

Environmental Distribution of Acetochlor, Atrazine, Chlorpyrifos, and Propisochlor under Field Conditions

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The environmental behavior, movement, distribution, persistence, and runoff by rainfall of the pesticides acetochlor, atrazine, chlorpyrifos, and propisochlor were studied under field conditions during a five-month period at normal weather conditions. The pesticide concentrations in soil depths of 0–5 and 5–20 cm, and in sediment and runoff water samples (collected from an artificial reservoir built in the lower part of the experimental plot) were measured every second week and following every runoff event. The contamination of a stream running across the lowest part of the plot was also monitored. The weather conditions were also recorded at the experimental site. The pesticide residues were quantified by a capillary gas chromatograph equipped with a nitrogen phosphorus selective detector (GC–NPD). There was a consistent decrease in pesticide residues in the 0–5 cm soil layer with time after spaying. At 140 days after treatment only atrazine and chlorpyrifos were present; acetochlor and propisochlor were not detected in this soil layer. Atrazine and chlorpyrifos in the soil at a depth of 5–20 cm were detectable during the whole experimental interval, whereas acetochlor and propisochlor concentrations were below the limit of detection. Pesticide losses by the surface runoff process and the contamination of the stream were closely related to the time of rainfall elapsed after treatment and amount of rain at the experimental plots. Losses were primarily dependent on surface rainfall volume and intensity. The maximum detected residues of atrazine and acetochlor in streamwater were 1 order of magnitude higher than the maximum residue limit specified by the European Union (EU) for environmental and drinking water (0.1 µg/L for individual compounds and 0.5 µg/L for total pesticides). Chlorpyrifos and propisochlor were not detected in this matrix.

Keywords: Pesticides; runoff; water; sediment; soil; pesticide analysis

INTRODUCTION

The progressive increase of production and application of chemicals for agricultural activities has converted the problem of environmental contamination into a national and international issue. A number of pesticides have been detected in groundwater and surface water in the United States and Europe in the past years. In 43 states of the United States, at least 143 pesticides and 21 transformation products have been detected, including compounds in every major chemical class (1–3). The most frequently detected pesticides were those that have been used extensively, such as triazines (atrazine, cyanazine, and simazine), acetanilide herbicides (metolachlor, acetochlor, and alachlor) and 2,4-D. The most frequently detected insecticides that are currently used were carbofuran and diazinon (4–6). German researchers detected 145 organic polluting chemicals in the river Elbe, including, among others, triazines, dimethoate, and their metabolites (7). In the Italian river Arno and in Greek natural surface water several pesticides have been measured as well (8, 9). According to the survey of the Hungarian Environmental Information and Monitoring System, phenoxyacetic acid derivatives (2,4-D and MCPA), triazine compounds (atrazine, terbutryn, and terbumeton) and chlorinated

hydrocarbons (DDE, DDT, DDD, lindane, and endosulfan) could be detected in 4.6% of the soil samples taken in agricultural areas in the 1997–1998 season. Analyses of drinking water and groundwater samples originated from different parts of Hungary have shown that 21 pesticides of different types were determined in 62 samples from 28 sampling points in the same time period. The concentration of pesticide exceeded the level of 0.1 µg/L in 8 cases for diazinon, atrazine, prometryn, 2,4-D, and MCPA (10).

As the need for water increases, and the amount of potable water in the world is limited, people are increasingly conscious of the need to protect water resources. To reduce the risk of contamination, it is essential to understand the factors that affect the behavior of pesticides in the natural environment. The fate and behavior of pesticides in the soil environment involve several different and often simultaneous phenomena. The pesticides may be transformed by chemical and biological processes or transported from the site of application by runoff, leaching, volatilization, and wind erosion. Attention should be focused on behavior within the soil environment (11–14). The behavior of a pesticide in soil depends on its physicochemical properties, the nature of the soil and the organisms it contains, climatic conditions, crop type, cropping practices, and water management methods. All of these properties and processes determine the biological availability of pesticides and their redistribution from the point of application (15–17). In practice, these various factors all

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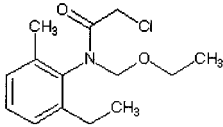
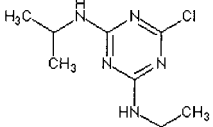
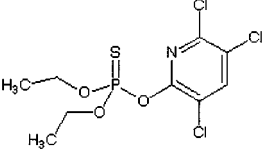
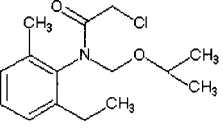
Common name	Molecular	IUPAC name	MW
ACETOCHLOR		2-chloro-N-ethoxymethyl-6'-ethylacet-o-toluidide	269,77
ATRAZINE		6-chloro-N ² -ethyl-N ⁴ -isopropyl-1,3,5-triazine-2,4-diamine	215,69
CHLORPYRIFOS		O,O-diethyl O-3,5,6-trichloro-2-pyridyl phosphorothioate	350,59
PROPISOCHLOR		2-chloro-6'-ethyl-N-isopropoxymethylaceto-o-toluidide	283,77

Figure 1. Chemical structures of the four pesticides studied.

interact, and ideally they should be considered together. In recent years there have been several works on the behavior of pesticides in soil. Many laboratory studies (18–23) and field experiments (24, 25) have been made. However, laboratory studies were mainly aimed at clarifying individual effects of the above-mentioned factors on pesticide–environment interaction. Such laboratory studies cannot be easily interpreted under real field conditions. A product's hazard cannot be assessed sufficiently exclusively from laboratory studies because of different environmental and socio-economic conditions of each country and the large number of interacting environmental and anthropogenic factors. In the present study we describe the fate and transport of four pesticides that are commonly used in Hungary. Three herbicides – acetochlor, atrazine, and propisochlor – and one insecticide – chlorpyrifos – were investigated to obtain information on the environmental behavior of these chemicals under field conditions.

MATERIALS AND METHODS

Chemicals, Reagents, and Standards. Acetochlor, atrazine, chlorpyrifos and propisochlor, all with purity higher than 99%, were obtained from Sigma Aldrich Kft. (Hungary, Budapest). The structures of these chemicals are shown in Figure 1. Chromatographic-grade acetone, methanol, methylene chloride, and *n*-hexane, and analytical-grade ammonium acetate, sodium sulfate (anhydrous), and acetic acid, were purchased from Merck (Darmstadt, Germany). Stock standard solutions of each pesticide (1 mg/mL) were prepared by dissolving the required amount in acetone and were kept under refrigeration. Dilutions were made daily with *n*-hexane to the desired final concentration before gas chromatographic analysis. Extraction cartridges used were Waters Sep-Pak octadecyl (C₁₈, 500 mg) columns (Waters Corp., Milford, MA).

Experimental Plot and Soil. The experiment was conducted on a plot plowed to a depth of 25 cm and sown with maize. Crop characteristics were as follows: sowing depth 5 cm, row width 74 cm, and plant spacing 20 cm. Average slope of the land was 2%. The type of soil was sandy loam (Luvisol according to classification of Food and Agriculture Organiza-

tion of the United Nations), which has the following composition: 1.26% organic matter, 23.7% silt, 14.6% clay, 60.7% sand, and 0.9% calcium carbonate. Formulations of commercial preparation were emulsified concentrate for propisochlor and chlorpyrifos, and aqueous suspension in the case of acetochlor and atrazine. Content of active ingredients (a.i.) of test substances were 480 g/L chlorpyrifos, 300 g/L acetochlor, 200 g/L atrazine, and 840 g/L propisochlor. The field was sprayed as a preemerge treatment at the recommended application rate: 1500 g a.i./ha, 1000 g a.i./ha, 720 g a.i./ha, and 2100 g a.i./ha dose of acetochlor, atrazine, chlorpyrifos, and propisochlor, respectively.

Weather Conditions. The meteorological conditions (daily minimum and maximum air temperature and precipitation) were recorded at the experimental site. The quantity of rainfall was recorded by a portable liquid-level recorder attached to an artificial reservoir. Precipitation and rainfall data are summarized in Table 1.

Sampling Method. An H-type flume (60 cm deep) was built in the lower part of the experimental plot to collect runoff water and sediment. The stream flowed along the lowest side of experimental plot. Prior to pesticide application, a mixed soil sample was taken from a depth of 0–20 cm as an untreated control sample. Composite soil samples were taken from 12 predetermined positions of the experimental plot from depths of 0–5 cm and 5–20 cm, immediately after application and on days 14, 28, 42, 56, 98, 126, and 140 after application. The sampling was carried out with a special drilling device for obtaining undisturbed soil cores in order to avoid contamination of the lower soil layers by soil particles from the upper layers. Also, water samples were collected from the stream at the same time. The runoff water and sediment samples were collected from the artificial reservoir after every runoff event during the experimental interval. The flume and the approach box were cleaned (sediment and collected water were removed) after each runoff event. Soil samples were collected in polyethylene bags. Water and sediment samples were collected after sedimentation onsite, in amber glass flasks and polyethylene bags, respectively. The samples were placed in cool boxes and transported to the laboratory and stored at –18 °C until preparation.

Sample Preparation. After removal of pieces of stones and plants, the soil or sediment samples were homogenized. Portions (50 g) were weighed and extracted with 100 mL of

Table 1. Precipitation during the Experiment and Amount of Rain during the Runoff Events Recorded at the Experimental Plot

time interval (DAT ^a)	precipitation (mm)	date of runoff event (DAT)	rainfall ^b (mm)
0–6	29.8		
6–13	20.3	13	17.2
13–23	28.6	23	17.5
23–25	21.0		
25–26	49.7	26	43.7
26–46	20.2		
46–52	19.6		
52–54	41.3	54	40.3
54–62	4.9		
62–67	34.0	67	29.4
67–68	21.0		
68–69	24.1	69	23.5
69–86	46.2		
86–89	15.1	89	14.1
89–101	27.3	101	25.4
101–105	3.2		
105–116	35	116	31.2
116–129	20.4		
129–137	33.6	137	30.6

^a DAT, days after treatment. ^b Amount of rain recorded at the experimental plot during the runoff events.

acetone (containing 2 mL of 2 M ammonium-acetate) on a flask shaker for 30 min. The extract was filtered, and the extraction was repeated two more times. Cleanup of sample extract was carried out by liquid-liquid partitioning. A 450-mL portion of 2% sodium sulfate solution was added to the combined filtrate, and it was extracted with 3 × 100 mL of methylene chloride. The methylene chloride phase was drained through 30 g of anhydrous sodium sulfate and was rotary evaporated. The residue was dissolved in 5 mL of *n*-hexane and analyzed. At the time of analysis, 50 g of soil or sediment was dried at 105 °C till constant weight was achieved in order to determine the moisture content of samples.

Extraction of pesticides from water samples was performed with the C₁₈ solid-phase extraction method. The collected water sample was equilibrated to room temperature and filtered through filter paper, and the pH was adjusted to 7.2–7.4 with acetic acid. SPE cartridges were activated by washing once with 2 mL of methanol and 2 mL of distilled water. A 100-mL aliquot of supernatant was loaded into the reservoir of the extraction cartridge and drawn slowly through the column. After sample addition, the stationary phase with the retained pesticides was dried for 5 min with air. Elution was performed with 10 mL of methylene chloride. The organic solvent was drained through anhydrous sodium sulfate and evaporated to dryness under a gentle stream of nitrogen. The residue was dissolved in 0.5 mL of *n*-hexane and analyzed.

Capillary Gas Chromatography Analysis. A Chrompack 9000 gas chromatograph equipped with a nitrogen phosphorus selective detector (GC-NPD) was used for simultaneous identification and quantification of acetochlor, atrazine, chlorpyrifos, and propisochlor. Chromatographic separation was achieved on a CP-SIL 8CB capillary column: length 25 m, column i.d. 0.53 mm, and film thickness 1 μm (Chrompack International B. V., Middelburg, The Netherlands). The GC operating conditions were as follows. The injector port temperature was 230 °C, and the detector temperature was 270 °C. The oven temperature was 140 °C for 1 min, 140–180 °C at 12 °C/min, 180 °C for 10 min, 180–250 °C at 30 °C/min, and isothermal hold at 250 °C for 10 min. Nitrogen was used as the carrier gas at a flow rate of 9.6 mL/min; hydrogen and air flow rate were 3.6 mL/min and 55 mL/min, respectively. Injection volume was 1 μL. Data acquisition and processing were accomplished by means of a Waters Maxima 820 data station running on an IBM PC/AT 486 computer.

Method Characteristics. The calibration curves were linear up to 20.0 ng/μL (regression coefficients 0.999). The limit of detection was 0.05–0.2 μg/L in water and 0.02–0.001 mg/kg in soil for each compound as determined according to the

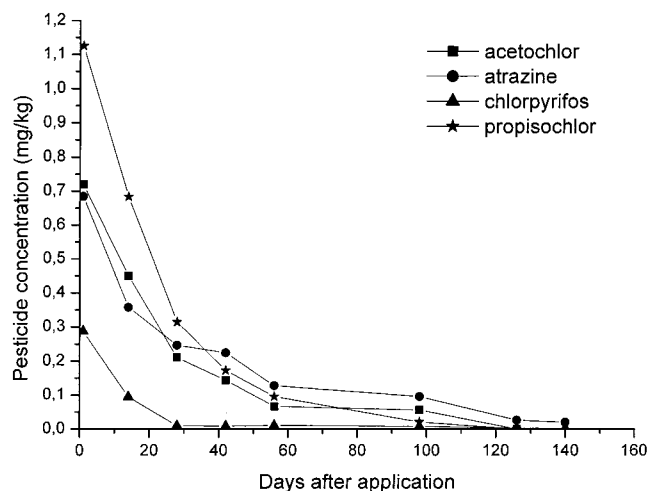


Figure 2. Changes of the amounts of pesticides in soil depth of 0–5 cm for the experimental interval, expressed on a dry material basis.

American Society of Testing and Materials ASTM D4210 standard. The analytical method was validated by analyzing two series ($n = 5$) of water samples spiked with the four pesticides at 0.5 μg/L and 10.0 μg/L concentration levels, and two series ($n = 5$) of soil samples spiked at 0.05 mg/kg, 0.10 mg/kg, and 0.5 mg/kg concentration levels. Reproducibility was appropriate with relative standard deviations lower than 5% in all cases and recoveries ranging from 87.4–96.7% for soil and 84.7–89.6% for water samples.

RESULTS

Pesticide Distribution and Persistence in Soil.

The initial concentrations of acetochlor, atrazine, chlorpyrifos, and propisochlor in soil were 0.740 mg/kg, 0.670 mg/kg, 0.298 mg/kg, and 1.105 mg/kg immediately after application. There was a consistent decrease in pesticide residue concentration in the 0–5 cm soil layer with increasing time after spraying. The residues of compounds decreased to 0.462 mg/kg (62.5% of the initial), 0.376 mg/kg (56.2%), 0.098 mg/kg (33.0%), and 0.672 mg/kg (60.8%) at the 14th day, and 0.217 mg/kg (29.3%), 0.242 mg/kg (36.1%), 0.010 mg/kg (3.5%), and 0.309 mg/kg (28%), respectively, at day 28. By 140 days after treatment only 1.37% atrazine and 0.69% chlorpyrifos of the initial concentration were measured, and acetochlor and propisochlor were not detected. (Figure 2.) A significant vertical downward movement could be observed in case of atrazine and chlorpyrifos. It took place intermittently during and immediately after periods of rainfall. The leaching was more pronounced during the first four weeks after application. This assumption is based on the concentrations of atrazine and chlorpyrifos in the 5–20 cm soil layer, which were 0.029 mg/kg and 0.030 mg/kg at the 28th day, respectively. No significant change could be observed from day 42 to day 140. Acetochlor and propisochlor were not detected in the 5–20 cm soil layer; presumably they remained in the surface 5 cm of the soil and dissipated. (Figure 3.)

Runoff of Pesticides. Runoff had enough velocity and transport capacity to carry soil particles during rainfall. The highest pesticide concentrations of runoff water were detected following the 3rd rainfall event, at 28 days after application: 89.1 μg/L acetochlor, 154 μg/L atrazine, 0.139 μg/L chlorpyrifos, and 47.4 μg/L propisochlor were detected. The maximum concentration of

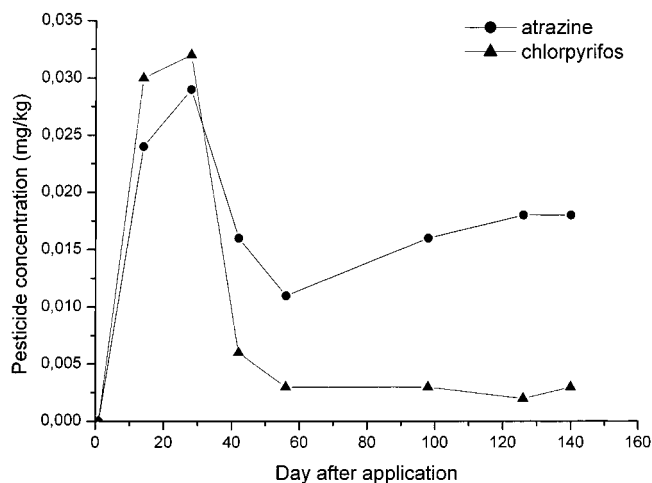


Figure 3. Changes of the amounts of pesticides in soil depth of 5–20 cm for the experimental interval, expressed on a dry material basis.

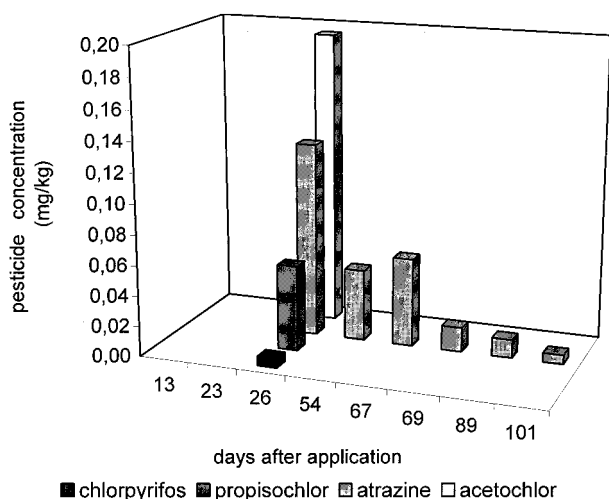


Figure 4. Acetochlor, atrazine, chlorpyrifos, and propisochlor concentrations in sediment phase of runoff, expressed on a dry material basis.

pesticides in sediment was detected at the same time: 0.198 mg/kg acetochlor, 0.128 mg/kg atrazine, 0.007 mg/kg chlorpyrifos, and 0.056 mg/kg propisochlor were detected. The concentrations of pesticides in the sediment and surface runoff water are presented in Figures 4 and 5. The maximum detected residues of atrazine and acetochlor in streamwater were 6.12 $\mu\text{g/L}$ and 1.47 $\mu\text{g/L}$ respectively, while chlorpyrifos and propisochlor were not detected.

DISCUSSION

On the basis of the results of the study it can be concluded that the pesticide downward movement, losses by the surface runoff process, and the contamination of stream are closely related to time of rainfall elapsed after treatment and amount of rain at the experimental plots.

The initial higher leaching of pesticides is explainable with the higher surface concentration and the rudimentary roots of plants (which could not decrease water percolation to deeper zone) at the beginning of the experiment.

The runoff caused losses that were primarily dependent on rainfall volume and intensity. In addition, runoff

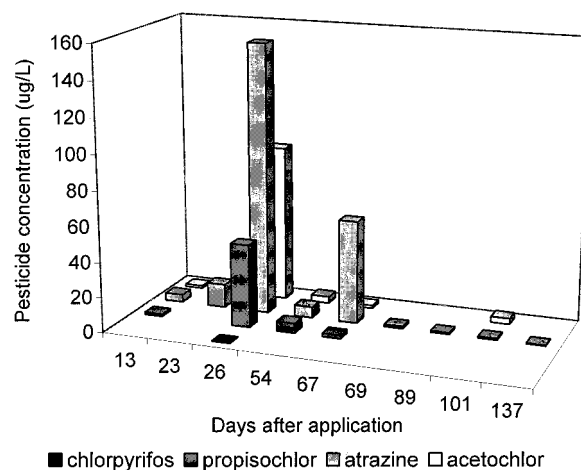


Figure 5. Acetochlor, atrazine, chlorpyrifos, and propisochlor concentrations in water phase of runoff.

Table 2. Physicochemical Properties of the Four Pesticides Investigated^a

pesticide	$\log P_{ow}$	K_{oc}	water solubility (mg/L)
acetochlor	3.03	313	223 (25 °C)
atrazine	2.50	39–155	33 (20 °C)
chlorpyrifos	4.70	8498 ^b	1.4 (25 °C)
propisochlor	3.50	—	184 (20 °C)

^a The logarithm of octanol–water partition coefficients ($\log P_{ow}$), soil sorption coefficients (K_{oc}), and water solubility values are from ref 32. ^b From ref 26.

and erosion were aggravated during the first part of the experiment by the lack of crop canopy, which could protect the soil from direct raindrop impact. The detected maximum concentrations of atrazine and acetochlor in sediment and runoff water were about 2–3 times higher than those of propisochlor and 2 orders of magnitude higher than chlorpyrifos concentrations. Downward movement of chlorpyrifos was more pronounced than those of the others. These phenomena might be explained only partly with the different physicochemical properties of the compounds. Atrazine, acetochlor, and propisochlor are characterized by relatively high water solubility and low soil sorption coefficient, whereas chlorpyrifos has low water solubility and high sorption coefficient (Table 2.) The typical field dissipation half-lives of chlorpyrifos for soil-surface and soil-incorporated applications at agricultural use rates range from 1–2 weeks and 4–8 weeks, respectively (26). Our results are in good agreement with these observations. The first runoff event happened at 26 days after application, and it is assumed there was not considerable chlorpyrifos amount remaining in the upper soil layer at that time. The half-life of other three pesticides was approximately 3 weeks.

The pesticide contamination of the stream was significant following the first rainfall. The European Union (EU) provides directives and regulations about the maximum residue levels (MRL) of pesticides in food-stuffs as well as in water, in accordance with the recommendations of the Codex Committee on Pesticide Residues (CCPR). In the case of environmental and drinking water, the EU directive declares that the concentration should not exceed the level of 0.1 $\mu\text{g/L}$ for individual compounds and 0.5 $\mu\text{g/L}$ for total pesticides (80/778/ECC). The detected residues of atrazine and acetochlor in streamwater were 1 order of magnitude

higher than the MRL at the end of the second week after application.

Knowledge of the fate of pesticides in soil, in terms of basic phenomena such as transport, retention, and transformation, is essential to the development of predictive tools for the prevention of surface water and groundwater contamination. Dissolved pesticides or those adsorbed to eroding soil particles can result in contamination of surface water resources (27–31). Further research will be undertaken to investigate the soil adsorption mechanism of these pesticides to understand more clearly the causes for losses and estimate the potential migration to groundwater.

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