

The use of enantiomeric ratios to assess the fate of mecoprop in groundwater

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ABSTRACT

Disposal of the chiral phenoxyacid herbicide mecoprop into landfills in the Lincolnshire Limestone has polluted an abstraction well 2.5 km away. Differences in the biological behaviour of the two mirror image structures of mecoprop (or enantiomers), means that changes in the enantiomeric ratio (ER) can help identify the extent of biodegradation down gradient of the landfill. Deposited as a racemic mixture (i.e. 50% of each enantiomer), there has been no change in the ER in the most polluted part of the landfill plume where conditions are sulphate reducing/methanogenic, indicating no degradation. In the iron and nitrate reducing zones of the plume (*S*)-mecoprop dominates suggesting either inversion of the (*R*)-mecoprop to (*S*)-mecoprop, or faster degradation of (*R*)-mecoprop. In the aerobic aquifer the gradual increase in the ER in favour of (*R*)-mecoprop suggests faster degradation of (*S*)-mecoprop. The persistence of mecoprop in the confined Lincolnshire Limestone further down dip is explained by degradation being inhibited by sulphate reducing conditions that develop naturally.

INTRODUCTION

Mecoprop exists in two mirror image forms (Figure 1). The (*R*)- and (*S*)- enantiomers have identical chemical and physical properties but can behave differently when they interact with other chiral compounds in biological systems. Only the (*R*)-mecoprop is herbicidally active (Loos 1975), but both enantiomers are known to degrade aerobically although at different rates (Harrison *et al.* 1996). Degradation under anaerobic conditions has been reported to be slight or absent. Muller & Buser (1997) provided evidence for enantiomeric inversion in both directions and also derived the quasi-first order reaction rates for the processes shown in Figure 2 (Buser & Muller 1998). Kohler *et al.* (1999) reported enrichment of (*R*)-mecoprop down gradient from a landfill in Switzerland and ascribed it to the faster degradation of the (*S*)-enantiomer. They concluded that changes in the enantiomeric ratio of mecoprop provide useful evidence in support of its biotransformation and natural attenuation in groundwater.

In this study, the degradation of mecoprop down gradient from a landfill site in the Lincolnshire Limestone aquifer has been investigated by determining changes in the enantiomeric ratio, and in the stable carbon isotope ratios of the enantiomers, in relation to the prevailing redox zones. Stable isotope fractionation is an established technique for identifying biologically mediated reactions and leads to an increase in the proportion of ^{12}C in the products and a consequent decrease in ^{12}C (increase in ^{13}C) in the reactant (Hoefs 1973).

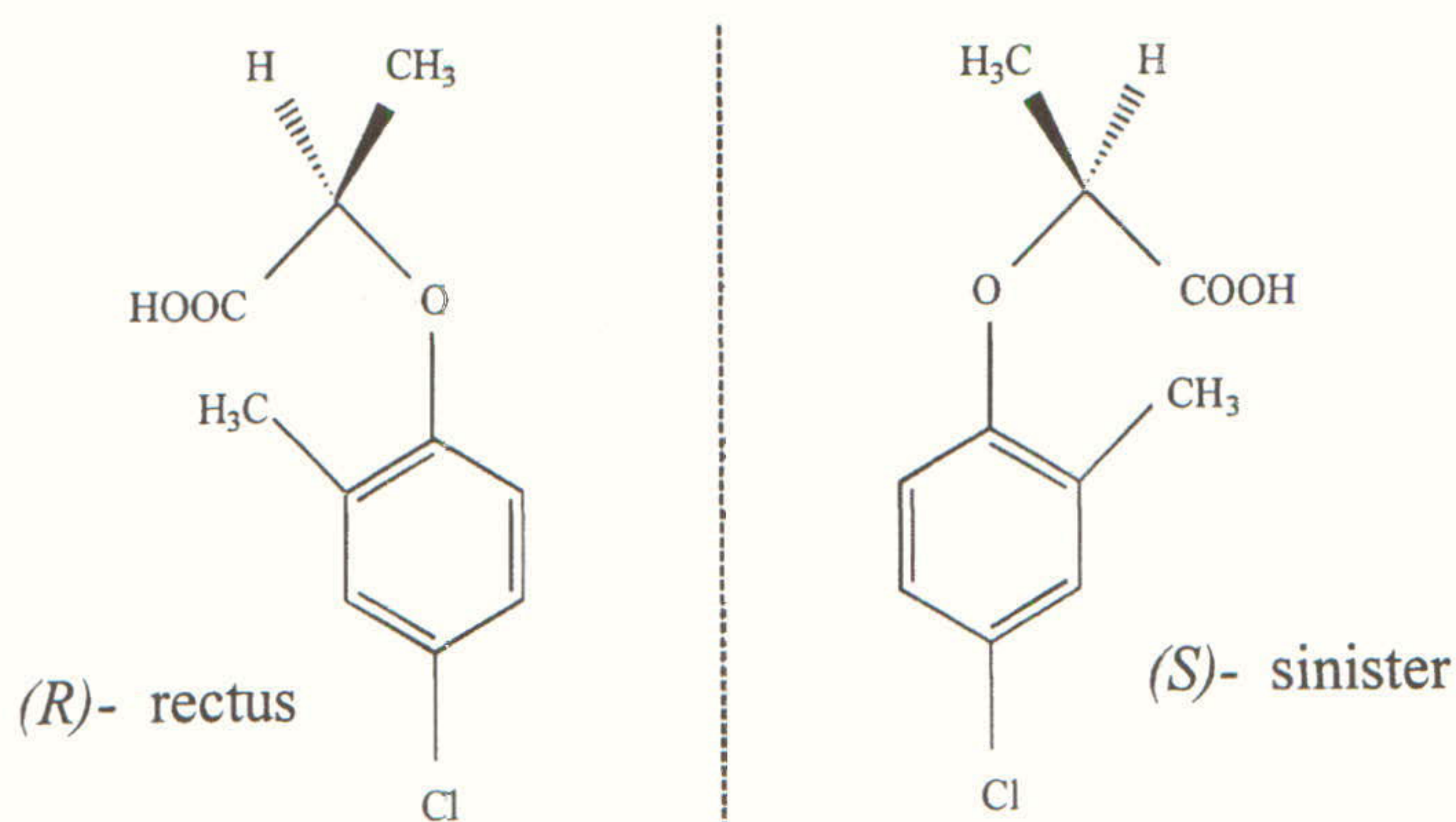


Figure 1. The enantiomers of 2-(4-chloro-2-methylphenoxy)-propionic acid (or mecoprop)

SITE DESCRIPTION

Three landfills which received an estimated 40 tonnes of the herbicide mecoprop as liquid waste, are situated in former quarries in the Lincolnshire Limestone which is a fractured highly permeable aquifer extensively used for water supply in the UK. The landfills lie just west of a major geological fault which at one time was considered to be a barrier to groundwater flow (Sweeney *et al.*, 1998). East of the fault the aquifer is overlain by relatively impermeable strata and, with increasing distance, the aquifer becomes confined with artesian flowing wells. Up to 8 $\mu\text{g/l}$ mecoprop has been detected in a public supply borehole, 2.5 km to the east of the landfills, and the supply is treated to reduce concentrations below the EEC recommended limit of 0.1 $\mu\text{g/l}$ (EEC 1980).

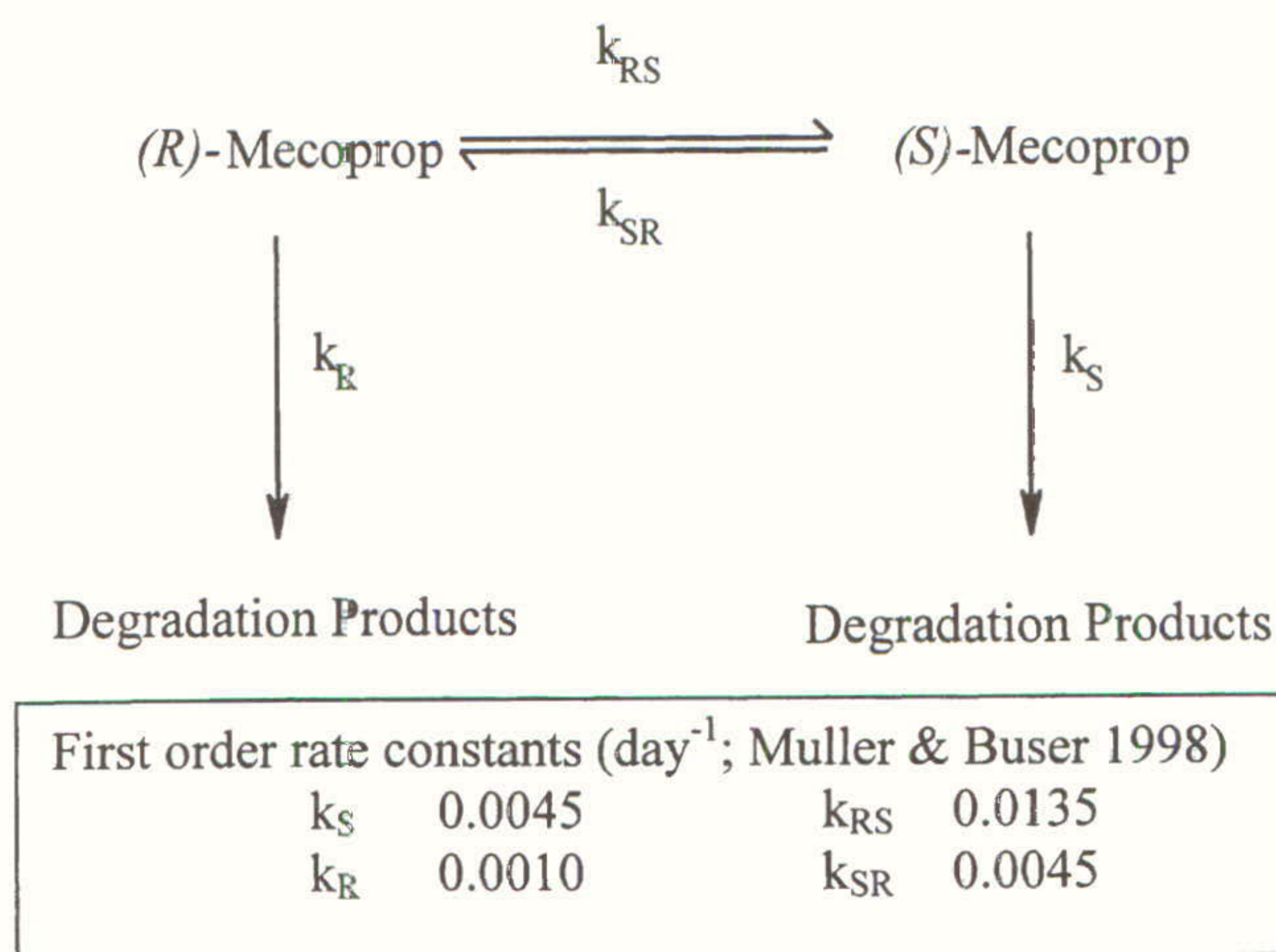


Figure 2. Scheme for mecoprop inversion and degradation (after Buser & Muller, 1998)

GROUNDWATER SAMPLING AND ANALYSIS

Groundwater samples from approximately 30 boreholes were analysed for major and trace inorganic species, for non-particulate organic carbon (NPOC) and for mecoprop. Mecoprop analysis differentiated between the two enantiomers and included measurement of their stable carbon isotope ratios ($\delta^{13}\text{C}$). Broad scan GC/MS was used to qualitatively identify organic compounds. Full details of the analytical methods and the compounds identified are reported in Williams *et al.*, (2001).

RESULTS AND DISCUSSION

The Piper diagram (Figure 3) is a convenient geochemical tool for displaying variations in major ion chemistry (Domenico & Schwartz 1998). The percentage equivalents of the total anions or cations are shown in the lower triangles whilst the overall composition is plotted in the trapezoid. The line joins groundwater compositions along a flow path from the landfill to the confined aquifer down dip to the east. Immediately surrounding the landfill the groundwater is highly mineralised, organically polluted, sulphate reducing and methanogenic (Zone I). With distance, the plume is diluted and disperses and redox conditions move towards iron and nitrate reducing (Zone II). Further east the aquifer becomes confined and initially contains aerobic calcium bicarbonate dominated groundwater (Zone III). Further away it becomes dominated increasingly by calcium sulphate, then becomes saline (Zone IV) as a result of sulphate reduction (Edmunds & Walton 1983).

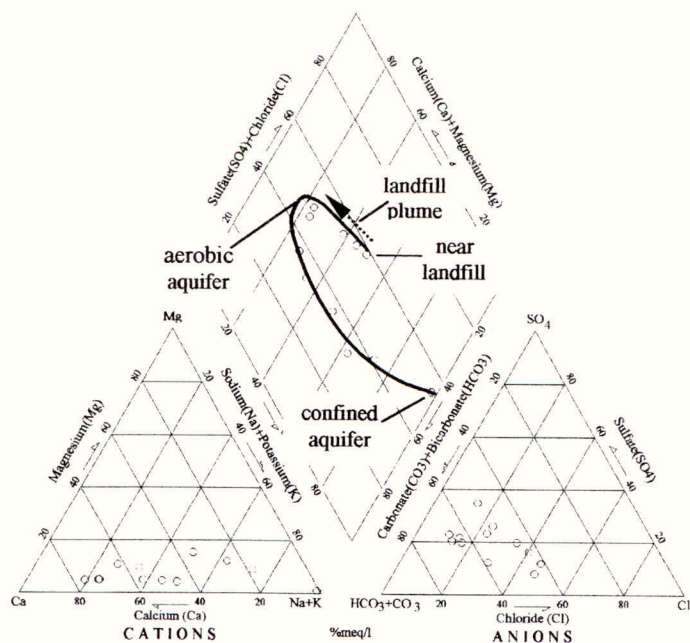


Figure 3. Piper diagram showing changes in water chemistry with distance from the landfill ('o' depicts water composition)

These four geochemical zones form a basis for comparing changes in the enantiomeric ratio (Table 1). Actually the enantiomeric fraction rather than the enantiomeric ratio is a more convenient expression to avoid infinite values (Harner *et al.* 2000). The enantiomeric fraction (EF) is defined as follows:

$$EF = [R] / ([R]+[S]) \quad (1)$$

Where [R] and [S] are the concentrations of (*R*)- and (*S*)-mecoprop respectively.

Change in the enantiomeric fraction (EF) along the flow path from the landfill (Figure 4) show that in the most polluted methanogenic part of the aquifer (Zone I, mecoprop \approx 7 mg/L) the mecoprop is still racemic (EF = 0.5) which suggests that no degradation has occurred within a period of 8 years since the last known disposal occurred in 1991.

With migration, dilution and dispersion, mecoprop concentrations reduce and the plume becomes more oxidising but not aerobic (Zone II). Here, at mecoprop concentrations around 500 μ g/L, and total organic carbon (TOC) still above background, Fe, Mn and nitrate reducing conditions prevail, and (*S*)-mecoprop dominates. This EF could be interpreted as either inversion of (*R*)- to (*S*)- and/or faster degradation of (*R*)-. As mecoprop concentrations fall further and conditions become aerobic (Zone III), the EF rises above 0.5 as (*R*)-mecoprop dominates. This is consistent with the faster degradation of (*S*)-mecoprop under aerobic conditions, and is similar to observations by Muller and Buser (1997), and Kohler *et al.*, (1999).

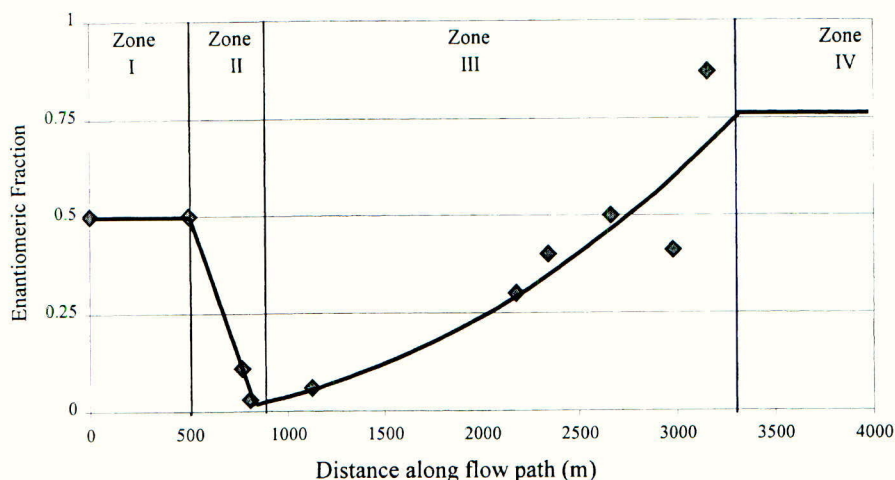


Figure 4. Changes in mecoprop EF with distance along the flow path from the landfill

It is noteworthy that the field samples show only slight changes in $\delta^{13}\text{C}$ ($<2\%$), and thus provide tenuous evidence for microbial transformation. However, changes would not be expected to be large since isotopic fractionation as a result of side chain attack would be diluted by the unchanged carbon isotope ratio in the aromatic moiety. Evidence of fractionation from controlled microcosm experiments not prone to field sample variability is expected to provide a better indicator of microbial mediation.

Table 1. Geochemical zones, changes in mecoprop EF and $\delta^{13}\text{C}$, and inferred processes

Zone	Organic C Content	Redox state	EF	Inferred processes	
			$\delta^{13}\text{C}$		
I	Close to landfill.	High 100-1000 mg/L	Sulphate reducing/ methanogenic	EF = 0.5 No change in $\delta^{13}\text{C}$	No degradation or inversion
II	In landfill plume	Medium 10-100 mg/L	Iron & nitrate reducing	EF < 0.5 slight (?) $\delta^{13}\text{C}$ increase in R & S	<ul style="list-style-type: none"> • Inversion of (<i>R</i>)- to (<i>S</i>)- and/or • faster degradation of (<i>R</i>)- than (<i>S</i>)-
III	Aerobic aquifer	Low <10 mg/L	Aerobic	trend to EF > 0.5 slight (?) increase for R, more for S	faster degradation of (<i>S</i>)- than (<i>R</i>)-
IV	Confined aquifer	Low <10 mg/L	Sulphate reducing	EF constant no $\delta^{13}\text{C}$ data	no degradation or inversion

This preliminary interpretation of the field data is presently being confirmed using laboratory microcosms to identify the conditions under which inversion or degradation occur; the rate constants; the microbial consortia present; and, if possible, the enzymes responsible for inversion/degradation. Early indications are that under anaerobic conditions (*R*)-mecoprop degrades but (*S*)-mecoprop does not. There also appears to be little evidence for inversion of (*R*) to (*S*).

CONCLUSIONS

Change in the enantiomeric ratio of mecoprop is a useful indicator of *in situ* processes such as degradation/inversion in different redox zones. In this study the dominance of the (*S*)-enantiomer in the anaerobic landfill plume may be explained by faster degradation of (*R*)-mecoprop, or inversion of (*R*)- to (*S*)-. This is the reverse of that reported for aerobic environments where (*S*)-mecoprop degrades faster than (*R*)-. In general, the fact that enantiomers may invert means that the fate of each enantiomer needs to be assessed even if enantiomerically pure formulations are manufactured and released into the environment.

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Biobeds: safe disposal of pesticide waste and washings

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ABSTRACT

The presence of pesticides in environmental waters can come from a number of point and diffuse sources. However, contamination arising from non-approved use, poor practice, illegal operations or misuse of pesticides is increasingly thought to be responsible. Biobeds appear to provide a low cost system for treating pesticide waste and washings, providing a matrix to adsorb the pesticide(s) and facilitate biodegradation. Water management is crucial in terms of biobed performance, construction costs and management. Unlined biobeds appear to have a similar level of performance as more costly treatment systems.

INTRODUCTION

When used correctly according to the label instructions and with the appropriate precautions pesticides should present minimal risk to the environment. However the small drips and spillages which occur as part of normal agricultural practice can result in a significant amount of surface water contamination (Mason *et al.*, 1999). In the UK washings from equipment and spray tanks should be disposed of in accordance with the Code of Practice for the Safe use of Pesticides on Farms and Holdings (1998, currently under review) and the Groundwater regulations (1998). However, due to the practicalities and costs associated with the recommended procedures and lack of awareness of the legislation, it is possible that many users do not comply with these requirements. A system is therefore required that is able to treat the small drips and spills which occur as part of the normal mixing procedure as well as larger volumes of tank and equipment washings. The system must be robust in terms of its ability to retain and degrade high concentrations of pesticide mixtures applied repeatedly, simple to construct and manage and require a low technical input. Biobeds appear to offer an alternative to current methods of treating pesticide waste and washings.

In its simplest form a biobed is a hole in the ground filled with a mixture of topsoil, peat and straw (Torstenson & Castillo 1996, 1997). The biobed is covered with grass and equipped with a ramp enabling the tractor and sprayer to be driven over the bed. Studies in Sweden have demonstrated that biobeds can effectively retain and degrade pesticide waste arising from accidental spillages of prepared pesticides on concentrate. In order to be used on UK farms biobeds would also need to be able to cope with much larger volumes of waste arising from tank and machinery washings. The objective of this study was to determine whether biobeds could be used to treat pesticide spills, waste and washings arising from UK farms. A series of laboratory and semi-field experiments were performed to determine the degradability and leaching potential of pesticides in a biobed under conditions that are likely in the UK. Previous studies using

lined biobeds have demonstrated that water management is crucial in terms of construction costs, management and performance. Although pesticides were effectively retained within the biobed, saturated conditions below 10 cm and poor degradation means that unlined biobeds are unlikely to work on UK farms. This paper focuses on results from a semi-field experiment.

MATERIALS AND METHODS

The degradability and leaching potential of 6 pesticides (Table 1) with a range of hydrophobicities and half lives were measured.

Table 1. Study compounds and their reported physico-chemical characteristics

Active Substance	K _{oc} ml/g	DT ₅₀	Water Solubility (mg/l)	Application rates	
				Topsoil vs Biomix	Water loading
Isoproturon	100	6 - 28	65	1114	255
Pendimethalin	5000	90 - 120	0.3	800	204
Chlorpyrifos	6000	60 - 120	1.4	583	73.4
Chlorothalonil	1600 - 14000	6 - 43	0.81	653	153
Epoxiconazole	957 - 2647	60 - 90	6.6	76	51
Dimethoate	16 - 52	7 - 16	22300	244	34.7

Values taken from Wauchop *et al.* (1992) and Tomilin (2000)

Pesticide leaching from topsoil and biomix

The leaching potential of pesticides in soil and biomix were compared. Two sets of four cores were prepared using unplasticised polyvinyl chloride (PVC-u) piping (20 cm diameter x 75 cm length) filled with 2-3 cm of gravel followed by 15 cm of washed sand and a 50 cm layer of either fresh biomix (50% straw, 25% peat substitute, 25% topsoil) or topsoil (69% sand, 13% silt, 18% clay, 1.95% organic matter, pH 6.15). The base of each core drained via Teflon tubing to a 2.5 l amber glass bottle. Three of the biomix filled cores and 3 of the topsoil filled cores were treated with the study pesticides. Split applications of isoproturon, pendimethalin, chlorpyrifos and chlorothalonil, epoxiconazole dimethoate were made in the autumn and spring respectively. A bromide tracer was also applied (628 mg/core) to check the hydrological integrity of the lysimeters, as well as looking at the breakthrough timing of infiltrating water. Collection vessels were monitored after all rainfall events and the total volume of leachate recorded. Volumes in excess of 500 ml were collected and stored at (0 - 10°C) prior to analysis. Where possible, a 60 ml sub-sample was also taken for bromide analysis. At the end of the study all cores were sectioned (0-5, 5-10, 10-20, 20-30 and >30 cm), homogenised and stored at -15°C prior to analysis.

Effect of water loading on pesticide leaching

The effect of water loading on pesticide leaching behaviour was also investigated. Twelve cores containing pre-composted biomix (composted for 97 days) were prepared. Cores were again constructed from PVC-u tubing and consisted of a 50 cm layer of biomix on a 5 cm layer of coarse gravel draining to an adjacent collection vessel. Three hydrological scenarios were investigated. Four cores were connected using plastic guttering to 0.54 m² concrete slabs, four cores were connected to 0.135 m² concrete slabs whilst the remaining cores received only direct inputs of rainfall. The study pesticides were applied as a mixture at an application rate based on concentrations of pesticides measured in second rinse tank washings. A bromide tracer was again applied (314 mg/core). Water samples were collected once leachate volumes exceeded 200 ml and the cores were destructively sampled at the end of the study (299 DAT). All samples were frozen at (-15°C) prior to analysis.

Analytical methods

Water samples (200 ml) were extracted 3 times into 30 ml dichloromethane (DCM). The DCM fractions were filtered through anhydrous Na₂SO₄ and then evaporated to dryness. The resulting residues were re-dissolved into 2 ml of a mixture containing 10% methanol and 90% DCM. Soil and biomix samples (40 g) were mixed with 60 g anhydrous Na₂SO₄ and extracted using 160 ml of a mixture containing 10% methanol and 90% DCM. Samples were shaken for 1 h and allowed to stand until clear with a 2 ml aliquot taken for analysis. Concentrations of each pesticide were determined using GC, (Hewlett Packard HP5890) fitted with a split/splitless injector, 12 m x 0.53 mm BPX5 column (SGE) and a nitrogen-phosphorous detector.

RESULTS and DISCUSSION

Topsoil Vs Biomix

Topsoil and biomix lysimeters received 116% of the long term average rainfall with 13 samples of leachate being collected over a 9 month period. With one exception cumulative leachate volumes were similar between columns with approximately 10 l collected. Rapid breakthrough of bromide was observed from topsoil lysimeters with highest concentrations observed 35 DAT. Movement of bromide through biomix filled cores was much slower with maximum concentrations not being observed until 102 DAT.

With the exception of pendimethalin concentrations of pesticide in leachate from biomix filled lysimeters were significantly lower than in leachate from topsoil (for example data for isoproturon are presented in Figure 1). Considering the physico-chemical properties of pendimethalin (i.e a high K_{oc}) this result cannot be explained without further investigation. Peak concentrations of active ingredient in leachate from biomix ranged from 0.15 µg/l (epoxiconazole) to 127 µg/l (isoproturon) whereas from topsoil cores concentrations ranged from 0.47 µg/l (pendimethalin) to 3845 µg/l (isoproturon). With the exception of dimethoate in soil, no pesticide was detected in the soil or biomix matrix below 30 cm depth with the majority being retained in the top

10 cm. A mass balance was performed to determine the fate of each of the study compounds in topsoil and biomix (Table 2). This indicated that in biomix only a small proportion is leached and between 70 and 93% is degraded.

Table 2. Mass balance (*) for topsoil and biomix lysimeters

Pesticide	TOPSOIL			BIOMIX		
	Leached %	Retained %	Degraded %	Leached %	Retained %	Degraded %
Isoproturon	1.5	0.7	97.8	0.1	7.0	92.9
Pendimethalin	0	37.4	62.6	0	28.8	71.2
Chlorpyrifos	0	14.6	85.4	0	13.1	86.9
Chlorothalonil	0.2	34.2	65.5	0	25.6	71.4
Epoxiconazole	0.3	24.7	75.0	0	30.0	70.0
Dimethoate	8.4	4.0	87.5	0	7.3	92.7

* Mass balance calculated 217 days after last application of isoproturon, pendimethalin and chlorpyrifos and 83 days after application of chlorothalonil, epoxiconazole and dimethoate

Effect of additional water loading

Rainfall between January and September 2000 was 17% above average with leachate samples collected on 28 occasions. Cumulative leachate volumes ranged from 3.4 to 5.1 l from biobeds receiving only direct rainfall, between 45.2 - 56.4 l were collected from biobeds receiving a medium water loading and between 103.7 and 177.6 l from biobeds subjected to a high water loading.

Bromide movement through biobed columns with a high water loading was rapid. Breakthrough occurred 7 DAT and coincided with mean maximum bromide concentrations. From biobeds subjected to a medium water loading, bromide breakthrough also occurred 7 DAT, however maximum concentrations were not measured until 29 DAT. Water movement through biobed columns receiving only direct rainfall was slow, and breakthrough was detected 57 DAT with mean maximum concentration not measured until 229 DAT.

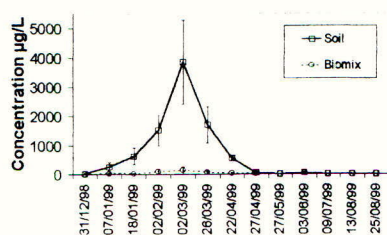


Figure 1. Mean concentrations (± 1 SE) of isoproturon in leachate from topsoil and biomix

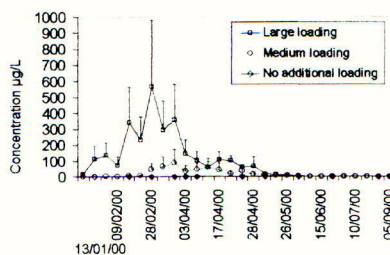


Figure 2. Mean concentrations (± 1 SE) of isoproturon in leachate from biobed columns subjected to a high, medium and no additional hydraulic loadings

Maximum pesticide concentrations were measured in leachate collected from biobed columns with a high water loading. These were an order of magnitude higher than from biobeds with a medium water loading and 3 orders higher than columns receiving only direct rainfall inputs (for example results for isoproturon are shown in Figure 2.) Generally, highest concentrations were observed for the most mobile compounds, isoproturon and dimethoate. Peak concentrations ranged from 1.21 µg/l (epoxiconazole) to 1367 µg/l (isoproturon) from biobeds with a high water loading, from 0.35 µg/l (epoxiconazole) to 258 µg/l (isoproturon) from the medium loading experiment; and from 0.57 µg/l (dimethoate) to 1.65 µg/l (chlorpyrifos) from biobeds with no additional water loading. From high water loading biobeds no concentrations of pesticide were measured below 30 cm depth, with 95 - 100% retained in the top 10 cm of biomix. A similar distribution was observed for the medium loading cores. No pesticide was detected below 20 cm depth with 97 - 100% retained in the top 10 cm. From lysimeters receiving only direct rainfall inputs no concentrations of pesticide were measured below 10 cm depth. A mass balance was performed to determine the fate of each of the study compounds under each of the three hydraulic scenarios investigated (Table 3) and indicated that with a medium water loading <1% of the applied pesticide leached, with between 66 and 99% degraded within 9 months.

Table 3. Mass balance for biobed columns receiving a high, medium and no additional hydraulic loading

	% Leached			% Retained			% Degraded		
	High	Medium	No	High	Medium	No	High	Medium	No
Isoproturon	6.4	0.2	0	0.1	0.1	1.2	93.5	99.7	98.8
Pendimethalin	0.1	0	0	12.8	14.9	17.2	87.1	85.1	82.8
Chlorpyrifos	0	0	0	0.4	0.7	3.5	99.5	99.3	96.5
Chlorothalonil	0.1	0	0	1.8	1.7	9.4	98.0	98.3	90.5
Epoxiconazole	0	0	0	33.5	33.9	32.8	66.4	66.1	64.1
Dimethoate	6.1	0.6	0	0	0	0.1	93.9	99.4	99.9

CONCLUSIONS

Studies using open biobeds confirmed that the biomix could retain and subsequently degrade high concentrations of pesticide. Performance of biobeds with a medium water loading was similar to other commercially available treatment systems with >99.4% of the applied pesticide retained of which >66% was degraded within 9 months. However, in order for biobeds to be approved for use in the UK performance will have to improve such that concentrations of pesticide potentially reaching ground water are <0.1 µg/l. Theoretically biobeds can achieve this target.

BIOBED DESIGN

Using the relationship between hydraulic loading expressed in l/m³ of biomix and the mean maximum concentrations of pesticide measured in leachate, the size of biobed required to achieve 0.1 µg/l pesticide concentration in leachate can be calculated.

Assuming that two identical biobeds are operated in series (Figure 3), to treat 10000 litres of dilute pesticide waste and to achieve pesticide concentrations less than 0.1µg/l the biobeds would need to be 3 m³ in size.

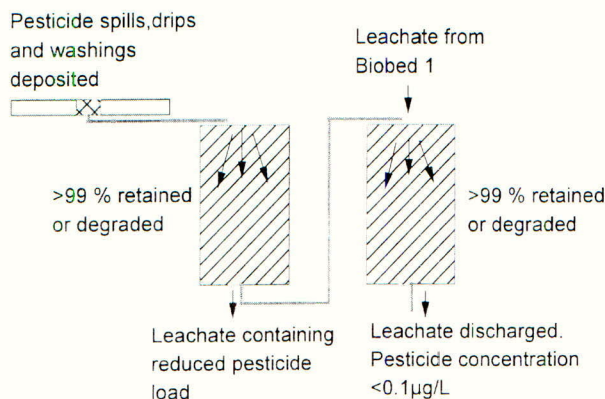


Figure 3. Schematic diagram of unlined biobed system

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