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Investigation of various physicochemical and environmental parameter influence on pesticide sorption to ditch bed substratum by means of experimental design.

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Abstract

Diffuse pollution by pesticide applied in rural catchments may contribute to alter water quality. Besides actions relative to the way the substances are introduced into the environment, it is also possible to limit the contamination by interfering on their transfer pathways from fields to the main river network. Especially, interface areas such as buffer strips or small ditches may play a major part in pesticide diffuse pollution decrease. In ditches a great variety of materials may act as sorbents for organic contaminants: grass, leaves, wood debris or sediments. In this study, laboratory experiments were designed to determine sorption characteristics for three herbicides with different physicochemical properties on sediment and leaves in decay commonly found in agricultural ditches. Sorption capacities were assessed for the herbicides isoproturon, diuron and diflufenican. Experimental design was carried out to investigate the effects of five parameters on herbicide sorption on sediment and dead leaves. These parameters have been chosen according to parallel field experiment needs. Thus, the influence of initial sorbent moisture, herbicide form, i.e. active substance or commercial formulation, water quality (tap or natural ditch water), bromide ions (used as conservative tracers) and solid/liquid ratio have been tested. Within the parameters investigated, pesticide formulation and

solid/liquid ratio were the most important parameters affecting pesticide sorption on both ditch materials.

Keywords : pesticides, adsorption, ditch materials, leaves, sediment, experimental design

1. Introduction

Agricultural non-point pollution originates from land areas which may intermittently contribute to the transport of pesticides to surface water by runoff, drainage, leaching, aerial deposition or drift. Many methods and levels of actions can be developed to reduce this transfer. For example, new or improved agricultural practices can be set up, such as choosing the best application period, controlling toxic substance impacts, or using non-chemical practices (Baker et al., 1995). However once pesticides are applied, diffuse losses cannot be totally prevented, due to the uncontrollable soil and climate characteristics involved in diffuse pollution. Pesticides leaving a plot in runoff may pass through different landscape components before reaching rivers, including other fields, ditches, brooks and vegetative or wooded buffer zones. Only few in situ experiments have been carried out concerning the role played by farm ditches in reducing water contamination by pesticides (Williams et al., 1999; Moore et al., 2001; Cooper et al., 2004). Despite the heterogeneity of the situations encountered, field experimentations in ephemeral ditches with rapid water flows revealed that pesticide retention was linked to the substratum nature, the hydrodynamic conditions, and also to the contaminant physicochemical properties. Therefore it is essential to better characterise pesticide sorption ability to typical sorbents found in such buffer zones by means of well controlled trials. Laboratory studies were then conducted to determine the sorption behaviour of three herbicides with different physicochemical properties (isoproturon: 3-(4-isopropylphenyl)-1,1-dimethylurea,

diuron: 3-(3,4-dichlorophenyl)-1,1-dimethyurea, and diflufenican: 2'4'-difluoro-2-(α , α , α -trifluoro-m-tolyloxy)nicotinilide) on bed sediment and dead leaves which are common substratum in French ditches (Margoum et al., 2001). In the present paper, herbicide sorption was studied as a function of different influencing parameters including the form of the sorbates, the sorbents and the water characteristics. The influence of some of these factors has already been studied with other sorbents or pesticides (Lickfeldt and Branham, 1995; Beck and Jones, 1996; Gaillardon, 1996; Walker and Jurado-Exposito, 1998; Chaplain et al., 2001). However, conclusions were in contradiction. On the one hand, Lickfeldt and Branham (1995) investigated the adsorption capacity of several non-ionic compounds on leaves and thatch, and concluded that these chemicals were more adsorbed when formulated or with the presence of dissolved organic matter. On the other hand, Beck and Jones (1996) observed non significant influence of dissolved organic matter on atrazine and isoproturon sorption on soils. Gaillardon (1996) showed that soil moisture enhanced sorption speed and increased final sorption for diuron on soils.

The aim of the present study was to complement preliminary field experimental results concerning the role played by ephemeral ditch materials on pesticide retention (Margoum et al., 2003), by studying the influence of five different but not exhaustive parameters on sorption. Then, we qualitatively estimated to which extent (a) initial sorbent moisture, since the hydration status may modify its hydrophobic properties ; (b) formulated herbicide substances used in field experiments as presented before ; (c) water quality, and especially organic matter content (Caron et al., 1985; Chiou et al., 1986; Flores-Cespedes et al., 2002) ; (d) bromide ions used as conservative tracers in experiments concerning pesticide transfer studies (Allaire-Leung et al., 1999; Schoen et al., 1999), and (e) solid/liquid ratio (Boesten and Van Der Pas, 1988; Walker and Jurado-Exposito, 1998) could play a significant role in herbicide sorption on typical ditch sorbents. A fractional factorial experimental design and columns of contrast coefficients calculation were used for rapid assessment of the effects of the five studied parameters.

2. Experimental

2.1 Chemicals and reagents

Three chemicals were used in this research. Isoproturon (99%), diuron (97.5%) and diflufenican (99.5%) were of analytical grade supplied by Cluzeau Info Labo (Sainte Foy la Grande, France) (Table 1). These herbicides are commonly used in agricultural catchments for weed control. The commercial formulations were Zodiac TX that contained isoproturon (500 g L⁻¹) and diflufenican (100 g L⁻¹), and Séduron (diuron at 500 g L⁻¹) both from Bayer Crop Science.

Table 1. Properties of the studied herbicides (Tomlin, 2000; INRA, 2005)

Characteristics	Isoproturon (IPU)	Diuron (DIU)	Diflufenican (DFF)
Solubility in water (mg L ⁻¹)	65 (22 °C)	36.4 (25 °C)	0.05 (25 °C)
K _{oc} (L kg ⁻¹)	36-241	480	1622-2369
log K _{ow}	2.5	2.85	4.9
Half life (day)	6-28	90-180	105-210

All organic solvents used for standard solution, extraction or chromatographic analysis were pesticide or gradient HPLC grade (SDS, Peypin, France).

2.2 Sorbents

Sorbents were chosen in accordance with agricultural ditches previously studied (Margoum et al., 2003), i.e. poor organic matter sediments and leaves in decay to have a large range of sorbent

properties. Ditch bed sediment was collected in Roujan, southern France (Louchart et al., 2001). The sediment (clay: 305 g kg⁻¹; silt: 483 g kg⁻¹; sand: 212 g kg⁻¹) was air-dried and passed through a 2-mm sieve before any experimental use. Dead leaves from oak and chestnut trees were collected in autumn, dried at ambient temperature, cut in fine pieces (about 5 mm diameter) and homogeneised. All sorbents were analysed for total and organic C and N content (Table 2) using a CHN autoanalyser (Carlo ErbaEA1108, Fisons Instrument). Moisture content was estimated after drying at 105 ± 5 °C for at least 24 hours.

Table 2

Characteristics of the sorbents.

Sorbent	Organic C (g kg ⁻¹)	Total N (g kg ⁻¹)	C/N	Moisture content (n=3)*	
					(%)
Sediment	9.8	1.0	9.8	Dried	5 ± 2
				Pre-soaked	39 ± 5
Dead leaves	482	9.3	52	Dried	10 ± 2
				Pre-soaked	82 ± 6

* : number of replicate measurements

2.3 Sorption experiments

All sorption experiments were conducted with the three herbicides using a modified batch technique to ensure equilibration. As the contact time between pesticide substances and bed sorbents may be limited in natural ditches, due to hydrological processes, we carried out laboratory controlled experiments under gentle shaking (50 rpm). Preliminary kinetic studies

indicated that sorption for the three herbicides on both sorbents reached apparent equilibrium within less than 24 h (results not shown).

Since the two sorbents had very different bulk densities, the initial masses were adjusted to ensure a suitable contact between the sorbent and the solution. Therefore, 50 g of dry sediment or 8 g of dead leaves were added to 250 mL of water in a 500 mL glass Petri dish. Initial pesticide concentrations in solution were chosen so as to get a compromise between observations in natural drainage ditches and analytical limits ($4 \mu\text{g L}^{-1}$ for DFF and $20 \mu\text{g L}^{-1}$ for IPU and DIU).

To test the effect of initial sorbent moisture on herbicide sorption, dry sediment or leaves were weighted and pre-soaked one night to obtain submerged conditions. Formulation solutions were made from commercial Zodiac TX and Séduron with the appropriate dilution in tap or natural ditch water and then added to the sorbents. Control samples without sorbent, or blanks with no herbicide were run and showed nor herbicide degradation or sorption to the Petri dishes, nor herbicide release from sorbents. Therefore, pre-contaminated water was added to the sorbents and left on orbital shaker at room temperature for 24 hours.

Water supernatants were then sampled and transferred into separatory funnel for liquid-liquid extraction with methylene chloride. PCB153 and linuron were added as internal control standards. Organic extracts were dried with sodium sulphate and separated in two fractions for gas (GC) and liquid (HPLC) chromatography. After evaporation, samples were recovered in isooctane for GC analysis and in water/acetonitrile (80/20–V/V) for HPLC analysis.

Analyses of DFF and PCB 153 were performed on a VARIAN 3400 gas chromatograph equipped with an electron capture detector and a capillary column DB1701 (30 m x 0.32 mm x 0.25 μm) from JW Scientific. The temperature program was as follows: initial temperature 40 °C raised at 20 °Cmin⁻¹ up to 200 °C, then raised at 10 °Cmin⁻¹ up to 280 °C. Injector and detector temperatures were respectively 250 °C and 300 °C. Under these conditions, the retention times

were 10.9 min for PCB 153 and 11.6 min for DFF. The detection limit was $0.02 \mu\text{g L}^{-1}$ for both compounds, and PCB 153 recovery rate was $89 \pm 10\%$ ($n=55$).

Analyses of IPU, DIU and linuron were performed on a WATERS Alliance 2690 liquid chromatograph equipped with a photodiode array detector (Alliance 996) and a SymetryShield RP8 cartridge (4.6 x 250 mm – 5 μm , Waters). The mobile phase was water/acetonitrile (55/45) at a rate of 1 mL min^{-1} . All compounds were detected at 245 nm. The retention times were 8.6, 10.4 and 16.7 min for IPU, DIU and linuron respectively. The detection limit was $0.1 \mu\text{g L}^{-1}$ for the three compounds and corresponding recovery rate for linuron was $101 \pm 6\%$ ($n=55$).

The herbicide concentration in supernatant solution (in $\mu\text{g L}^{-1}$) was directly measured. The amount of sorbed herbicide, (in $\mu\text{g kg}^{-1}$ sorbent) was calculated from the difference of herbicide concentration in solution before and after the sorption experiment.

2.4 Factorial design

Firstly, a two level factorial experimental design was applied to investigate the influence of two main parameters on herbicide sorption. Each parameter is tested with a low and a high level presented in brackets, i.e. initial sorbent moisture (dried or pre-soaked), which depends on hydrological conditions in ephemeral ditches, and pesticide form (pure active substance used for laboratory trials or formulated product for field experiments).

Secondly, the following parameters have been considered: concentration of bromide (0-100 mgL^{-1} , which corresponds to field experiment concentration level), solid/liquid ratio related to the variation of initial sorbent mass (1/8 or 1/5 for sediment and 1/100 or 1/35 for leaves), water quality (tap water, that is usually used for laboratory experiments or natural ditch water which contains organic matter) and herbicide form as it had been assumed as influential in the first experimental design.

The evaluation of the effect of the investigated parameters basically consists of a two-level full factorial design where $2^{\text{number of variables}}$ experiments have to be carried out. The main advantage of such a model is that cross-effects between variables can be easily evaluated (Lundstedt et al., 1998; Goupy, 2001). For our study, it was reasonable to assume that the influence of the interactions of third order were negligible. Then, we applied a fractional factorial design $2^{\text{number of variables}-1}$, with two blocks in order to replicate each experiment. A total of 8 and 16 runs was then obtained for the first and the second series of experiments respectively. The order of execution was randomly selected (Table 3). The percentage of pesticide adsorbed on sorbent was chosen as final criterion for the response value.

Table 3

Experimental matrixes with two parameters (first) and four parameters (second). – and + correspond respectively to low and high level for each parameter

First experimental matrix			Second experimental matrix				
Exp. number	Pesticide form	Sorbent moisture	Exp. number	Bromide concentration	Solid/liquid ratio	Pesticide form	Water quality
1	-	-	1 and 9	-	-	-	-
2	+	-	2 and 10	+	-	-	+
3	-	+	3 and 11	-	+	-	+
4	+	+	4 and 12	+	+	-	-
5	-	-	5 and 13	-	-	+	+
6	+	-	6 and 14	+	-	+	-
7	-	+	7 and 15	-	+	+	-
8	+	+	8 and 16	+	+	+	+

3 – Results and discussion

Mean values of duplicates for sorption capacity, expressed as the percentage of herbicide adsorbed on sorbent, are reported in Table 4 for the two series of experiments. Whatever the experimental conditions, our results showed that diflufenican is the more adsorbed compound, which is consistent with pesticide physicochemical characteristics (Table 1) and that sorption on dead leaves is higher than on sediment (Margoum et al., 2001). The comparative study between herbicide sorption on both sorbents demonstrated that pesticide sorption rises with the increase of organic content (Table 2), which makes leaves in decay much more efficient to retain pesticides than sediment. These experiments complemented and confirmed previous field and laboratory results (Margoum et al., 2001; Margoum et al., 2003). Pesticide sorption on natural sorbents is indeed mainly influenced by sorbent organic matter content (Doherty and Warren, 1969; Dell et al., 1994; Lickfeldt and Branham, 1995; Delle Site, 2001).

Table 4

Responses values from the two experimental matrixes

Experiment number	% adsorbed on sediment (\pm standard deviation)			% adsorbed on leaves (\pm standard deviation)		
	IPU	DIU	DFF	IPU	DIU	DFF
	First experimental matrix					
1 and 5	18 (\pm 1)	34 (\pm 2)	85 (\pm 7)	42 (\pm 2)	52 (\pm 8)	99.3 (\pm 0.1)
2 and 6	10 (\pm 3)	31 (\pm 1)	77 (\pm 3)	32 (\pm 4)	43 (\pm 4)	98.7 (\pm 0.6)
3 and 7	16 (\pm 6)	35 (\pm 4)	87 (\pm 3)	36 (\pm 3)	45 (\pm 3)	99.1 (\pm 0.1)
4 and 8	9 (\pm 1)	33 (\pm 6)	80 (\pm 4)	33 (\pm 6)	43 (\pm 4)	98.4 (\pm 0.8)

Second experimental matrix						
1 and 9	23 (± 1)	36 (± 1)	95 (± 6)	55 (± 1)	85 (± 1)	99 (± 1)
2 and 10	20 (± 3)	27 (± 5)	78 (± 1)	70 (± 6)	92 (± 6)	98 (± 1)
3 and 11	27 (± 1)	31 (± 1)	83 (± 7)	61 (± 1)	72 (± 7)	85 (± 4)
4 and 12	28 (± 6)	30 (± 7)	76 (± 5)	41 (± 1)	85 (± 10)	92 (± 5)
5 and 13	27 (± 1)	0 (± 5)	84 (± 5)	65 (± 6)	87 (± 1)	89 (± 4)
6 and 14	22 (± 10)	0 (± 16)	83 (± 1)	64 (± 3)	86 (± 1)	89 (± 5)
7 and 15	37 (± 2)	8 (± 7)	66 (± 6)	48 (± 1)	70 (± 4)	85 (± 2)
8 and 16	16 (± 8)	0 (± 12)	64 (± 4)	50 (± 6)	62 (± 10)	87 (± 3)

194

195 The effect of each parameter was estimated from these results according to theoretical equations
 196 presented in Goupy (2001).

197 For each experimental matrix, the main effect of a factor is represented by a centred coefficient
 198 response, which is calculated for the three herbicides independently by using the signs of the
 199 corresponding column (Table 3) and either add or subtract the value of the response (Table 4).

200 This summation is finally divided with the number of experiments.

201 Standard deviations on replicates are used to estimate uncertainties, which allows to identify
 202 significant effects. In Figures 1 and 2, the influence of the selected factors on the response is
 203 displayed in bars with confidence intervals. Factors having small piles within the boundaries of
 204 the confidence interval have no significance, while those with more important piles and higher
 205 that confidence interval are more influential.

206

207 Regarding the first experimental design results (Figure 1), pesticide form appears to have an
 208 influence on herbicide sorption on both ditch materials whereas initial sorbent moisture does not
 209 have any significant effect. In addition, the centred response coefficient represents the variation

of the response when the parameter goes from the low level to the high level considered for the experimental design. Therefore, sorption measured with pesticide formulation is in general lower than with active substance.

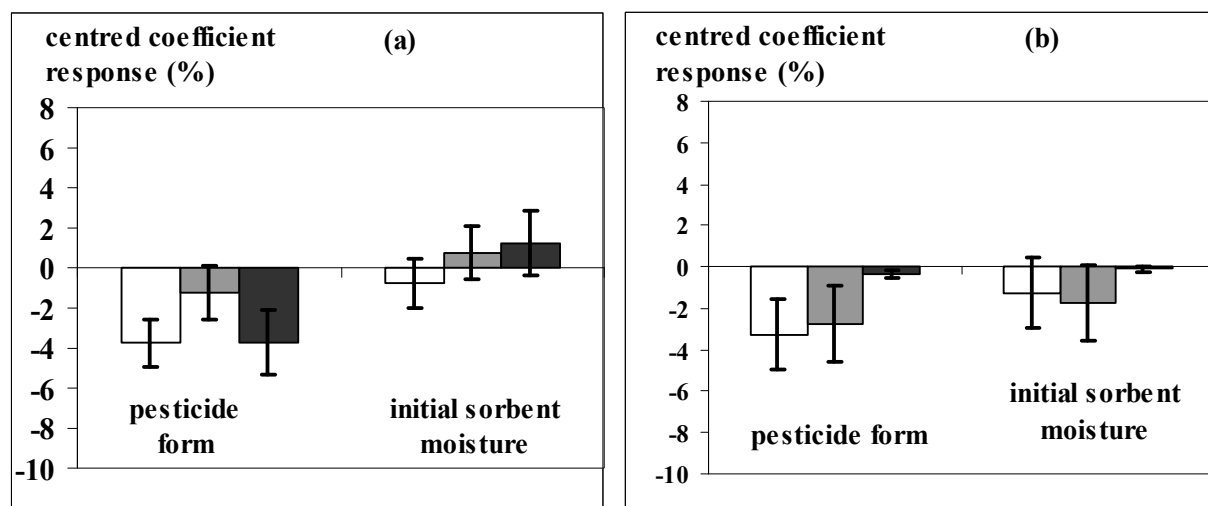


Figure 1. Centred coefficients (%) for the response and associated confidence interval for sorption on (a) sediment and (b) dead leaves (in white, isoproturon ; in grey, diuron and in black, diflufenican)

The DFF sorption results obtained in the second series of experiments dealing with the study of four parameters, i.e. bromide concentration, solid/liquid ratio, water quality, and herbicide form in order to confirm preliminary results, are reported in Figure 2.

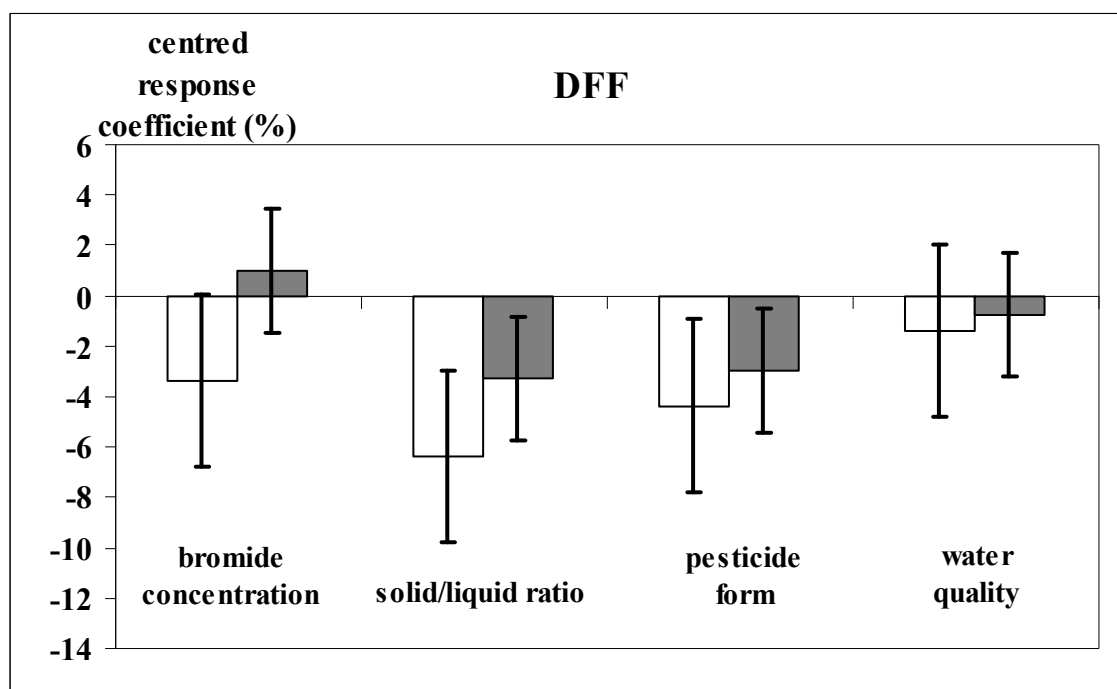


Figure 2. Centred coefficients (%) for the DFF response and associated confidence interval for sorption on sediment (white bars) and dead leaves (grey bars)

When studying the individual variables in details (Figure 2), herbicide form and solid/liquid ratio appear to be the only significant variables for the sorption of the three herbicides on both sorbents, which is consistent with the literature. Boesten (1990) and Crum et al. (1999) demonstrated that sorbent/solution ratio had a great influence on pesticide sorption on various environmental matrixes. Sorption capacity decrease with a higher ratio.

We did not observe any impact of water quality on herbicide sorption. These observations are in accordance with Beck and Jones (1996) who found that isoproturon sorption was clearly influenced by the organic matter content of the sorbent, but that dissolved organic matter in solution had no effect on sorption. We didn't notice any effect of initial sorbent moisture. As the influence of this parameter has not been reported in the literature, we can only mention the results of Gaillardon and Dur (1995) who studied isoproturon and diuron sorption on soil with different moisture contents. They stated that even if adsorption was enhanced by higher soil

moisture at sorption times greater than thirty minutes, increasing the soil moisture had little effect on the final sorption capacity. Finally, we found that the most influential parameter was the herbicide form. We observed that hydrophobic organic compounds are less sorbed when formulated than pure substances. These results are similar to those of Lickfeldt and Branham (1995). The formulation forms micelles that solubilize organic compounds and therefore partially increase their apparent solubility in water (Chaplain et al., 2001).

4. Conclusion

In our study, we used experimental designs (i) to assess pesticide sorption on two different sorbents that were collected in natural French ditches and (ii) to estimate the influence on herbicide sorption of different parameters primarily chosen in accordance with field variables such as sorbent, compound or water characteristics. Sorption on dead leaves is greater than on sediment, which makes leaves in decay more useful to decrease the concentrations and the fluxes of pesticides as they move through agricultural ditches.

Within the investigated experimental area, two main parameters have to be considered as influential on pesticide sorption processes at different scales (from laboratory to field experiments), i.e. pesticide form and solid/liquid ratio. As a consequence, pesticide sorption in field experiments with formulated pesticide might be lower than sorption assessed from laboratory batch technique with active substance. As for the solid/liquid ratio, it is quite difficult to extrapolate its real impact in field studies, since the ratio between the ditch water height and the sorbent is eminently variable, especially in ephemeral ditches and storms. Indeed, in natural ditches, sorption is critically influenced by bottom substratum nature, but also by hydrodynamic conditions such as water flow and height. This cannot be assessed by means of laboratory

studies, therefore other experiments are now designed with an experimental channel in which substratum and hydrodynamics are well controlled.

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