

RESEARCH CONCERNING THE REMANENT CHARACTER OF SOME INSECTICIDES IN LETTUCE (*LACTUCA SATIVA*) ROOTS CULTIVATED IN PROTECTED AREAS

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Abstract: Insecticides used in pest control to obtain higher and more stable yields are one of the sources of chemical pollution of the environment with bad impact on all biological systems. In this context, in this paper we show the remanent character of the insecticides imidacloprid and dimethoate in the roots of the tested plant – lettuce (*Lactuca sativa*) – cultivated in protected areas. The experimental field was set according to the randomised block method with three replications both in the solarium and in the glasshouse, between 2009 and 2010. Sampling was done 24 hours and 7 days, respectively, after using the insecticides. We suggested and applied the high-performance liquid chromatography method to detect insecticides UV-VIS. As a result of processing chromatographic information, we could draw the conclusion that the remanence of imidacloprid **24 hours** after

application in the lettuce roots cultivated in the solarium and in the glasshouse was **3.40411 ppm** (parts per million or mg/kg) and **9.02731 ppm**, respectively. The remanence of the imidacloprid **7 days** after application in the tested lettuce roots, according to the chromatogrammes was **1.23337 ppm** in the lettuce roots cultivated in the solarium and **6.30109 ppm** in the lettuce roots cultivated in the glasshouse. **Twenty-four hours** after application, dimethoate was identified in the lettuce roots cultivated in the glasshouse in concentration of **3.32746 ppm**, while it could **not be detected** in the lettuce roots cultivated in the solarium. **The absence** of the dimethoate in the lettuce roots was also identified **7 days** after application in the roots of the lettuce cultivated in both the glasshouse and the solarium.

Keywords: remanence, imidacloprid, dimethoate, roots, lettuce.

INTRODUCTION

Pesticides used in the control of pests and in plant protection are one of the sources of chemical pollution of the environment with serious impact on animal health and noxious effects on biological systems. They were used to obtain larger and more stable yields. According to the estimates of such international organisations as F.A.O. and W.H.O., prohibiting the use of pesticides in intensive agriculture countries would result in a 50% decrease of vegetable, fruit, and cereal yields (ALEXA, 2008).

The residues of these substances are the most serious threat from an ecological and biological point of view since, if used irrationally, they can pollute directly or indirectly the sources of food of man and animals. The physical and chemical features of pesticides lead to a damaging effect on humans: their liver, central nervous system, heart and veins, respiratory apparatus, and suprarenal glands are the most impacted. This is why it is necessary to accumulate precise data through laboratory analyses to ensure protection from toxic effects on the environment and, implicitly, on the humans (ALEXA, 2008).

In this context, the authors of this paper aimed at determining the remanent character of some insecticides in lettuce roots cultivated in protected areas.

MATERIAL AND METHOD

Research was carried out within a trial set within the perimeter of the Didactic Station of the Banat University of Agricultural Science and Veterinary Medicine (BUASVM) in

Timisoara (Romania), between 2009 and 2010, and in the laboratories for the detection of organophosphoric pesticide residues of the same University. The plant to be tested was lettuce (*Lactuca sativa*) and for the study of the remanent effect in the lettuce roots we used the following insecticides:

1. Commando containing 200 g/l of active substance *imidacloprid*.
2. Sinoratox 35CE containing 35% active substance *dimethoate*.

The experimental field was set after the randomised block method with three replications (CIULCĂ, 2002), with trials in protected areas (solarium and glasshouse) on areas of 12 and 6 m², respectively. Harvesting was done manually at intervals of 24 hours and 7 days, respectively, and at the same time. The samples were stored in bags and kept in the freezers of the laboratories for the detection of organophosphoric pesticide residues of the BUASVM in Timisoara. In order to study the remanence of the active substances mentioned above, we chose the HPLC (High Performance Liquid Chromatography) technique. We also suggested and applied the chromatography method in the distribution and detection of insecticides in UV-VIS (JANTSCHI, 2004). We used a Shimadzu chromatograph observing the standards of the present legislation according to the State standard STAS 12905-90. The principle of the method consists in extracting the organophosphoric residues from the petroleum ether and acetonitrile sample, purifying through florisil column passage, and eluting with a mixture of petroleum ether and ethylic ether. We determined quantitatively the pesticide residues in the concentrated eluate (MANDIC, 2002).



Figure 1. Filtering the mixture of petroleum ether



Figure 2. Passing the samples through the florisil column



Figure 3. Collecting the samples with the micropipette



Figure 4. Introducing the samples in the analysis chromatograph.

RESULTS AND DISCUSSION

In order to validate the working method and to confirm the accuracy and the reproducibility of analytical results we established the calibration curves for the active substances used: *imidacloprid* and *dimethoate* (figure 5 and figure 6).

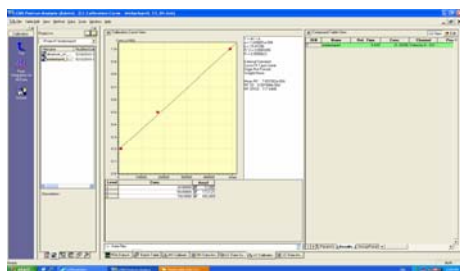


Figure 5. Imidacloprid. The calibration curve

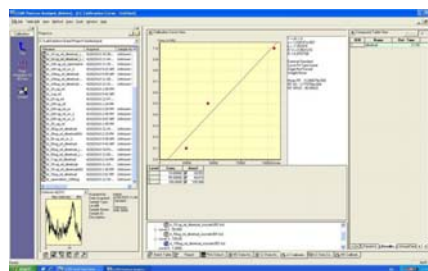


Figure 6. Dimethoate. The calibration curve.

We also designed for the beginning the chromatogrammes for the control sample – lettuce roots. After analyzing them, we could see there were no traces whatsoever of substances (figure 7).

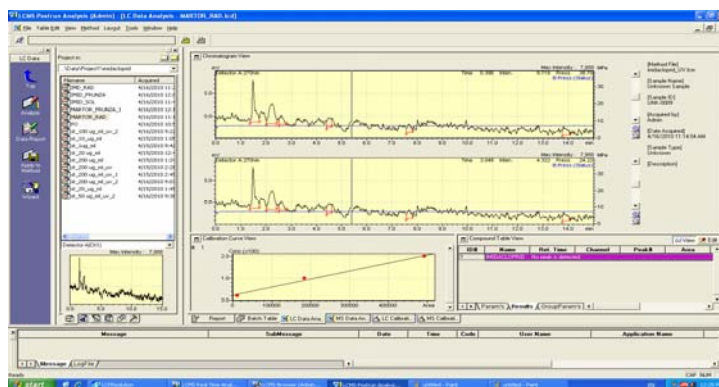


Figure 7. Chromatogramme of the control root sample.

1. Determining the remanence of imidacloprid in the roots of the tested plant (*Lactuca sativa*) cultivated in protected areas.

The amounts of roots used to determine the remanence of the imidacloprid **24 hours** after application in the roots of lettuce cultivated in the solarium and in the glasshouse were 6.74 g and 2.68 g, respectively.

According to the chromatogrammes, we detected a concentration of 22.94372 $\mu\text{g/ml}$ in the roots of lettuce cultivated in the solarium (figure 1.1.), and a concentration of 24.19320 $\mu\text{g/ml}$ in the roots of the lettuce cultivated in the glasshouse (figure 1.2.). These values, converted into ppm (parts per million or mg/kg) are **3.40411 ppm** for the lettuce roots cultivated in the solarium and **9.02731 ppm** for the lettuce roots cultivated in the glasshouse.

We thus determined the amount of insecticide in mg related to the amount of roots under study in kg, obtaining the residues in ppm.

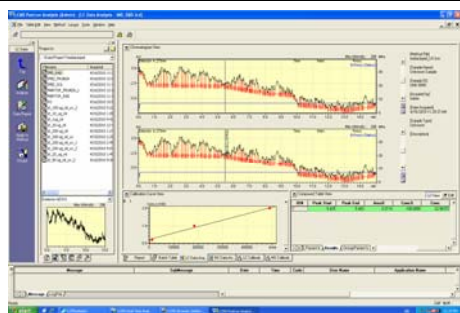


Figure 1.1. Imidacloprid. Chromatogramme of the root extract (lettuce/solarium) 24 hours after application

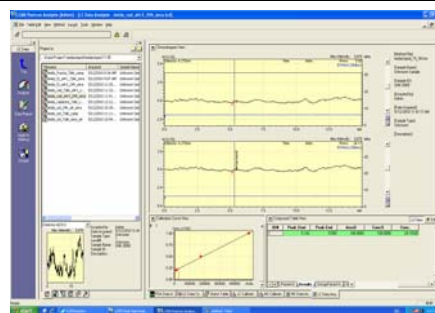


Figure 1.2. Imidacloprid. Chromatogramme of the root extract (lettuce/glasshouse) 24 hours after application.

The amount of lettuce roots for the determination of the remanence of imidacloprid **7 days** after application in the lettuce roots cultivated in the solarium and in the glasshouse were 16.33 g and 3.10g, respectively.

According to chromatogrammes, we detected a concentration of 20.14108 $\mu\text{g/ml}$ in the lettuce roots cultivated in the solarium (figure 1.3.) and a concentration of 19.53339 $\mu\text{g/ml}$ in the lettuce roots cultivated in the glasshouse, respectively (figure1.4.). These values, converted into ppm are **1.23337 ppm** for the lettuce roots cultivated in the solarium and **6.30109 ppm** for the lettuce roots cultivated in the glasshouse.

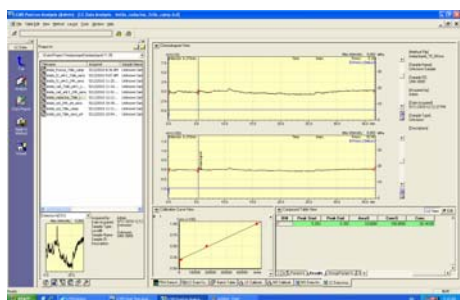


Figure 1.3. Imidacloprid. Chromatogramme of the root extract (lettuce/solarium) 7 days after application.

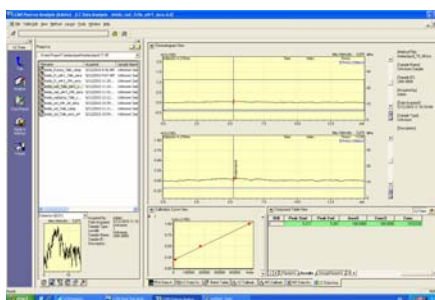


Figure1.4. Imidacloprid. Chromatogramme of the root extract (lettuce/glasshouse) 7 days after application.

2. Determining the remanence of dimethoate in the roots of the tested plant (*Lactuca sativa*) cultivated in protected areas.

The amounts of lettuce roots used to determine the remanence of the active substance dimethoate **24 hours** after application in the lettuce roots cultivated in the solarium and in the lettuce roots cultivated in the glasshouse were 5.64g and 2.28g, respectively.

According to the chromatogrammes, we detected a concentration of 0.0000 $\mu\text{g/ml}$ in the lettuce roots cultivated in the solarium (figure 2.1.) and a concentration of 7.58663 $\mu\text{g/ml}$ in the lettuce roots cultivated in the glasshouse, respectively (figure 2.2). These values converted into ppm are **0.0000 ppm** for the lettuce roots cultivated in the solarium and **3.32746 ppm** for the lettuce roots cultivated in the glasshouse, respectively.

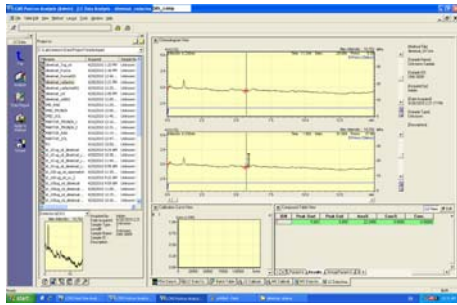


Figure 2.1. Dimethoate. Chromatogramme of the root extract (lettuce/solarium) 24 hours after application.

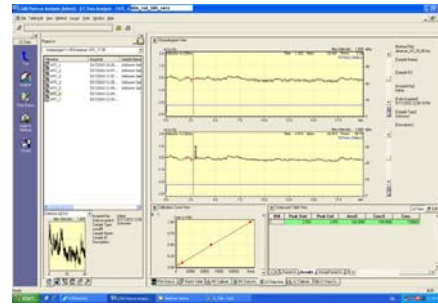


Figure 2.2. Dimethoate. Chromatogramme of the root extract (lettuce/glasshouse) 24 hours after application.

Analysing the chromatogrammes below (figures 2.3. and 2.4.) we can see that the active substance dimethoate could not be detected in the lettuce roots cultivated in the solarium or in the lettuce roots cultivated in the glasshouse 7 days after application (**0.0000ppm** concentration).

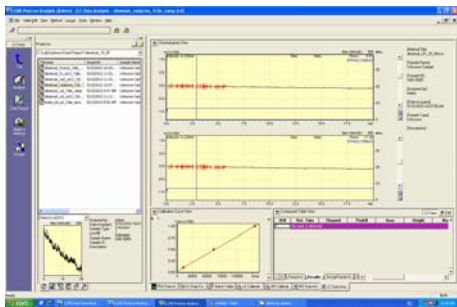


Figure 2.3. Dimethoate. Chromatogramme of the root extract (lettuce/solarium) 7 days after application.

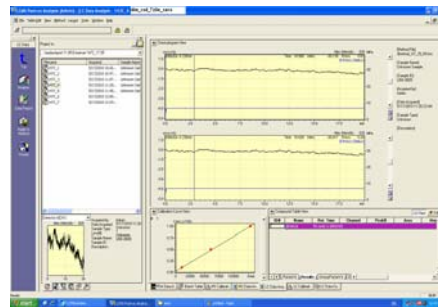


Figure 2.4. Dimethoate. Chromatogramme of the root extract (lettuce/glasshouse) 7 days after application.

Table 1

The remanence determination results of the insecticides imidacloprid and dimethoate in the roots of the tested plants –lettuce (*Lactuca sativa*)-cultivated in protected areas, 2009-2010

Active substance	The remanence in the lettuce roots			
	24 hours after application		7 days after application	
	solarium (ppm)	glasshouse (ppm)	solarium (ppm)	glasshouse (ppm)
DIMETHOATE	0,0000	3,32746	0,0000	0,0000
IMIDA CLOPRID	3,4041	9,0273	1,2333	6,3010

Table 1 presents the remanence determination results of the insecticides imidacloprid and dimethoate in the roots of the tested plants.

CONCLUSIONS

1. Analysing the chromatogrammes of the control sample of lettuce roots (*Lactuca sativa*) under study shows no traces whatsoever of the tested active substances.

2. Assessing imidacloprid **24 hours** after application in the lettuce roots cultivated in the solarium ad in the glasshouse, we detected concentrations of **3.40411 ppm** and **9.02731 ppm**, respectively.

3. The remanence of the imidacloprid **7 days** after application in the tested lettuce roots according to the chromatogrammes was **1.23337 ppm** in the lettuce roots cultivated in the solarium and **6.30109 ppm** in the lettuce roots cultivated in the glasshouse.

4. According to the chromatogrammes, the remanence of the dimethoate **24 hours** after application in the lettuce roots cultivated in the solarium was **0.0000 ppm**, while in the lettuce roots cultivated in the glasshouse it was **3.32746 ppm**.

5. Analysing the chromatogrammes we could see that **7 days** after application, **we could not detect** the active substance dimethoate in the lettuce roots cultivated in both the solarium and the glasshouse.

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Figure 3. "Lettuce cultivated in the glasshouse".