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

3  
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8  
9 **Abstract**

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

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

28

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

30

## 31 **1. Introduction**

32

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

49 Laboratory studies were then conducted to determine the sorption behaviour of three herbicides  
50 with different physicochemical properties (isoproturon: 3-(4-isopropylphenyl)-1,1-dimethylurea,

51 diuron: 3-(3,4-dichlorophenyl)-1,1-dimethyurea, and diflufenican: 2'4'-difluoro-2-( $\alpha$ ,  $\alpha$ ,  $\alpha$ -  
52 trifluoro-m-tolyloxy)nicotinilide) on bed sediment and dead leaves which are common  
53 substratum in French ditches (Margoum et al., 2001). In the present paper, herbicide sorption  
54 was studied as a function of different influencing parameters including the form of the sorbates,  
55 the sorbents and the water characteristics. The influence of some of these factors has already  
56 been studied with other sorbents or pesticides (Lickfeldt and Branham, 1995; Beck and Jones,  
57 1996; Gaillardon, 1996; Walker and Jurado-Exposito, 1998; Chaplain et al., 2001). However,  
58 conclusions were in contradiction. On the one hand, Lickfeldt and Branham (1995) investigated  
59 the adsorption capacity of several non-ionic compounds on leaves and thatch, and concluded that  
60 these chemicals were more adsorbed when formulated or with the presence of dissolved organic  
61 matter. On the other hand, Beck and Jones (1996) observed non significant influence of  
62 dissolved organic matter on atrazine and isoproturon sorption on soils. Gaillardon (1996) showed  
63 that soil moisture enhanced sorption speed and increased final sorption for diuron on soils.  
64 The aim of the present study was to complement preliminary field experimental results  
65 concerning the role played by ephemeral ditch materials on pesticide retention (Margoum et al.,  
66 2003), by studying the influence of five different but not exhaustive parameters on sorption.  
67 Then, we qualitatively estimated to which extent (a) initial sorbent moisture, since the  
68 hydratation status may modify its hydrophobic properties ; (b) formulated herbicide substances  
69 used in field experiments as presented before ; (c) water quality, and especially organic matter  
70 content (Caron et al., 1985; Chiou et al., 1986; Flores-Cespedes et al., 2002) ; (d) bromide ions  
71 used as conservative tracers in experiments concerning pesticide transfer studies (Allaire-Leung  
72 et al., 1999; Schoen et al., 1999), and (e) solid/liquid ratio (Boesten and Van Der Pas, 1988;  
73 Walker and Jurado-Exposito, 1998) could play a significant role in herbicide sorption on typical  
74 ditch sorbents. A fractional factorial experimental design and columns of contrast coefficients  
75 calculation were used for rapid assessment of the effects of the five studied parameters.

76

## 77 **2. Experimental**

78

### 79 *2.1 Chemicals and reagents*

80

81 Three chemicals were used in this research. Isoproturon (99%), diuron (97.5%) and diflufenican  
82 (99.5%) were of analytical grade supplied by Cluzeau Info Labo (Sainte Foy la Grande, France)  
83 (Table 1). These herbicides are commonly used in agricultural catchments for weed control.

84 The commercial formulations were Zodiac TX that contained isoproturon (500 g L<sup>-1</sup>) and  
85 diflufenican (100 g L<sup>-1</sup>), and Séduron (diuron at 500 g L<sup>-1</sup>) both from Bayer Crop Science.

86

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

Characteristics	Isoproturon (IPU)	Diuron (DIU)	Diflufenican (DFF)
Solubility in water (mg L <sup>-1</sup> )	65 (22 °C)	36.4 (25 °C)	0.05 (25 °C)
Koc (L kg <sup>-1</sup> )	36-241	480	1622-2369
log Kow	2.5	2.85	4.9
Half life (day)	6-28	90-180	105-210

88

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

91

### 92 *2.2 Sorbents*

93

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

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

103

104 Table 2

105 Characteristics of the sorbents.

Sorbent	Organic C (g kg <sup>-1</sup> )	Total N (g kg <sup>-1</sup> )	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

106 \* : number of replicate measurements

107

### 108 2.3 Sorption experiments

109

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

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

116 Since the two sorbents had very different bulk densities, the initial masses were adjusted to  
117 ensure a suitable contact between the sorbent and the solution. Therefore, 50 g of dry sediment  
118 or 8 g of dead leaves were added to 250 mL of water in a 500 mL glass Petri dish. Initial  
119 pesticide concentrations in solution were chosen so as to get a compromise between observations  
120 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  
121 DIU).

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

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

134 Analyses of DFF and PCB 153 were performed on a VARIAN 3400 gas chromatograph  
135 equipped with an electron capture detector and a capillary column DB1701 (30 m x 0.32 mm x  
136  $0.25 \mu\text{m}$ ) from JW Scientific. The temperature program was as follows: initial temperature  $40 \text{ }^\circ\text{C}$   
137 raised at  $20 \text{ }^\circ\text{Cmin}^{-1}$  up to  $200 \text{ }^\circ\text{C}$ , then raised at  $10 \text{ }^\circ\text{Cmin}^{-1}$  up to  $280 \text{ }^\circ\text{C}$ . Injector and detector  
138 temperatures were respectively  $250 \text{ }^\circ\text{C}$  and  $300 \text{ }^\circ\text{C}$ . Under these conditions, the retention times



139 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  
140 compounds, and PCB 153 recovery rate was  $89 \pm 10\%$  (n=55).

141 Analyses of IPU, DIU and linuron were performed on a WATERS Alliance 2690 liquid  
142 chromatograph equipped with a photodiode array detector (Alliance 996) and a SymetryShield  
143 RP8 cartridge (4.6 x 250 mm – 5  $\mu\text{m}$ , Waters). The mobile phase was water/acetonitrile (55/45)  
144 at a rate of  $1 \text{ mL min}^{-1}$ . All compounds were detected at 245 nm. The retention times were 8.6,  
145 10.4 and 16.7 min for IPU, DIU and linuron respectively. The detection limit was  $0.1 \mu\text{g L}^{-1}$  for  
146 the three compounds and corresponding recovery rate for linuron was  $101 \pm 6\%$  (n=55).

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

150

#### 151 *2.4 Factorial design*

152

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

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

164 The evaluation of the effect of the investigated parameters basically consists of a two-level full  
 165 factorial design where  $2^{\text{number of variables}}$  experiments have to be carried out. The main advantage of  
 166 such a model is that cross-effects between variables can be easily evaluated (Lundstedt et al.,  
 167 1998; Goupy, 2001). For our study, it was reasonable to assume that the influence of the  
 168 interactions of third order were negligible. Then, we applied a fractional factorial design  $2^{\text{number}}$   
 169  $\text{of variables}-1$ , with two blocks in order to replicate each experiment. A total of 8 and 16 runs was  
 170 then obtained for the first and the second series of experiments respectively. The order of  
 171 execution was randomly selected (Table 3). The percentage of pesticide adsorbed on sorbent was  
 172 chosen as final criterion for the response value.

173

174 Table 3

175 Experimental matrixes with two parameters (first) and four parameters (second). – and +  
 176 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	+	+	+	+

177

178 **3 – Results and discussion**

179

180 Mean values of duplicates for sorption capacity, expressed as the percentage of herbicide  
 181 adsorbed on sorbent, are reported in Table 4 for the two series of experiments. Whatever the  
 182 experimental conditions, our results showed that diflufenican is the more adsorbed compound,  
 183 which is consistent with pesticide physicochemical characteristics (Table 1) and that sorption on  
 184 dead leaves is higher than on sediment (Margoum et al., 2001). The comparative study between  
 185 herbicide sorption on both sorbents demonstrated that pesticide sorption rises with the increase  
 186 of organic content (Table 2), which makes leaves in decay much more efficient to retain  
 187 pesticides than sediment. These experiments complemented and confirmed previous field and  
 188 laboratory results (Margoum et al., 2001; Margoum et al., 2003). Pesticide sorption on natural  
 189 sorbents is indeed mainly influenced by sorbent organic matter content (Doherty and Warren,  
 190 1969; Dell et al., 1994; Lickfeldt and Branham, 1995; Delle Site, 2001).

191

192 Table 4

193 Responses values from the two experimental matrixes

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

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

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.

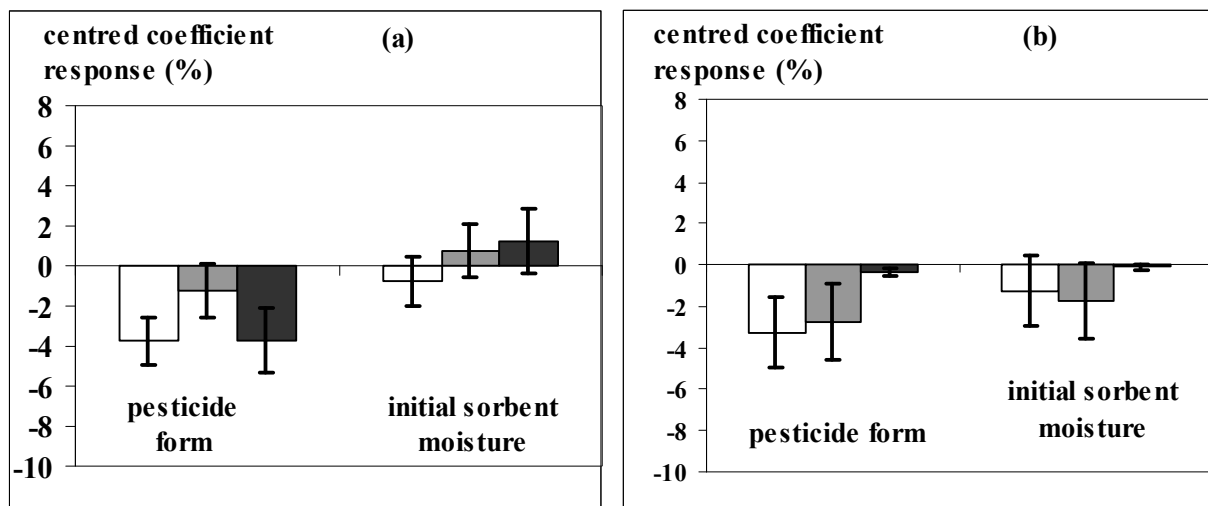
201 Standard deviations on replicates are used to estimate uncertainties, which allows to identify  
 202 significant effects. In Figures 1 and 2, the influence of the selected factors on the response is  
 203 displayed in bars with confidence intervals. Factors having small piles within the boundaries 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

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

213



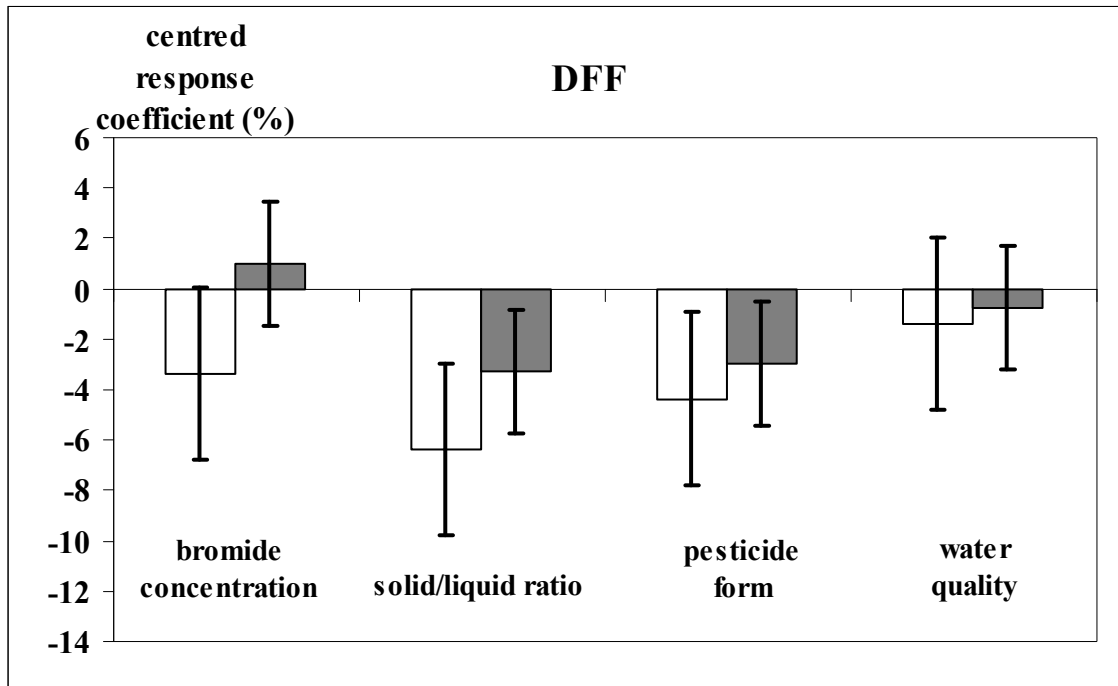
214

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

218

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

222



223  
 224 Figure 2. Centred coefficients (%) for the DFF response and associated confidence interval for  
 225 sorption on sediment (white bars) and dead leaves (grey bars)

226  
 227 When studying the individual variables in details (Figure 2), herbicide form and solid/liquid ratio  
 228 appear to be the only significant variables for the sorption of the three herbicides on both  
 229 sorbents, which is consistent with the literature. Boesten (1990) and Crum et al. (1999)  
 230 demonstrated that sorbent/solution ratio had a great influence on pesticide sorption on various  
 231 environmental matrixes. Sorption capacity decrease with a higher ratio.

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

239 moisture at sorption times greater than thirty minutes, increasing the soil moisture had little  
240 effect on the final sorption capacity. Finally, we found that the most influential parameter was  
241 the herbicide form. We observed that hydrophobic organic compounds are less sorbed when  
242 formulated than pure substances. These results are similar to those of Lickfeldt and Branham  
243 (1995). The formulation forms micelles that solubilize organic compounds and therefore  
244 partially increase their apparent solubility in water (Chaplain et al., 2001).

245

#### 246 **4. Conclusion**

247

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

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

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

265

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267

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271

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