while the *LOQ* was 0.03 mg/kg. In order to evaluate the influence of the pear matrix on the detector response, the *ME* was determined. The performed determination was based on the comparison of the calibration curve of the spinetoram standard in acetonitrile and pear matrix (0.03–2.0 µg/mL). Achieved *ME* of 99% can be considered that there is no influence of the pear matrix on the determination of spinetoram residues.

Dynamic dissipation. A validated method was applied for the determination of spinetoram residues in pear fruits, after the application of PPP based on this insecticide. Although the ME was 99%, the determination of spinetoram residues in pear fruits was performed using matrix-match calibration curve, and the residues were expressed as the sum of isomers XDE-175-J and XDE-175-L. The obtained results are shown in Table 3.

The initial deposit of spinetoram in pear fruits was 0.51 mg/kg. In the samples collected one day after the treatment, the average content of spinetoram was 0.43 mg/kg, with a loss of 15.69%. Following day the content of spinetoram residues decreased (0.29 mg/kg), indicating the presence of residues of 56.86%. Further analysis revealed a gradual decrease in the content of spinetoram in pear samples. On the third day after the application of PPP, loss of initial content of spinetoram was 58.82%, while on the fifth day it was more than 80%. Seven days after the application, the residues of spinetoram were not determined, i.e. they were below the *LOD* of 0.01 mg/kg (Table 4).

The prescribed maximum residue level (*MRL*) of spinetoram in pear fruits (European

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Table 4. Insecticide spinetoram residues in pear fruits

Day after treatment	Spinetoram residues (mg/kg)	Persistance (%)	Loss (%)
0*	0.51	100.00	0.00
1	0.43	84.31	15.69
2	0.29	56.86	43.14
3	0.21	41.18	58.82
4	0.16	31.37	68.63
5	0.09	17.65	82.35
6	0.07	13.73	86.27
7	≤ LOD	0.02	99.98
MRL (mg/kg)	0.2		
DT_{50} (days)		2.17	

*Initial amount of spinetoram, after the drying deposit; *MRL* – maximum residue level; DT_{50} – half-life; *LOD* – the limit of detection

Commision 2020) is 0.2 mg/kg. In this research, the content of spinetoram was at the *MRL* level between the third and fourth days after application.

Spinetoram residue concentrations were plotted against elapsed time after the application and the obtained results have shown that the dissipation dynamic of pesticides is a first-order kinetic reaction (Equation 1) with R^2 = 0.979.

$$C_t = C_0 e^{kt} \quad (1)$$

where: C_0 – the initial amount of pesticide residues after application; k – degradation constant; C_t – the pesticide residues over time.

Half-life (DT_{50}) of spinetoram insecticide in pear fruits was calculated using results in the dissipation study (Figure 3) and the formula $DT_{50} = \ln 2/k$; where

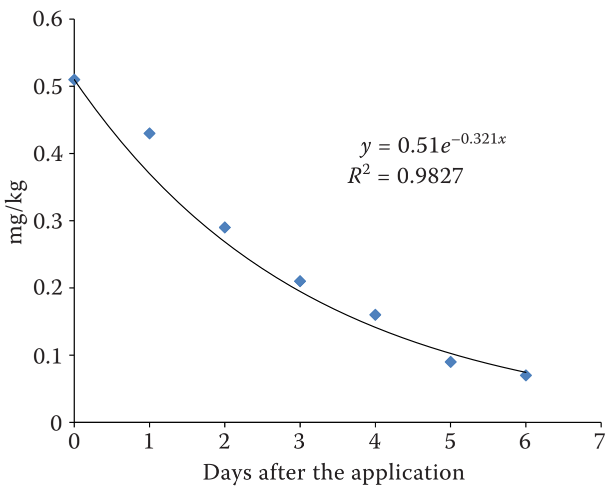

Figure 3. Dissipation of spinetoram in sampled pear fruits

the k is the degradation constant, and ln is the natural logarithm (Gupta et al. 2008). Based on the obtained results, the DT_{50} of spinetoram in pear fruits is 2.17 days. Pre-harvest interval (PHI) is the time between pesticide application and harvest, when pesticide residues are at the *MRL* level or lower. It was calculated considering *MRL* for spinetoram in pear fruit and the dissipation study and the obtained results indicated the PHI of 3 days.

DISCUSSION

The application of PPP based on spinetoram in the amount of 0.3 kg/ha achieved satisfying efficacy, especially in the control of L_1–L_3 larvae, which is very important from the aspect of successful pear protection from the most significant pest. Also, these results indicate the susceptibility of pear psylla populations to spinetoram, which was expected considering that spinetoram is a newer generation insecticide, i.e. it has recently been used for this purpose. Pear psylla has several generations per year, mostly overlapped, which require intensive plant protection with several insecticide treatments. As a consequence, there is a possibility of the occurrence of a resistant population of *C. pyri*, as well as residue accumulation in fruits. According to the available data, the resistance of pear psylla to spinetoram has not been developed, and spinetoram could be an important tool in the insecticide resistance management IPM.

In the experiment conducted in Switzerland (2003), the efficacy of different insecticides (neem, pyrethrin, spinosad, and rotenone) and a repellent

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(kaolin) applied with different strategies (single or repeated applications). Insecticides were used for the control of the over-wintering pear psylla and first generation of nymphs was tested in a field conditions. To determine the efficacy of the tested substances, the number of adults was monitored. Spinosyn insecticide (spinosad) was applied individually (0.03%) and in combination with rape oil (0.03%+ rape oil 1%) and in in combination with rape oil spinosad showed slightly higher efficacy. Similar efficacy against pear psylla expressed rotenone based PPP (Daniel & Wyss 2004). In order to evaluate the efficacy of spinetoram in the control of apple moth and pear psylla in orchards in Italy, six experiments were performed. The achieved results showed excellent activity of spinetoram, with no harmful effects on beneficial arthropods and other non-target organisms (Boselli & Sccannavini 2014).

However, besides its positive effects on the target species and environment, it is still necessary to evaluate the presence and the behavior of their residues in order to determine dissipation dynamics and persistence in fruits. For the determination of pesticide residues in different matrices, it is necessary to apply a sensitive enough and reliable method, validated according to SANTE 12682/2019 criteria. Method for the determination of spinetoram residues in pear fruits validated completely fulfill the mentioned standard. HPLC-DAD was used for the analysis of spinetoram residues in tomato and leafy vegetables (Liu et al. 2011; Malhat 2013; Hafez et al. 2016), corresponding to the results obtained in this study.

The behavior of spinetoram was analyzed in some previous research. Half-lives of spinetoram in tomato fruits obtained in open field conditions were 2.6 and 2.71 days (Malhat 2013; Hafez et al. 2016). Dissipation dynamics of spinetoram in pepper and cabbage, under greenhouse production, were evaluated (Ali et al. 2018) and the results showed that spinetoram in these vegetables rapidly degrade, with a half-life of 1.95 days and 1.29 days, respectively. The half-lives of spinetoram determined in paddy water, soil and rice straw were 0.35, 6.8 and 1.1 day, respectively (Li et al. 2015). Study of residue behaviour of spinetoram in cauliflower has shown that spinetoram swiftly degraded, with the half-life of 4.85 days (Lin et al. 2020). After the application of spinosyne insecticides in green onion in the recommended rates, half-lives for spinetoram was 1.2 days and for spinosad 1.42 days (Malhat & Abdallah 2019). Using results obtained in the dissipation studies, PHI determined in tomato was 10 days (Malhat 2013; Hafez et al. 2016), while in pepper and cabbage in is only one day (Ali et al. 2018). In this study, spinetoram insecticide applied during overlapping pear psylla generations, expressed satisfying efficacy, especially in the control of L_1–L_3 larvae (81.6%). In addition, QuEChERs based method followed with HPLC-DAD for the analysis of spinetoram residues in pear fruits was developed and validated. Residue analysis showed that spinetoram dissipated rapidly in pear fruits, with a half-life of 2.17 days, similar to those obtained in other crops. The results obtained in the field experiment confirmed the safe use of spinetoram in the pear protection and recommended PHI of 3 days. This study provided the first results of the residues and behavior of spinetoram in pear fruits.

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