OCCURRENCE, REMOVAL AND POSSIBLE EFFECTS OF PESTICIDES IN MSW LEACHATE

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Grab samples from municipal solid waste leachate show pesticide residues from 0 to 30 µg/l. The total number of compounds found in leachate is 14 of a total of more than 50 compounds included in the analytical methods. Samples from MSW leachate contains herbicides and fungicides, while leachate from locations such as tree nursery plants contains more insecticides. Field monitoring samples from surface and ground waters show a large variety of contemporary and obsolete pesticides, with a frequency of detection depending on the frequency and amount of application, this is probably also reflected in leachate samples. Studies of different filter materials showed that the most important factor for pesticide partitioning is organic carbon measured as TOC. The variation in binding properties (Kd) correlated highly with lipid partitioning (Kow, r=0.71), but showed little correlation, but negative as expected, to water solubility (r=-0.15). The correlation between Kow and water solubility was r=-0.31.

For highly nonpolar compounds such as DDT and to a lesser degree, aclonifen and lindane, the binding properties depends mostly on the type and amount of organic material in the filter medium, and specifically to the high molecular C and low O type organics. For more polar compounds such as e.g. herbicides, the binding properties seems to be linked to the number of H molecules in the chemical structure of the pesticide. Many compounds exceed concentration limits for environmental effects. Indications on more chronic effects in downstream biological systems will be carried out in Norway. Studies of binding properties to specific materials are useful for targeting filter media in runoff systems. Predicting leaching is an entirely more difficult exercise that so far has been relatively unsuccessful.

Key Words—Municipal solid waste leachate, pesticides

1. Introduction

Microorganic pollutants including pesticides are a major concern when emitted to the environment. A municipal landfill constitutes most substances used in the society, and both mother compounds and metabolites are suspect to leach out of the waste body. It is generally assumed that landfills contain pesticides, at least in areas with a large agricultural production, or near pesticide production sites or retailers. There are about 200 active municipal landfills in Norway, but in total more than 3000 sites have been registered. The total sale of pesticides amounted to 955 ton active ingredients pr. year in 1998, according to the Norwegian Agricultural Inspection Service (2000), adding up to about 1 kg per hectare arable land. 68 tons, or 7%, of the compounds are not related to agricultural activities. There are about 120 approved active ingredients in pesticides sold in Norway, compared to more than 500 compounds in Spain and France (EEA, 1999).

The pesticide concentration in the leachate will generally depend on adsorption and degradation inside the waste body and in the leachate, which again depends on the waste characteristics, the management of the landfill, climate, topography and geology of the site

(Öman & Rosqvist, 1999). Also the content and type of colloidal matter and suspended particles in the leachate are important, and pesticides have been reported to be associated more with colloidal matter mobilised shortly after precipitation events (Worral et al., 1999). In the liquid phase the content and type of organic matter may significantly influence the fate of pesticides (Chiou et al., 1986; Chiou et al., 1987; Piccolo et al., 1996; Haarstad & Fresvig, 2000). Usually data are available for lumped parameters such as TOC, DOC, NVOC, COD and BOD, which are easy to measure and give information about the oxygen consumption potential, but say very little about the toxicity, bioavailability and persistence of the compounds (Ledin et al., 1999). Generally there are a number of compound specific factors that influence the environmental behaviour of pesticides in water and soil (Bailey and White, 1970): 1) chemical character (molecular shape and configuration), 2) acidity or basicity defined by pKa or pKb, 3) water solubility, 4) charge distribution on the organic cation, 5) polarity, 6) molecular size and 7) polarizability. The properties of solid materials are of course also important. It is mainly the soil organic C that influences soil mobility through four important mechanisms for the retention of organic chemicals by humic substances in soil: (1) ion exchange, (2) hydrogen bonding, (3) van der Waals forces, and (4) coordination through an attached metal ion through ligand exchange (Stevenson, 1985).

There are relatively few reports on pesticide concentrations in leachate. Leachate samples from a waste disposal site in Switzerland showed concentrations up to 124 μg/l of mecoprop, and downgradient groundwater with surprisingly high concentrations up to 975 μg/l (Zipper et al., 1998). The change of the ratio of the enantiomeric isomers of mecoprop is taken as an indication of in-situ bioremediation in the aquifer. In a testing program for leachate characteristics in Sweden, it was found that 5 out of 8 leachate samples contained residues of phenoxy acid herbicides, and only one sample out of 20 of leachate particles contained pesticide residues (Öman, 1999; Öman & Rosqvist, 1999). Analyses of leachate samples from a landfill in Denmark showed little detection of pesticides (Ledin et al., 1999).

A municipal landfill constitutes most substances used in the society. Pesticides are among the compound suspect to produce biological effects in the environment, and thus they are a major concern. At Lake Molnbyggen Sweden, the measured biological effects show the same biochemical effect pattern as the antifungal agent Ketoconazole (KTZ) without excluding other components. There are 5 active ingredients in pesticides belonging to the azole group licensed for sale in Norway: propikonazole, tiabendazole, cyprokonazole, penkonazole and tebukonazole. The pesticide concentration in the leachate will generally depend on adsorption and degradation inside the waste body and in the leachate. Sorption depends on the waste characteristics, the management of the landfill, climate, topography and geology of the site (Öman and Rosqvist, 1999). MSW leach showed pesticides residues from 0 to 30 μg/l. Leachate samples from a waste disposal site in Switzerland showed concentrations up to 124 μg/l of mecoprop, and down gradient groundwater showed concentrations up to 975 μg/l, but these findings were related to sources with very high content of pesticides (Zipper et al., 1998). In a testing program for leachate characteristics in Sweden, it was found that 5 out of 8 leachate samples contained residues of phenoxy acid herbicides (Öman, 1999; Öman & Rosqvist, 1999).

Leachates from landfills generally contain some components that have acute effects on aquatic organisms, e.g. ammonia in combination with bicarbonate (high pH), but will also contain components that act on organisms following a chronic exposure. Ammonia and alkalinity in leachates are associated with increasing toxicity of duckweed, micro-crustacean algae (daphnia) and other organisms Clèment et al. (1997); Cossu et al., 2000). The Institut för Tillämpad Miljöforskning (ITM), Stockholm University, has studied the biological effects of leachates on fish, which may be pertinent to Norwegian aquatic habitats. In 1994 to 1995 it was claimed that perch (*Perca fluviatilis*) and northern pike (*Esox lucius*) from Lake Molnbyggen, located in the central part of Sweden, showed signs of illness and had increased frequencies of various skin lesions. Leakage water from a municipal landfill was the suspected source of contamination. A comparison of fish from Lake Molnbyggen with fish from Lake Djursjön, a reference lake, found severe reproduction disorders in Lake Molnbyggen (Noaksson et al., 1998; Noaksson et al., 1999; Noaksson et al., 2000a; Noaksson et al., 2000b). Only one fourth of the female perch in Lake Molnbyggen was found to be sexually mature, compared with 96%, which is normal, in Lake Djursjön. Furthermore, there were high frequencies of open sores and fin erosion, lower aromatase activity (the enzyme that catalyses the conversion of androgens into estrogens) in the brain, and lower circulating levels of testosterone and 17β-oestradiol (Noaksson et al., 2000a; Noaksson et al., 2000b). No signs of parasites, pathogens or infections were found in the perch. Since several species of fish were found to be similarly affected, and the biomarkers - DNA adducts and ethoxyresorufin O-deethylase (EROD) - showed no or only a weak induction, it was concluded that the fish were exposed to specific endocrine disrupting substances (EDS). The species was possibly able to interact with important functions in the synthesis of steroids and thereby disrupting a normal development of the gonad.

In order to further investigate if the leakage water from the public municipal refuse dump at Lindbodarna could be the source of EDS causing the reproductive disorders and endocrine disruption, brook trout (*Salvelinus fontinalis*) from the stream Vadbäcken, known to be contaminated by leachate from the refuse dump, was investigated. Female brook trout from Vadbäcken was found to show the same kind of reproductive disorders and endocrine disruption as female perch in lake Molnbyggen e.g. a very low number of sexually mature females paralleled by lower aromatase activity in the brain as well as lower circulating levels of testosterone and 17β-oestradiol. Male brook trout also showed decreased size of the gonad, very similar to male perch and roach from Molnbyggen. However, skin lesions, such as sores and fin erosion, were not found on brook trout from Vadbäcken (Noaksson et al., 1999; Noaksson et al., 2000a; Noaksson et al., 2000c). These results clearly indicate that the reproductive disorders and endocrine disruption found in fish from Lake Molnbyggen and from the Vadbäcken stream are due to EDS in water leaking from municipal landfill.

Another biological indicator has been studied by ITM. An amphipod, *Monoporeia affinis* was exposed to sediments from Lake Molnbyggen. After three months there was a significant lack of gonad growth. In testing leachates it is important to be able to distinguish between properties or components that can easily be removed or degraded from components that remain in the system and are able to cause effects over time. The selection of an appropriate test protocol will always pose problems as the methods needed to remove the acutely toxic components, e.g. through pH adjustment or aeration, will modify or remove other components. There is a need for developing standard tests for biological characterisation of leachates. Other types of complex industrial wastewater are regularly analysed for toxicity in Norway (e.g. Tobiesen, 1996, Efraimsen and Källqvist, 1998).

Here we summarise findings of pesticide residues in leachate samples from 3 major active Norwegian landfills, before and after leachate treatment. We also summarise monitoring of special waste leachate from tree plant production waste sites. Studies on pesticide reduction in different filter materials are also reported.

2. Materials and methods

Grab samples of leachate is routinely collected from most Norwegian landfills, and analysed for a limited number of factors. Pesticide is usually not included in the analytical program. Here selected grab samples from three Norwegian landfills have been analysed for pesticide residues. The landfills are typical municipal solid waste deposits (Mæhlum & Haarstad, 1995), and received annually an estimated total average of 38 000 tons of waste, or about 23% of the waste produced in the relevant counties. Two of the sites, Bølstad (B) and Esval (E), collect all the leachate directly from the waste body, and pipe it through a treatment system consisting of an anaerobic collection dam, and aerated treatment dam and finally through a constructed wetland filter. The last landfill, Spillhaug (S) is identical to the former, except that the leachate is discharged into an aquifer before it is collected and treated. The leachate was collected directly from the leachate pipe into clean, dark glass bottles, and transported directly to the laboratory in a cooled condition. The samples were treated with liquid extraction by dichloromethane and analysed by gas chromatography at the Pesticide Laboratory of the Norwegian Crop Research Institute, according to internal method M03 and M15 (Holen and Svensen, 1994), comprising 47 compounds (pesticides and metabolites) in 1998, and 52 compounds in 1999, see Table 1. Leachate samples are generally difficult to analyse for pesticides due to high TOC values and possible interference problems.

In addition two samples were analysed with HPLC. According to Ledin et al. (1999) 220 polar pesticides were screened by HPLC and GC-MS.

In Norway there will be picked out three reference locations nearby with similar ecological conditions (species composition, nutrient trophy level etc.) in order to study biological effects from leachate. There will be captured 20 fish for sampling on each location. Each fish will be examined for abnormalities as wound and fungus infections etc., and size and age will be registered. From each fish will there be taken tissue samples from the brain, liver, bile and kidney besides the blood samples. These samples will be tested for, vitellogenin (oestrogen effects), EROD/CYP1A/AHH (PAH`s and dioxin), antioxidant enzymes (GR,GST,CAT), PAH-metabolites, ALA-D (Pb), testosterone, and 17β-oestradiol in blood plasma. The circulating levels of progesterone, 17α- as well as the aromatase activity in the brain will be analysed and the results obtained will be compared to female brook trout from reference streams. Analysis of the enzymatic 17α-hydroxylase activity in the kidney (the enzyme which catalyse progesterone to 17α-hydroxyprogesterone) will also be included in addition to the ethoxyresorufin O-deethylase (EROD) activity in the liver of the same fish. The reason for analysing the 17α-hydroxylase activity is that this activity could serve as an earlier biomarker for endocrine disruption in the biosynthesis of steroids prior to aromatase. On each test site there will be taken a set of chemical tests of water quality and mud sediments to link biological founds to the characterisation of pollutants in landfill leaches (activity 2B). Measurement of the EROD activity is a good general biomarker for exposure to many different environmental pollutants, not only EDS(s).

Table 1. Pesticides and metabolites included in the analyses.

Pesticide	Type	$logk_{ow}$	pK_a	Sol. (mg/l)	pol.	Mol.	Location	Class
2,4-D	H	2.7	2.64	311	-	221	be	aryloxyalcaonic acid
aclonifen	H	4.4		1			r	
atrazin	H	2.5	1.7	33	-	216	fw	1,3,5-triazin
bentazon	H	5.84	3.3	570	-	240	bes, h,ww,fw	-
cypermetrin-alfa	I	7		0.01		416		pyretroid
cyprokonazole	F	2.9		93				azole
DDT	I			0.001	0	354	f	organoklorin
diazinon	I	3.3		60		304		organic P
dikamba	H	3.98	1.87	6.5	-	221		benso-acid
dichloroprop	H	1.77	3	350	-	235	bes, h, ww	aryloxyalkonic acid
dimetoat	I	0.7		23		229	fw	organic P
endosulfane	I	4.7		0.3		407		organic Cl
esfenvalerat	I	6.2		0.002		420		pyretroid
fenitrotion	I	3.4		21		277		organic P
fenpropimorf	F	2.6	6.98	4.3	+	304	b, f	morfolin
fenvalerat	I	5		<0.010		420	f	pyretroid
fluazinam	I					465		2,6dinitroanilin
fluroksypyr	H	-1.2	2.94	91	-	255		aryloxyalkonic acid
ioksynil	H		3.96	50	-	371		OH-benzonitril
iprodion	F	3		13		330	f	dicarboximid
klorfenvinphos	I	3.85		145		360		org.phosphorous
lindan	I			7		291	f	org.chlorine
linuron	H	3		81		249	ww	urea
mancozeb	F	1.75	<<0	8.4		279	h, ww, fw	acylalinin
MCPA	H	2.75	3.07	734	-	201	be, h, ww	aryloxyalkonic acid
mecoprop	H	1.29	3.78	860	-	215	bes, fw	aryloxyalkonic acid
metalaksyl	F	1.75	<<0	8.4		279	h, ww, fw	acylalinin
metamitron	H	0.83		1.7		202	ww	1,2,4-triazinon
metribuzin	H	1.58		1		214	h, ww, fw	1,2,4-triazinon
penkonazol	H	3.72	1.51	73	-	284		azole
permetrin	I	6.1		0.2		391		pyretroid
pirimicarb	I	1.7	pkb	3000	+	238		carbamat
prokloaz	F	4.38	3.8	34	-	377		azole
propaklor	H	2		613		212	ww	kloracetanilide
propikonazol	F	3.72	1.09	100	-	342	fw, f	azole
simazin	H	2.1	pkb	6.2	-	202		1,3,5-triazin
tebukonazol	F	3.7		32		308		azole
terbutylazin	H	3.04	pkb	8.5	-	230		1,3,5-triazin
tiabendazol	F		4.73	30 (ph 5)	-	201	e, fw	benzimidazole
vinklozolin	F	3		3.4		286		dicarbox-imid

Location: b=bølstad landfill, e=Esval landfill, s=Spillhaug landfill, f=forest production landfill, fw=farmland well, ww=water work, r=rivers

3. Results and discussion

Leachate samples with positive detection of pesticides have concentrations between 0.03 to 30 µg/l (Table 2). Some substances have detection in all samples, such as the phenoxy acid mecoprop and dichloroprop, and bentazone. These are also very common in surface and ground water in agricultural areas in Norway. The concentrations are however generally higher in MSW leachate.

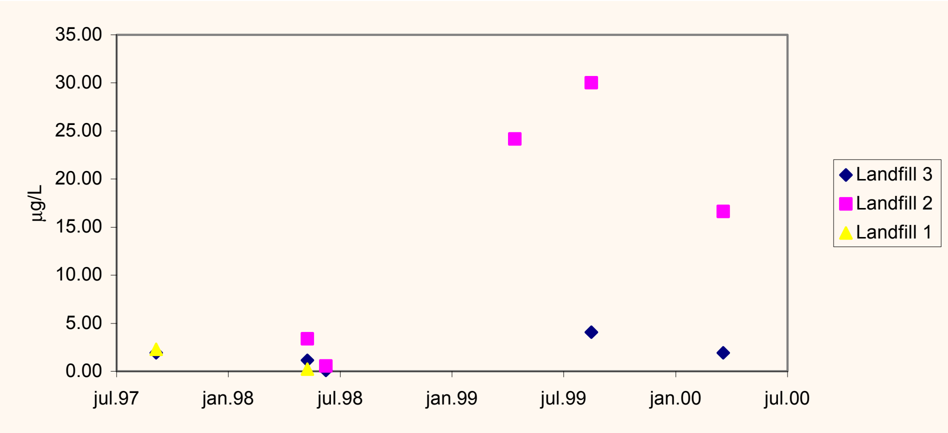

Figure 1. Pesticide concentrations (µg/l) found in MSW leachate grab samples in Norway

Table 2 shows a high frequency of detection of insecticides from tree nursery farm landfills.

Table 2. Pesticides (maximum concentrations) found in leachate or leachate polluted ground-water at waste sites in the forest trees nurseries (□g/l)

Pesticide	Max. C
fenvalerate	0,20
fenpropimorf	0,15
iprodion	0,13
lindan	3,31
propikonazole	0,25
tolylfluanid	1,80
DDT	5,00

It is seen that the obsolete pesticides lindane and DDT still occur in relatively high concentrations decades after application, and that the new insecticides and fungicides also leach to some extent.

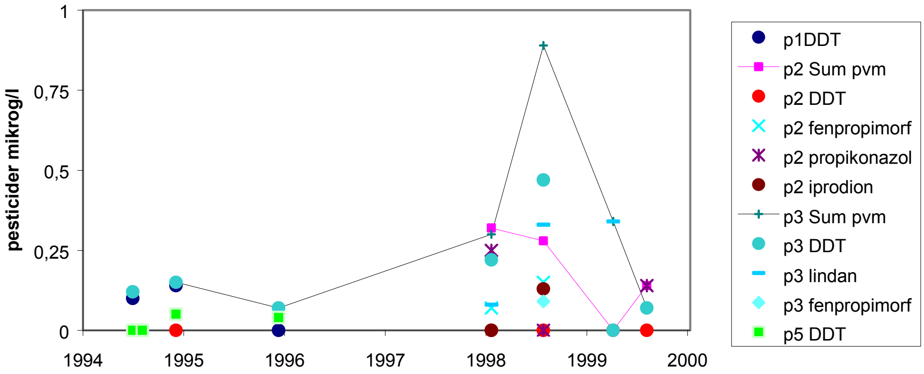

Figure 2. Time series of pesticide concentrations in groundwater polluted with leachate from a tree nursery farm.

Table 3. Pesticides properties of pesticides found in MSW leachate (Tomlin, 1994)

	pK_a*	Water Solubility (mg/l)	K_{ow} log	Ion.	Type
fenpropimorf	6.98	4.3	4	0	F
tiabendazol	nd	< 0.05	Nd	nd	F
mecoprop	3.11	620	1.26	-	H
MCPA	3.1	734	0.46	-	H
dichloroprop	3.0	350	1.77	-	H
2,4-d	2.64	311	2.6	-	H
clopyralid	2.3	118000	-1.8	-	H

* pK_a = log acid equilibrium constant, K_{ow} = octanol –water partitioning, Ion = probable ionization at leachate pH 6-8 (- = anionic), nd=no data, F=fungicide, H=herbicide

The slightly polar phenoxy acids have been significantly removed from the leachate samples in preliminary studies of removal of pesticides in bark columns, but not in filters with crushed concrete. These pesticides are relatively water soluble and mobile in soil as anions, but are retained in the bark, as seen in Figure 3.

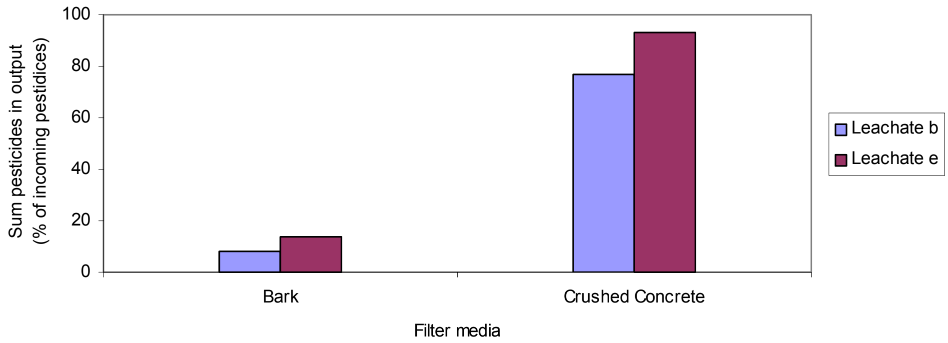

Figure 3. Removal of pesticides (pesticide in output from filters in % of incoming) from leachate type b and e by filtration through bark and concrete.

A more detailed study of binding capacities of 10 contemporary Norwegian pesticides could separate different materials as seen in Figure 4.

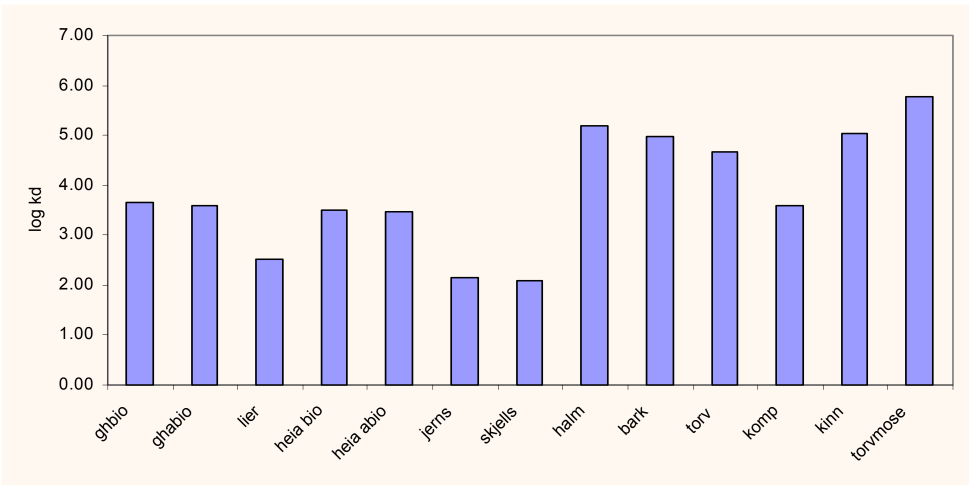

Figure 4. Mean partitioning (equals the log of the means of the partition coefficient) for filter media. (ghbio=clay soil, biological active, ghabio=clay soil sterilised, lier=clay soil, heia=organic soil, jerns=iron rich sand, skjells= carboniferous shell sand, halm=straw, bark=tree bark residues, torv=peat material, komp=compost, kinn=organic soil, tormose=fresh peat plants

The most effective filter media for pesticide removal were organic rich material such as fresh peat, straw, bark and peat.

The variation in binding properties (K_d) correlated highly with lipid partitioning (K_{ow}, r=0.71), but showed little correlation to water solubility (r=-0.15). The correlation between K_{ow} and water solubility was r=-0.31.

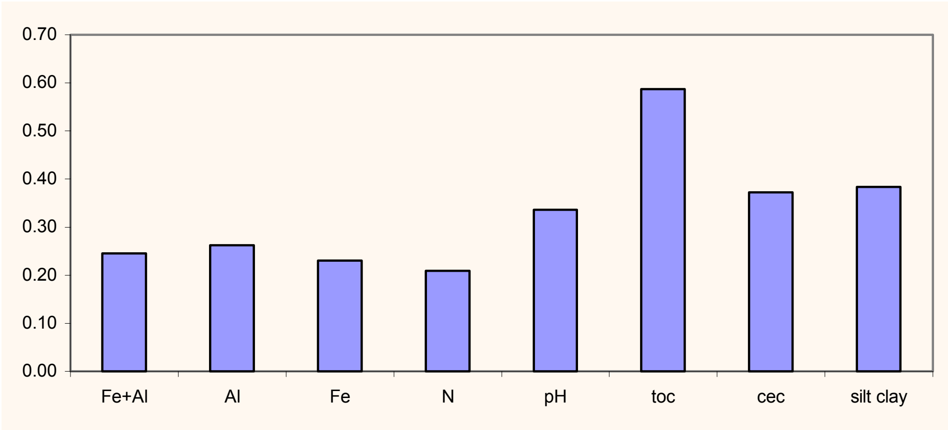

Figure 5. Correlation between measured characteristics in filter media and Kd variation.

Figure 5 shows that TOC explained most of the variation in K_d-values, followed by silt and clay content, cation exchange capacity and pH. A correlation analysis between K_d-values and chemical properties of the compounds showed that H molecules and free methyl groups were most important, see Figure 6.

Figure 6. Correlation between water solubility of pesticides and chemical characteristics of the compounds.

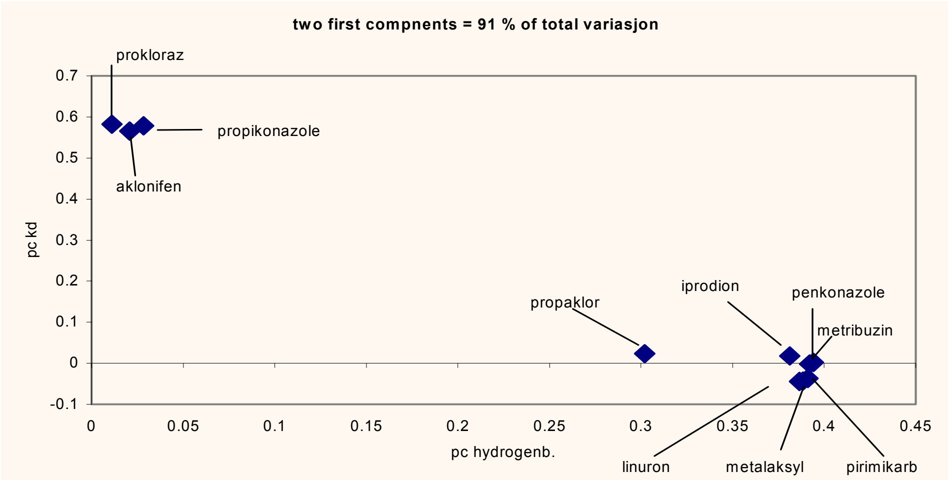

Figure 7. Principal components in Kd variation

Two principal components derived from a multivariable analysis explained more than 90% of the variation in binding properties, the K_d-values and the number of H molecules in the pesticide chemical structure, see Figure 7.

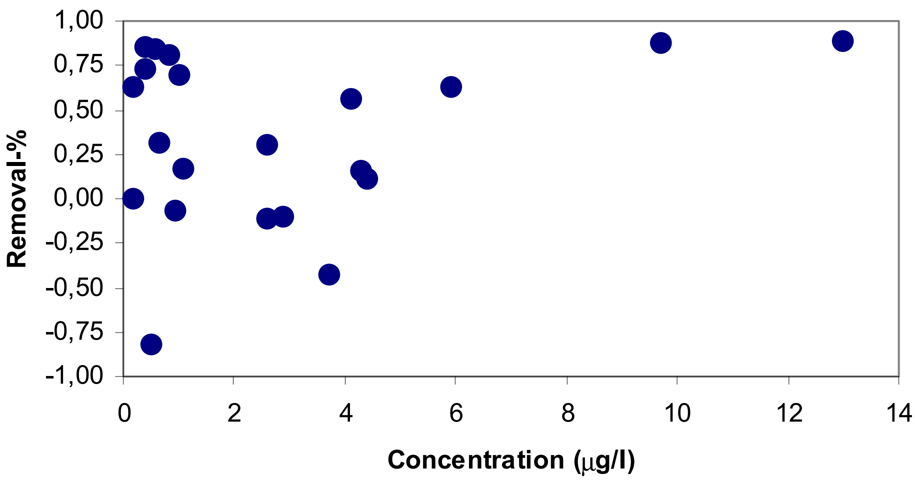

Figure 8. Removal of pesticides from Bølstad (B) and Esval (E) landfill leachates.

Figure 8 indicates that the removal of pesticides in aerobic leachate treatment systems is efficient when the pesticide concentration is above 5 µg/l. The removal starts to show large variation at lower concentrations.

The effects of environmental concentrations are usually evaluated by some kind of concentration limit, see examples in Table 4.

Table 4. Examples of concentration limits in µg/l (MAC=maximum allowable concentration (can be based both on environmental effects (e.g. $EC_{50}/100$), leaching model calculations or health effects), HA=health advisory limit, US EPA=Environmental Protection Agency, USA, WHO=World Health Organisation, FRG=Federal Republic of Germany, SSSR=Soviet Union

Pesticide	Type	Norwegian MAC	US EPA MAC (HA)	WHO MAC	FRG MAC	SSSR MAC
atrazine	H**	0,43	3	2	3	500
bentazone	H	53.5		30	10	
2,4 – D	H	14	70	30	10	100
dichloroprop	H	41			10	
glyphosate	H	12				100
mecoprop	H	52			10	2000
metribuzin	H	0.22	(200)			
MCPA	H	50		2	1	2000
diazinon	F	0,01				300
fenpropimorf	F	1.7				
iprodione	F	2.5				50
propikonazole	F	0.02				
thiabendazole	F	2.8				
lindane	I	1.6			3	3
DDT	I	0,004	nst			

The Norwegian Agriculture Inspection Service, 1996; USEPA, 1992; Beitz et al., 1994.

USEPA =Maximum contaminant level or lifetime health advisory level (metribuzin), ** H=herbicide, F=fungicide, I=insecticide, nst=no standard

4. Conclusions and recommendations

Leachate grab samples show pesticide concentrations from 0- 30 µg/l. The concentrations are both above and below environmental target concentrations such as maximum concentration levels or health advisory limits. The problem with such limits is that they are constantly changing as new experiments are carried out, and the trend is that they are reduced. Batch experiments show large variation in binding capacities but promising results for local remediation purposes for some compounds.

Studies of different filter materials showed that the most important factor for pesticide partitioning is organic carbon measured as TOC. The variation in binding properties (K_d) correlated highly with lipid partitioning (K_{ow}, r=0.71), but showed little correlation, but negative as expected, to water solubility (r=-0.15). The correlation between K_{ow} and water solubility was r=-0.31.

Grab samples from leachate treatment systems show that they can be effective in removing pesticides if the concentration is relatively high, i.e. above 5 µg/l. Studies on filter materials have given evidence of strong retention of most compounds in selected materials that can improve pesticide removal in leachate treatment systems.

Field samples of DDT shows that the mobility of highly nonpolar compounds can be much higher than anticipated, probably because the pesticide residues are contacted by organic solutes found e.g. in a typical municipal landfill leachate. Such information can also be important when considering the recycling of treated organic waste product to agricultural soils thus having different properties depending on the type of humus produced. Strong partition for hydrophobic and nonpolar compounds is often improved for compounds high in organic carbon and low in molecular oxygen. The binding properties of more polar compounds seem to depend on the number of H molecules in the compounds.

We believe that the observations of pesticides above maximum concentrations limits in the leachate show a need for local treatment of such waters, both if the water is discharged to local recipients, but even if the leachate is piped to a central water treatment plant.

Needs for further research: Since the number of samples analysed for a comprehensive list of pesticide active ingredients is low an intensified sampling is necessary. Also there is a need for better knowledge of sampling and extraction methods to avoid interference with the high TOC content in leachate. Investigation on removal methods and of course of biological effects of these compounds is strongly needed.

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