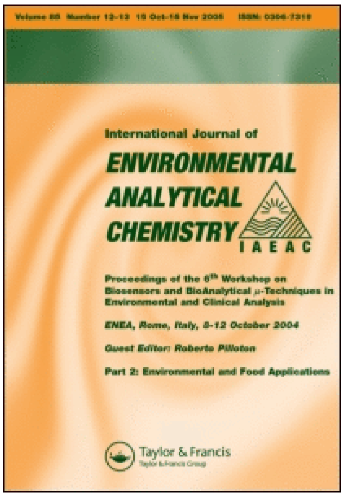

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Retention of pesticides and nutrients in a vegetated buffer root zone compared to soil with low biological activity

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A laboratory study has been conducted to examine the retention processes of nitrogen and pesticides through vegetated buffer zones compared to bare soil. Soil columns with low biological activity and vegetation columns with normal biological activity were tested. Pesticides frequently used in vegetable production (namely aclonifen, azinphos-methyl, chlorpropham, diazinon, dimethoate, fluazinam, iprodione, linuron, metalaxyl, metamitron, metribuzin and propachlor) equal to 1/50 to 1/5 part of recommended doses, and nutrients equal to 1, 5 and 20 mg N/L and 0.2 mg P/L, were added. The pesticide retention was more than 60% for all pesticides, except dimethoate, with a retention of about 30% in columns with low microbial activity. Biological transformation and plant uptake were important for removal of nitrogen and organic matter. Nitrogen retention was high (over 90%) in vegetation columns. Plant uptake and phosphorus content in soil were important for phosphorus retention.

Keywords: Buffer zones; Pesticides; Nutrients; Chemical transformation; Biological degradation

1. Introduction

The risk of pesticide pollution to watercourses has increased with the intensification of agriculture. Vegetable production has a high input of pesticides and nutrients. Monitoring shows that vegetable production has a high risk of leaking of pesticides and nutrients to groundwater and surface water [1]. Pesticides and nutrients are sorbed and degraded in the environment due to retention processes such as sedimentation, adsorption to particles and organic matter, biological degradation, dilution, evaporation and photo-degradation. The retention rates are influenced by a number of environmental factors such as climate and soil condition. Vegetated buffer zones (BZs) adjacent to watercourses can be effective filters for retention of nutrients. According to the Norwegian Plan of Action for Pesticide Reduction (1998–2002) [2], BZs are one recommended measure. Knowledge about retention processes through BZs and their efficiency of pesticide removal is, however, scarce. Recently completed studies show a high retention of particle-bound pesticides through BZs [3, 4].

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Sedimentation of pesticides (glyphosate, fenpropimorph and propiconazole) was the dominating retention process in those studies. The same results have been shown in several studies [5–8], regarding particle-bound nutrients (phosphorus). Other pesticides have, however, a low sorption to soil and relatively high water solubility (e.g. herbicides used in vegetable production). One question to ask is whether pesticides and nutrients (for instance nitrogen) with a lower affinity to soil have the same ability to remain in the BZ. It is likely that the predominant retention process will be different in the root zone of a BZ compared to bare soil with a lower microbial activity and organic matter content.

The aim of the experiment was to quantify the difference in pesticide and nutrient retention in vegetated buffer zones with a normal biological activity compared to bare soil with a low biological activity. All the compounds used in the experiment are used in vegetable production (potatoes, carrots, cabbage, onions). Different retention processes possibly occurring in the experiments are also discussed.

2. Experimental

2.1. *Experimental design*

Selected pesticides and nutrients were added on top of five glass columns (diameter 28 cm, height approx. 50 cm) to create vertical flow in a laboratory scale study. The flow was partly unsaturated and partly saturated, where the lowest part of the column (17 cm) was saturated, and the upper 7–12 cm was unsaturated. The columns consisted of (1) two 'sterilized' columns to produce soil with a low microbial activity (soil columns), (2) two columns with soil and vegetation and a normal microbial activity (vegetation columns) and (3) one reference column to test possible sorption of pesticides and nutrients to the equipment (table 1). Aluminium covers sterilized every day were placed on top of the soil columns to maintain a low microbial activity. All equipment and solutions used in the soil columns were sterilized. The predominant plant species in the vegetation columns were: *Elymus repens* (L.) Desv. Ex. Nevski, *Chenopódium album* L., *Rubus idaéus* L. and *Urticáceae dioíca* L. A plant-light simulated daylight for 16 h per day. The soil type was loamy sand with 5.4% clay, 9.7% silt and 84.9% sand. The chemical characteristics of the soil are listed in table 2. The cation-exchange capacity was about 10 meq/100 g soil. Plant-available phosphorus (P-Al) was quite high, 20 mg/100 g soil. The C/N ratio was 13.

To obtain a good distribution of solution added to the soil and vegetation columns, a glass-pellet distribution layer of 1–2 cm depth was placed at the top of the columns. All columns had a plastic net under the soil and a layer of Perlite to ensure good distribution of water through the columns and to prevent siltation of sediments at the outlet of the columns. Perlite consists of expanded vitreous, volcanic rock containing about 65% SiO_2, with little or no sorption to pesticides [9, 10]. All columns were covered by black plastic to prevent light penetration into the solution/soil/root zone.

The selected pesticides were herbicides, fungicides and insecticides frequently used in vegetable production: aclonifen, azinphos-methyl, chlorpropham, diazinon, dimethoate, fluazinam, iprodione, linuron, metalaxyl, metamitron, metribuzin and propachlor. The pesticide concentration added to the columns was equal to 1/50 (Experiment 1) and 1/5-part (Experiment 2) of each recommended pesticide

Table 1. Design of the experiment.

Experiment 1: Low pesticide concentration (1/50 part of recommended application)			Experiment 2: High pesticide concentration (1/5 part of recommended application)		
Low microbial activity obtained by γ-radiation	High microbial activity	Reference column with pesticide/nutrient solution	Low microbial activity obtained by adding NaN_3[a]	High microbial activity	Reference column with pesticide/nutrient solution
1, 5 and 20 mg N/L and 0.2 mg P/L applied[b]	1, 5 and 20 mg N/L and 0.2 mg P/L applied	1, 5 and 20 mg N/L and 0.2 mg P/L applied	1, 5 and 20 mg N/L and 0.2 mg P/L applied	1, 5 and 20 mg N/L and 0.2 mg P/L applied	1, 5 and 20 mg N/L and 0.2 mg P/L applied

[a] The method for sterilization was changed in Experiment 2 due to the fairly high amount of bacteria after finishing Experiment 1. 0.5 g NaN_3/L pesticide/nutrient solution was added;
[b] nitrogen was added as KNO_3, phosphorus as $KH_2PO_4/Na_2PO_4 \cdot 2H_2O$ (pH = approx. 7).

Table 2. Chemical characteristics of the soil used in the laboratory experiment.[a]

pH	Phosphorus	Aluminium	Iron	Nitrogen	Total carbon	Organic carbon
6.0	1220	6940	17 200	1600	32 100	21 000

[a] All values are measured in $mg\,kg^{-1}$ dry soil.

Table 3. Concentration of pesticide added equal to 1/50 (low concentration, Experiment 1) and 1/5 part (high concentration, Experiment 2) of recommended application doses and chemical characteristics of the pesticides.[a]

Pesticide	Low conc. ($\mu g\,L^{-1}$)	High conc. ($\mu g\,L^{-1}$)	Type	Log K_{oc} ($kg\,L^{-1}$)[b]	Log K_{oc} ($kg\,L^{-1}$)[c]	DT_{50} (days)	Water solubility ($mg\,L^{-1}$)
Aclonifen	2	20	H	5.51	3.93	50	1
Chlorpropham	5	50	H	n.a.	2.70	30	89
Linuron	0.5	5	H	4.23	2.70	82	75
Metamitron	3	30	H	n.a.	0.85	30	1700
Metribuzin	0.2	2	H	3.13	1.72	47	1000
Propachlor	5.5	55	H	n.a.	1.90	12	613
Fluazinam	0.2	2	F	n.a.		33–62	2
Iprodione	1	10	F	4.46	2.82	50	14
Metalaxyl	1	10	F	2.96	2.23	80	8400
Azinphos-methyl	1	10	I	n.a.	2.97	10	28
Diazinon	1.5	15	I	n.a.	3.15	27	60
Dimethoate	0.5	5	I	n.a.	1.30	3	3900

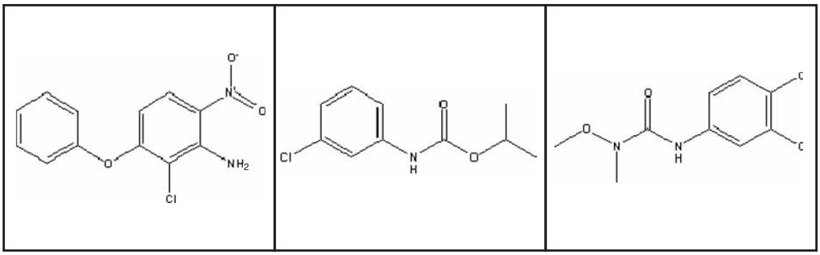
[a] H: herbicides; F: fungicides; I: insecticides; K_d: ratio between the content of pesticide adsorbed to the soil and the mass concentration of pesticide in the aqueous solution; K_{oc}: K_d adjusted for organic carbon in the soil; DT_{50}: half-life time; n.a.: not analysed; [b] local value [10]; [c] literature value, empty field: not found in literature.

Table 4. Literature data on biological degradation and ecotoxicology (PNEC) [11, 12].

Pesticide	Aerobic degradation	Anaerobic degradation	PNEC ($\mu g\,L^{-1}$)	Phytotoxicity	Root adsorption
Aclonifen				Yes	
Chlorpropham		Mean-moderate	10		Yes
Linuron	Low		0.70		Yes
Metamitron			11		
Metribuzin	Low	Very low	2.2	Yes	Yes
Propachlor	Yes (co-metabolism)		2.9		
Fluazinam			0.55		
Iprodione	Low	Moderate	2.5		Yes
Metalaxyl	Moderate	Low	280		No
Azinphos-methyl			0.01		No
Diazinon	Moderate		0.01	No	Yes
Dimethoate	High		0.20	No	

Empty field: no data. PNEC: predicted no effect concentration, or measured effect concentration (algae or crustacean) times safety factor (usually 10 or 100).

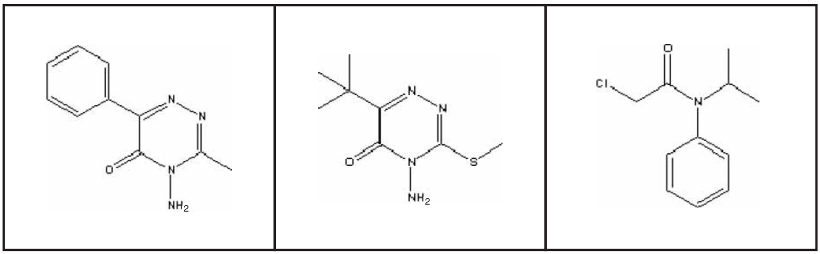
application doses. The lowest concentration added was 10 times the level of detection (table 3). The accumulated effect of all the pesticides in the solution is expected to be more phytotoxic to the vegetation in the columns than each pesticide separately. Table 3 also shows the chemical characteristics of the pesticides. K_{oc} values (local value) are according to [10], where the same soil as in this experiment has been tested. Table 4 shows literature data on biological degradation and ecotoxicology for the pesticides used in the experiments [11, 12]. Figure 1 shows the chemical formulae of the pesticides [11]. The hydraulic load applied was 28 mm day^{-1} (2 L day^{-1}). There was no pesticide/nutrient application during the weekends. The experimental duration for each combination of pesticide- and nutrient level was 2 weeks, with a total



Aclonifen, herbicide, $C_{12}H_9ClN_2O_3$

Chlorprofam, herbicide, plant grow. regulator, $C_{10}H_{12}ClNO_2$

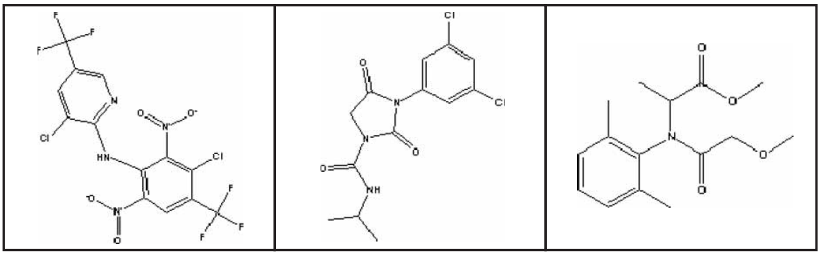
Linuron, herbicide, $C_9H_{10}Cl_2N_2O_2$



Metamitron, herbicide, $C_{10}H_{10}N_4O$

Metribuzin, herbicide, $C_8H_{14}N_4OS$

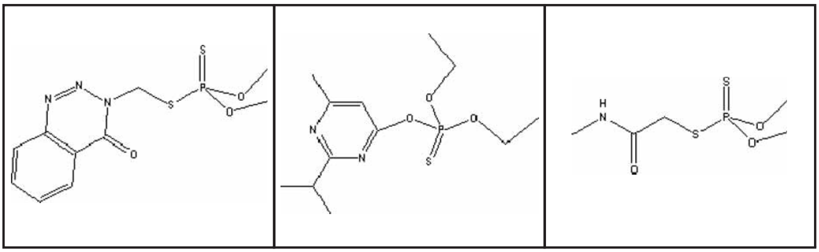
Propachlor, herbicide, $C_{11}H_{14}ClNO$



Fluazinam, fungicide, $C_{13}H_4Cl_2F_6N_4O_4$

Iprodione, fungicide, $C_{13}H_{13}Cl_2N_3O_3$

Metalaxyl, fungicide, $C_{15}H_{21}NO_4$

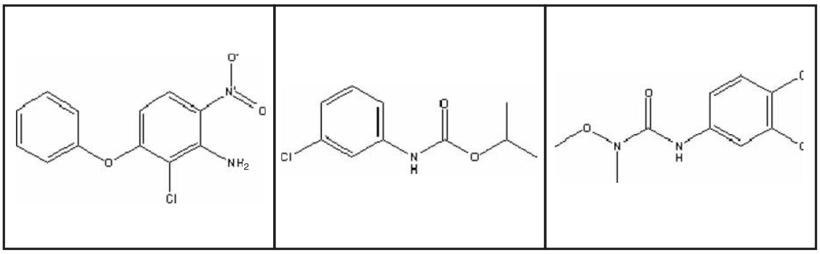


Azinphos-methyl, insecticide, $C_{10}H_{12}N_3O_3\,PS_2$

Diazinon, insecticide, acaricide, $C_{12}H_{21}N_2O_3\,PS$

Dimethoate, insecticide, acaricide, $C_5H_{12}NO_3\,PS_2$

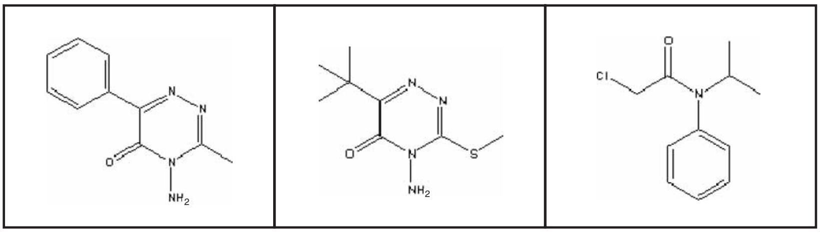
Figure 1. Chemical formulae for pesticides used in the experiments [11].



Aclonifen, herbicide,
$C_{12}H_9ClN_2O_3$

Chlorprofam, herbicide,
plant gr. regulator,
$C_{10}H_{12}ClNO_2$

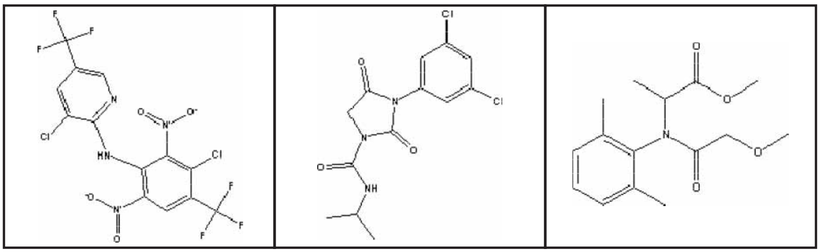
Linuron, herbicide,
$C_9H_{10}Cl_2N_2O_2$



Metamitron, herbicide,
$C_{10}H_{10}N_4O$

Metribuzin, herbicide,
$C_8H_{14}N_4OS$

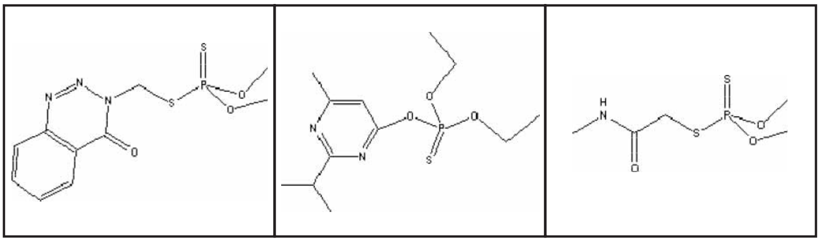
Propachlor, herbicide,
$C_{11}H_{14}ClNO$



Fluazinam, fungicide,
$C_{13}H_4Cl_2F_6N_4O_4$

Iprodione, fungicide,
$C_{13}H_{13}Cl_2N_3O_3$

Metalaxyl, fungicide,
$C_{15}H_{21}NO_4$



Azinphos-methyl, insecticide,
$C_{10}H_{12}N_3O_3\ PS_2$

Diazinon, insecticide, acaricide,
$C_{12}H_{21}N_2O_3\ PS$

Dimethoate, insecticide, acaricide,
$C_5H_{12}NO_3PS_2$

Figure 1. Continued.

of 6 weeks for each experiment. All column filters were replaced with new ones after finishing Experiment 1. Four or five water samples per column per combination were taken from the outlet of the column for pesticide and nutrient analyses. Background values of pesticides and nutrients were analysed in water samples collected before starting the experiments. Residues of pesticides in the soil after finishing the experiments were also analysed.

After finishing Experiments 1 and 2, hydraulic conductivity was tested in the columns. The saturated hydraulic conductivity was measured by keeping a constant water level (gradient) in the columns by using tap water and measuring the water flow given by a accurate piston pump (Antrieb gamma/4-W no. 9086281 45). Breakthrough curves were established by using tap water with elevated NaCl (named as tracer test later) concentrations and continuous measurements of electrical conductivity in the outflow.

2.2. *Analytical laboratory methods and calculation of retention*

Water samples from the outflow of the columns were analysed for total amount of bacteria, selected pesticides, total nitrogen (TotN), total phosphorus (TotP) and total organic carbon (TOC). Selected pesticides were analysed by gas chromatography (GC-MULTI M03) [13]. Chemical and biological parameters were analysed according to Norwegian standards (NS), European Norms (EN) and the ISO-systems for water analysis. Retention, R (%), was calculated according to the following equation as an expression for change in amount of pesticide/nutrient through the columns:

$$R = [(Q_{\text{in}}C_{\text{in}} - Q_{\text{out}}C_{\text{out}})/Q_{\text{in}}C_{\text{in}}] \times 100, \tag{1}$$

where Q_{in} were Q_{out} are the average amounts of water into and out of the columns with normal (vegetation columns) and low biological activity (soil columns), and C_{in} and C_{out} were the concentration in the reference column and average concentration out of the columns with low and normal biological activity. Retention values were corrected for the measured recovery of pesticides and background values of pesticides and nutrients in the outflow of the columns prior to the experiments.

3. Results and discussion

The hydraulic conductivity in the columns with low microbial activity is approximately one order of magnitude lower than in the columns that are biologically active (table 5). The mean detention time of the NaCl tracer was 335 and 98 min in the soil columns (low microbial activity), and 53 and 32 min in the vegetation columns, in Experiment 1 and 2, respectively. Faster transport in the vegetation columns can be caused by the channelling effect of the plant roots or can result if the soils in the columns with a low microbial activity were packed harder and thus were more compacted than the natural soil. In Experiment 1, the radiation of the soil destroyed much of the organic material, which can also have a reducing effect on the porosity.

Table 5. Measured hydraulic conductivity ($\mathrm{m\,s^{-1}}$) in the columns.

Column	Experiment 1	Experiment 2
Low microbial activity	$3.6–4.2\,\mathrm{e}^{-05}$	$5.2–8.0\,\mathrm{e}^{-05}$
Normal microbial activity	$1.5–5.8\,\mathrm{e}^{-04}$	$2.2–2.4\,\mathrm{e}^{-04}$

3.1. *Low concentration of pesticides added (Experiment 1)*

Only five of the pesticides were found in the outflow of the soil columns in Experiment 1 (low microbial activity); metamitron, metalaxyl, diazinon, dimethoate and metribuzin (figure 2, top). Metribuzin arrived simultaneously with the tracer (at pore volume 4.5), and metalaxyl and dimethoate showed a greater degree of retardation. The tracer also had significant retardation in the columns. In the biologically active columns with vegetation, the pesticides were detected in the outflow both earlier and at higher concentrations than in columns with low microbial activity (figure 2, bottom). Pesticides with the highest concentration in the outflow water were linuron, metribuzin, fluazinam, iprodione and dimethoate. The number of compounds detected also increased in biologically active columns.

There was no significant difference in removal efficiency for the three levels of added nitrogen to the biological active vegetation columns, indicating a high metabolic activity through the root zone. The removal efficiency of phosphorus was also high. There was a net loss of organic matter, nitrogen and phosphorus from the soil columns with a low microbial activity (table 6).

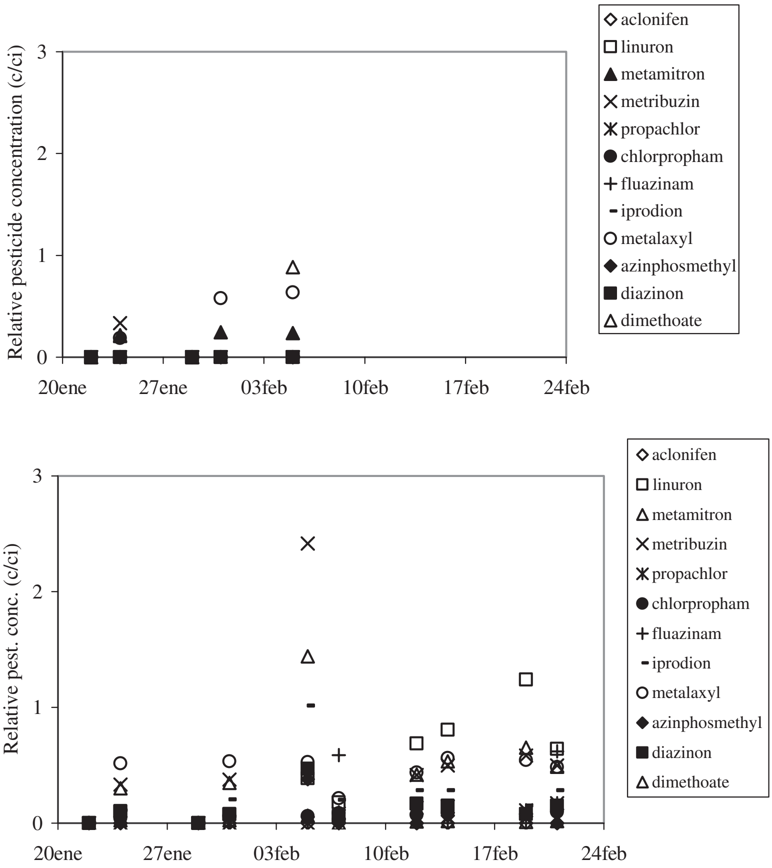


Figure 2. Analyses of pesticides in the outflow (relative pesticide concentration output/input) from columns in experiment 1 with low (top) and normal (bottom) microbial activity.

Table 6. Retention (%) of pesticides and nutrients for Experiment 1 (low concentration of pesticides added) and Experiment 2 (high concentration of pesticides added).

Pesticide concentrations	Low		High		Average	
Biological activity	Low	Normal	Low	Normal	Low	Normal
Aclonifen	100	100	100	93	100	93
Chlorpropham	100	97	99	87	99	89
Linuron	100	80	89	84	90	85
Metamitron	88	83	78	66	75	64
Metribuzin	98	75	54	62	61	66
Propachlor	100	93	85	71	87	75
Fluazinam	100	90	97	94	98	93
Iprodione	100	85	100	76	100	78
Metalaxyl	77	78	100	56	97	63
Azinphos-methyl	100	98	100	89	100	91
Diazinon	98	93	94	81	95	84
Dimethoate	84	83	28	63	36	68
TOC	n.e.[a]	61	n.e.	30	n.e.	44
TotN	n.e.	100	n.e.	75	n.e.	93
TotP	n.e.	100	n.e.	n.e.	n.e.	n.e.

[a] n.e.: negative effect.

3.2. *High concentration of pesticides added (Experiment 2)*

The number of pesticides detected increased from Experiment 1 to Experiment 2 in soil columns with a low microbial activity (figure 3, top). The concentration of the pesticides in outflow water was, as expected, also higher. If we compare Experiments 1 and 2 and vegetation columns with normal microbial activity, both the numbers of pesticides detected and the concentration in outflow water were quite similar (figure 3, bottom). The time for detection of pesticides was, however, at an earlier stage in Experiment 2. This shows that BZs have a fairly high capacity to retain pesticides. The tracer test after Experiment 2 was unsuccessful, possible due to preferential flow problems.

There were no significant differences in removal efficiency in Experiment 2 regarding nitrogen concentration added in the biologically active vegetation columns. The removal efficiency was, however, lower than in Experiment 1, possibly due to the phytotoxic effect of vegetation by adding a high concentration of herbicides (in particular). There was a visual withering of vegetation during Experiment 2. There was also a net loss of organic matter, nitrogen and phosphorus from the soil columns with a low microbial activity in this experiment (table 6).

3.3. *Average removal efficiency*

A parametric, multiple-variance test (MANOVA-test, test limit $p < 0.05$) was performed to test the differences in retention efficiency between the concentration of pesticides added (Experiments 1 and 2) and the biological activity (low and normal activity). The results show that a low pesticide concentration (Experiment 1) resulted in about 6% higher retention efficiency than a high pesticide concentration (Experiment 2). Also, soil columns with a low microbial activity had a higher retention efficiency than vegetation columns with a normal activity. This may be due to a higher

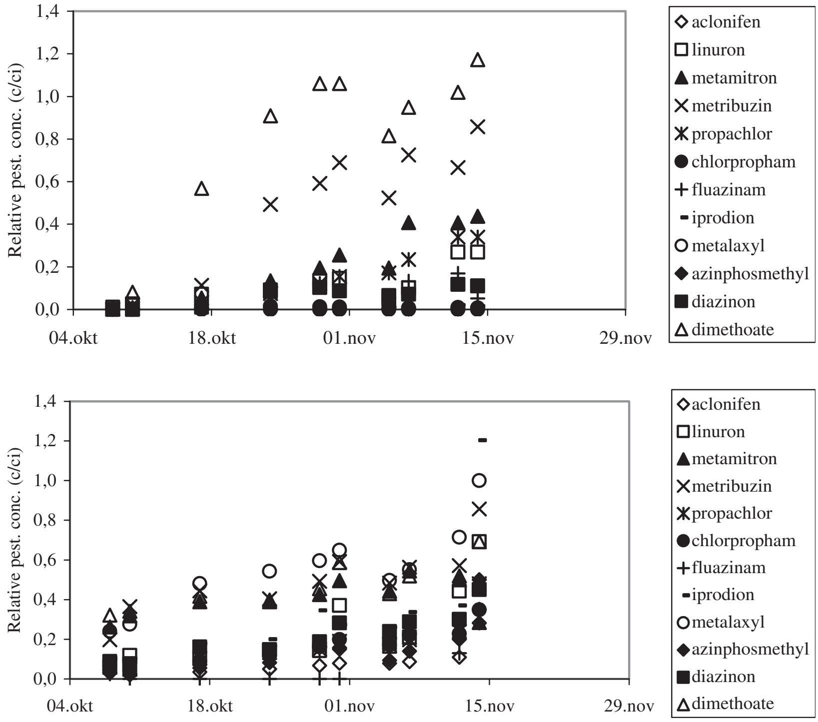


Figure 3. Analyses of pesticides in the outflow (relative pesticide concentration output/input) from columns in experiment 2 with low (top) and normal (bottom) microbial activity.

retention time in soil columns with a low microbial activity and high availability of organic matter due to the sterilization methods for the columns (in Experiment 1).

The pesticide retention was high; more than 60% for all pesticides, except dimethoate, which had a retention of 30% in soil columns with a low microbial activity (table 6). Dimethoate has a low soil adsorption, high water solubility and high biological removal under aerobic conditions (tables 3 and 4). This indicates that the columns with vegetation have higher oxygen levels than columns without vegetation. The average nitrogen retention is high in vegetation columns; over 90%, compared to soil columns which have a negative retention efficiency. These results indicate a high microbial activity in the vegetation columns. The average retention efficiency for phosphorus is negative, possibly due to the very high amount of plant-available phosphorus (P-Al) in the soil. The adsorption capacity is then expected to be low. There was a visual withering of vegetation during the experiment with high pesticide concentrations added. There was also a negative retention of phosphorus in this experiment. In contrast, there was a high phosphorus retention in vegetation columns with a low concentration of pesticide added, indicating that plant uptake was a significant phosphorus-retention process.

3.4. *Residues of pesticides in vegetation and soil*

Figure 4 shows nearly complete accumulation of aclonifen and iprodion, and significant accumulation for the other compounds (except propachlor and metamiron) in the

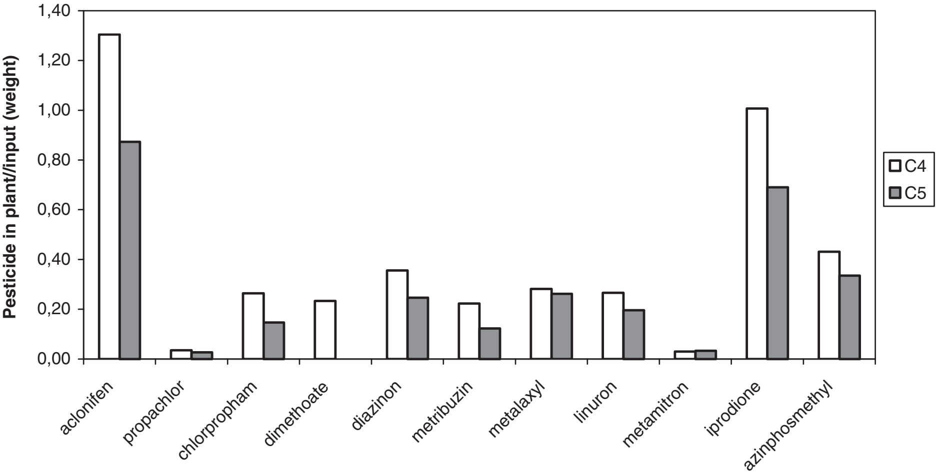


Figure 4. Ratio of pesticide input and residues in plants in two vegetation columns (C4 and C5).

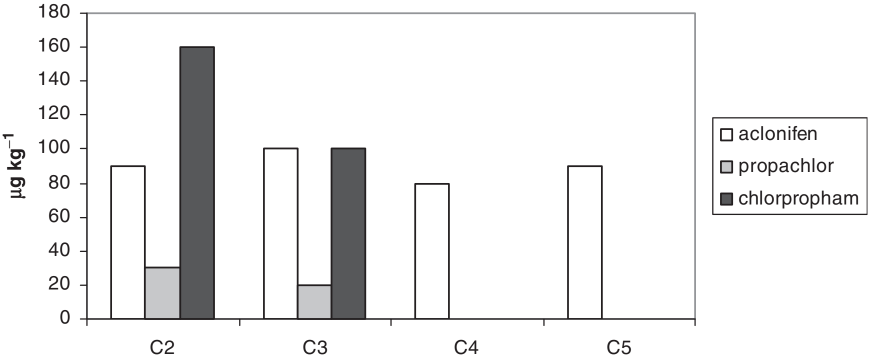


Figure 5. Pesticide residues in soil columns with low (C2 and C3) and normal (C4 and C5) biological activity.

vegetation in the columns with normal biological activity. According to table 4, metalaxyl and azinphosmethyl are not taken up directly from the soil by the plants. Our results show the opposite.

Aclonifen was found in nearly equal amounts in soil in all columns (figure 5), which corresponded to the highest K_d value for pesticides used in the experiments (table 3). Columns with a normal biological activity had no residues from other compounds, while columns with a low activity had residues of propachlor and chlorpropham.

According to [14–19], metribuzin and metamitron are showing relatively high residues in soils, although studies show variable penetration depths depending on TOC, CEC, content of iron silicates, colloidal clay and humic acid particles. pK_a is only given for metribuzin (1.0), so we can assume that the other compounds are not ionic at the pH values in the experiment (soil pH varied from 6.3 to 6.7). Therefore, we assume that metribuzin is anionic, water-soluble and fairly leachable. This can be seen from the high relative concentrations of metribuzin in the outflow of all columns (figure 3). Linuron penetrates to greater depths in soils, although sorption increases with soil moisture and exogenous organic matter, but not with temperature [20–24]. Sorption of linuron to aquatic macrophytes could be described by the equation $K_d = 3.20 - 0.65 \log S$, where S = water solubility [25]. A maximum of 1% of the applied dose of linuron was found in macrophytes in an outdoor ditch experiment [26]. Metribuzin was found to be highly toxic to freshwater macrophytes, with a median plant EC_{50} of $31\,\mu g\,L^{-1}$ [27].

The soil samples represent only a small volume of the upper unsaturated 5–20 cm of the columns and are thus not necessarily representative of the total pesticide content in the soil. This may explain the lower residue levels of pesticides in the soil compared to those in the literature. There is also the question of whether the extraction of pesticides from soil is complete with the available analytical methods. Variations in analytical methods may also explain the lower levels of residues in soil than expected.

4. Conclusions

- The hydraulic conductivity was one order of magnitude lower in the soil columns with a low biological activity compared to columns with vegetation.
- Nitrogen retention was high in vegetation columns with normal biological activity and negative in soil columns with a low biological activity.
- Phosphorus retention was high in vegetation columns with normal biological activity when the pesticide concentration was low, but negative in vegetation columns with a high pesticide concentration. The phosphorus retention was also negative in soil columns.
- The experiments indicate that nitrogen retention is more dependent on microbiological factors, and that phosphorus retention is negative when the phosphorus content in the soil is high, and also when vegetation is removed.
- Pesticide retention was high in both columns with low and normal biological activity.
- The retention efficiency was more than 60% for all pesticides, except for dimethoate, which had a retention efficiency of about 30% (low microbial activity).
- A low concentration of pesticides showed a higher retention efficiency than a high concentration of pesticides added.
- A low microbial activity in soil columns had a higher pesticide retention than normal activity in vegetation columns.
- Two compounds, propachlor and metamitron, were not taken up in the plants.
- Two compounds, aclonifen and iprodion, were completely taken up in the plants.
- The other compounds showed a significant plant uptake.
- Residues of aclonifen were found in all soil samples, and propachlor and chlorpropham were found in soil samples from columns with a low biological activity.

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