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To cite this Article Mårtensson, Lennart, Bergström, Staffan, Svensson, Britt-Marie and Mathiasson, Lennart (2007) 'Development and application of an analytical protocol for evaluation of treatment processes for landfill leachates. II. Evaluation of leachate treatment efficiency of different steps in a constructed pilot plant', International Journal of Environmental Analytical Chemistry, 87: 1, 17 - 27

To link to this Article: DOI: 10.1080/03067310600929233 URL: http://dx.doi.org/10.1080/03067310600929233

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Development and application of an analytical protocol for evaluation of treatment processes for landfill leachates. II. Evaluation of leachate treatment efficiency of different steps in a constructed pilot plant

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(Received 27 February 2006; in final form 10 July 2006)

Different methods for treatment of leachate from a municipal solid waste (MSW) landfill were tested in a pilot plant. Raw leachate was pre-treated with aeration and sedimentation, followed by several parallel individual steps such as bioremediation, chemical oxidation, ozonation, and geo-bed filters. The efficiency of different treatment steps was evaluated according to one previously developed protocol, which includes measurements of several parameters such as conductivity, pH, nutrients, chloride, metals, organic compounds, and acute toxicity before and after a treatment step. The treatment steps which showed the highest efficiency towards organic pollutants in leachate were ozone treatment and chemical oxidation. The use of an adsorption filter, a geo-bed with a mixture of peat and bottom ash with ca 10% remaining carbon, also had good effects. A combination of pre-treatment and a geo-bed filter with peat and carbon ash gave the best overall treatment results when water-quality parameters such as total organic carbon and ammonia-nitrogen were also considered.

Keywords: Treatment methods; Landfill leachate; Waste; Organic pollutants; Metals; Ammonium

1. Introduction

Existing landfills will pose a threat towards the environment for decades. A serious problem is the leaching of a wide variety of toxic compounds and nutrients from these landfills. Thus, parameters such as inorganic nitrogen compounds, chemical oxygen demand (COD), total organic carbon (TOC), biochemical oxygen demand (BOD), and heavy metals are regularly measured in leachates collected at different landfills. However, it is not sufficient just to consider the reduction of water-quality parameters

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to a low level since highly toxic organic substances may still be present. Several studies have shown the presence of a large number of different organic pollutants in landfill leachate waters [1–6]. From those measurements, we know that leachate may contain pollutants such as heavy metals, polychlorinated biphenyls (PCBs), polybrominated biphenyl ethers (PBDEs), phenols, and phthalates at concentrations which are a threat to the environment.

Often, the leachate is mixed with other types of wastewaters and then treated in a municipal wastewater-treatment plant. This leads to pollution of the sludge in this plant, which then cannot be used for soil improvements [7–10]. A better approach, promoted by EU directives, is to treat the leachate locally at the landfill. This approach leads to an increasing need for development of new efficient local treatment systems. Several studies have accordingly been presented dealing with these matters [11–16]. A few such plants are now in use in Sweden.

In this article, we present results on the efficiency of different treatment procedures including ozonation and chemical oxidation, biological treatment and absorption on filter media. The results have been obtained in a pilot plant which allows different steps to be combined. An analytical methodology previously developed for sampling and leachate characterization was used for evaluation of efficiency [17].

2. Experimental

2.1 Pilot plant

A pilot plant (figure 1) was constructed at the Härlöv Landfill on the outskirts of Kristianstad in Southern Sweden. The pilot plant in figure 1 is a flow-through system,



Figure 1. Schematics of the overall design of the pilot plant.

where the raw leachate water from the Härlöv landfill is first pre-treated by aeration combined with sedimentation followed by one of four different treatment procedures: bioremediation, ozonation, chemical oxidation by Fenton's reagent, or geo-bed filters. All treatment methods included in the study were operated with the best management procedures according to the suppliers.

For the pre-treatment (*Pre-tr.*), three similar containers were used, each with a volume of 4.9 m^3 . The total residence time in the pre-treatment step was ca 11 h.

For the bioremedation treatment (*Bio*), an aerated 2.6 m^3 tank was used with a suspended biofilm carrier (50% filling rate), according to the NatrixTM process. The biofilm carrier was a plastic carrier media, kept in suspension and continuous movement by the aeration process. In the reactor tank, the micro-organisms were growing as a biofilm on the surface of the plastic carrier. Constant pH and addition of phosphate (PO₄³⁻) ensured high activity of the micro-organisms. The retention time of the leachate in the tank was about 10 h.

When ozone (*Ozone*) was used for treatment, it was produced in an ozone generator and bubbled together with excess of oxygen through the leachate, in a counter-current flow, in a tank with a diameter of 300 mm and a height of 3 m. The flow through the tank was about 57 L h^{-1} , giving a retention time of ca $3\frac{1}{2}$ h.

Chemical oxidation with Fenton's reagent (*Chem. Ox.*) was performed in a flow-through reactor fed with leachate water. The reagent, a mixture of sulphuric acid, iron (II) (as $FeSO_4$), and hydrogen peroxide forms hydroxyl radicals which provide very aggressive oxidation and also leads to some sedimentation due to formation of solid iron oxide (Fe_2O_3). Before entering the outflow of the reactor, the pH was adjusted to ca 7. The total retention time in this treatment step was ca 2 h.

For investigation of treatment using geofilter, three different geofilter cells connected in three parallel lines were tested. Two of the filters contained the same batch of peat, and one contained this peat mixed with a batch of bottom ash (mixing ratio peat/ash 3:1 v/v). The carbon content in the bottom ash was ca 10%. The filter materials in the cells had a dry solid content of ca 40%. In two of the cells, the water was irrigated on the surface, and in the third cell, the water was introduced at the bottom. The individual filters are referred to in the text as *GeoO* (peat and overflushed), *GeoU* (peat and inflow from bottom), and *GeoCO* (peat mixed with carbon containing ash, overflushed). The three quadratic cells ($2 \times 2 \times 0.6 \text{ m}$) were designed with rubber liners at the bottom and at all four sides ensuring control of in- and outflow water volumes. The flow of leachate through the two cells irrigated from the top varied between 50 and 55 L h^{-1} , giving a retention time of about 31 h. For the cell irrigated from the bottom, the flow was somewhat lower giving a retention time of ca 40 h.

2.2 Sampling

Time integrated weekly samples were obtained with timer-controlled sampling pumps (Iwaki Co. Ltd Tokyo) according to the procedures described previously [17]. Samples were taken from the landfill leachate as well as from the inlet and outlet of each treatment procedure according to figure 1.

2.3 Analytical procedures

The analyses were performed according to the analytical procedures in the LAOUA protocol, thoroughly described by Bergström et al. [17]. In short, polluted water samples were divided into three subsamples for determination of inorganic and waterquality parameters, organic pollutants, and acute toxicity, respectively. Parameters like conductivity, pH, and oxygen were measured with direct reading instruments in direct connection with the sampling procedure as samples were taken. Parameters like TOC, BOD₇, P-tot, PO₄³⁻, N-tot, NH₄⁺, NO₃⁻, NO₂⁻, and a number of metals-Cd, Cu, Cr, Pb, Zn, and As-were analysed at an accredited laboratory using Swedish standard methods. For organics analysis, each sample was divided into two sub-samples: one for polar compounds and one for non-polar compounds. Phenolic compounds, used as markers for polar compounds, were determined using the fully automated analytical system described previously [17], consisting of a supported liquid membrane extraction unit (SLM) connected to an HPLC-DAD (diode array detection) instrument. Non-polar pollutants, such as PBDEs, PCBs, and phthalates, were determined by GC-ECD after work-up using solid-phase extraction (SPE) discs and supercritical fluid extraction (SFE) as described by Bergström et al. [17]. Here, the total concentration of pollutant was determined, i.e. both the free and the particle-bond fraction. To account for acute toxicity in the leachate, a new toxicity test was utilized [18] based on the crustacean Artemia salina. This organism tolerates high concentrations of chloride ions, which is essential for measurements in leachate waters that generally have high concentrations of such ions.

An essential component of the measurement strategy, as described in the previous paper [17], was the utilization of difference measurements, i.e. concentrations of different species were measured at the inlet and outlet of the different treatment steps. Furthermore, efficiency estimations for removal of organic pollutants were based on changes in peak area concentrations for certain marker compounds.

2.4 Statistical

The data from all identified and quantified compounds (organic as well as inorganic, and sum parameters) were treated with a multifactor analysis of variance (ANOVA) with treatment and dates as factors (Statgraphics plus, Statistical Graphics Corp. Rockville, MD). Multiple range tests were done according to Fisher's least significant difference (LSD) procedure in order to determine whether treatments differed significantly from each other. A confidence level of 95% was used for all statistical calculations.

3. Results

3.1 Chemical composition of the landfill leachate

Before any investigations of the efficiency of a certain treatment procedure for any leachate, it is important that this leachate is well characterized. The composition of leachate water used in the pilot plant studies collected from the Härlöv landfill site is summarized in table 1. The tables contain values of a number of parameters over

	Unit	Average 1993-2002	8-Week average during the tests
Mercury, Hg	$\mu g L^{-1}$	< 0.1	
Zinc, Zn	$\mu g L^{-1}$	63 (55)	60 (24)
Chromium, Cr	$\mu g L^{-1}$	15 (6)	15 (27)
Nickel, Ni	$\mu g L^{-1}$	16 (5)	
Copper, Cu	$\mu g L^{-1}$	20 (15)	5 (29)
Lead, Pb	$\mu g L^{-1}$	< 50	3.1 (67)
Cadmium, Cd	$\mu g L^{-1}$	<5	0.22 (75)
Calcium, Ca	mgL^{-1}	368 (76)	
Arsenic, As	$\mu g L^{-1}$	< 50	5.9 (10)
Phenol, total ^a	$\mu g L^{-1}$	57 (46)	
Phenolic compounds ^b	$\mu g L^{-1}$		151 (27)
PCBs	$ng L^{-1}$	<100	13 (22)
pH	e	7.2 (4)	7.1 (3)
Conductivity, 25°C	$\mathrm{mSm^{-1}}$	722 (32)	729 (3)
Suspended solids	mgL^{-1}	144 (56)	
Dry substance, TS	gL^{-1}	5.1 (3)	
Chemical oxygen demand, COD _{Cr}	mgL^{-1}	661 (43)	
Biochemical oxygen demand, BOD ₇	mgL^{-1}	27 (48)	29 (33)
Total organic carbon, TOC	mgL^{-1}		128 (10)
Nitrite-nitrogen, NO ₂ -N	mgL^{-1}	0.036 (114)	0.03
Nitrate-nitrogen, NO ₃ -N	mgL^{-1}	0.42 (161)	3.2 (29)
Ammonium-nitrogen, NH ₄ -N	mgL^{-1}	248 (28)	236 (5)
Nitrogen, total-N	mgL^{-1}	274 (29)	246 (12)
Phosphorus, total-P	mgL^{-1}	1.3 (30)	1.1 (34)
Boron, B	mgL^{-1}	1.7 (30)	
Chlorine, Cl ⁻	mgL^{-1}	1552 (30)	1243 (3)
AOX ^c	$\mu g L^{-1}$	327 (47)	~ /

Table 1. Chemical and physico-chemical composition of leachate water from Härlöv landfill (mean values), with relative standard deviation (RSD) percentage values shown in parentheses.

^aDetermined as the phenol index.

^bAnalysed according to Bergström et al. [17] as the sum of four analysed phenols.

^cAdsorbable organically bound halogens according to ISO 9562:2004.

a 9-year period with sampling every sixth month, obtained from the control programme of the waste-management company.

The values in table 1 of many parameters in the leachate used in our pilot plant study were well in accord with average values over the 9-year period. However, the values for copper were significantly lower and the value for nitrate-nitrogen significantly higher during the test period. The leachate in table 1 has a composition typical for an aging methanogenic landfill leachate. The leachate was relatively particle-free and had a dark olive-green to grey colour.

3.2 Evaluation of the efficiency of different treatments

The results from the analysis of chosen sum parameters, metals, and organic compounds according to the LAQUA protocol are collected in table 2. The relative standard deviation values presented are based on three to eight determinations.

3.2.1 Water-quality parameters. Table 2 shows that in the pre-treatment procedure, the pH in the leachate water increased from 7.1 to 8.6, and the conductivity decreased, probably due to sedimentation. The variation in pH after the other treatment steps

I able 2. Average values based on three to eig	ght determinatio	ons of chosen] percentage v	parameters before alues shown in p	and after treatiarent	ment (mean valu	es), with relativ	ve standard dev	ation (KSD)
Parameter	Raw	Pre-tr.	Chem. Ox.	Ozone	Bio	GeoU	GeoO	GeoCO
Metals								
Zinc, Zn ($\mu g L^{-1}$)	60 (24)	51 (21)	84 (12)	72 (26)	425 (5)	55 (24)	55 (15)	60 (19)
Chromium, Cr ($\mu g L^{-1}$)	15 (27)	11 (20)	9 (33)	10 (22)	15 (38)	10 (17)	11 (37)	7 (45)
Copper, Cu $(\mu g L^{-1})$	5(29)	5 (38)	10 (49)	25 (28)	10 (17)	9 (62)	5(38)	9 (32)
Lead, Pb (µg L ⁻¹)	2.2 (28)	1.7 (4)	1.1 (26)	1.7 (17)	1.9(27)	3.0(30)	3.4 (34)	2.1 (22)
Cadmium, Cd ($\mu g L^{-1}$)	0.2 (75)	0.2 (73)	0.4(65)	0.2(68)	0.4(46)	0.5(58)	0.4(40)	0.5(31)
Arsenic, As $(\mu g L^{-1})$	5.9 (10)	5.3 (16)	3.6 (52)	5.2 (19)	6.2 (6)	6.2 (26)	5.6 (21)	15 (12)
Organic compounds ^a								
Phenolic compounds (sum of the	151 (27)	108 (57)	25 (112)	2.9 (76)	72 (40)	64 (29)	25 (18)	27 (81)
four compounds below) ($\mu g L^{-1}$)								
Phenol ($\mu g \hat{L}^{-1}$)	3.5 (72)	3.8 (45)	2.8 (142)	0.6(283)	67 (38)	4.3 (73)	7.2 (48)	20 (125)
Paracresol ($\mu g L^{-1}$)	118 (24)	76 (73)	21 (105)	1.5 (53)	3.1 (26)	40 (36)	9.9 (35)	4.3 (34)
2,4-Methyl phenol ($\mu g L^{-1}$)	4 (75)	6.2 (28)	< 0.5	0.6(263)	< 0.5	5.1 (28)	1(110)	< 0.5
4-Chloro, 3-methyl phenol ($\mu g L^{-1}$)	25 (47)	22 (31)	0.5 (265)	0.3(283)	1.4(141)	15 (34)	6.7 (42)	2.1 (15)
PCBs (sum of 28 congeners) (ng L^{-1})	13 (22)	8.6 (52)	4.9 (67)	3.4 (50)	8.4 (19)	5.5 (43)	6.9 (74)	6.8 (76)
BDE-153 ($\log L^{-1}$)	14 (69)	16(69)	11 (118)	13 (78)	s.e. ^b	28 (85)	12 (71)	14 (70)
BDE-183 $(ng L^{-1})$	107 (43)	91 (45)	42 (51)	58 (62)	63 (37)	92 (71)	55 (34)	60 (45)
Unidentified non-polar organic compounds	100	52 (85)	13 (84)	20 (47)	19 (128)	24 (94)	26 (129)	17 (130)
(sum of 10 peaks, normalized) (%)								
Sum parameters								
hd	7.1 (3)	8.6 (2)	7.6 (9)	8.0 (3)	7.7 (2)	7.5 (7)	7.8 (4)	7.6 (2)
Conductivity, 25° C (mS m ⁻¹)	729 (3)	632 (2)	781 (5)	629 (2)	544 (7)	599 (6)	607 (3)	629 (3)
$BOD_7 (mg L^{-1})$	29 (33)	14 (58)	31 (23)	29 (44)	s.e.	24 (47)	10(40)	5 (56)
TOC $(mg L^{-1})$	128 (10)	108 (12)	90 (17)	57 (26)	94 (9)	198 (22)	129 (28)	64 (12)
$NO_{3}-N (mg L^{-1})$	3.2 (29)	2.2 (39)	4.4 (18)	24 (32)	150 (27)	< 0.2	11 (39)	68 (30)
$NH_{4}-N (mg L^{-1})$	236 (5)	227 (8)	211 (11)	201 (13)	5 (76)	215(1)	182 (24)	108 (23)
Total-N (mg L^{-1})	246 (12)	231 (11)	224 (6)	228 (5)	175 (13)	223 (14)	218 (10)	198 (16)
^a Analysed according to Bergström <i>et al.</i> [17]. ^b s.e.: sampling error.								

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was small. Most notable for BOD and TOC is their large decrease after the GeoCO treatment step. This might be related also to a biological activity, which is obvious when considering the decrease in NH_4 -N and increase in NO_3 -N. The TOC increase for GeoU was probably due to release of particulate matter from the peat material when feeding the leachate water from the bottom of the filter bed. This approach creates another flow pattern in the peat with a larger risk for channel formation. Thus, this mode of applying leachate water should be avoided. This point of view is supported by the good results obtained in a full-scale plant with overflow irrigation (P. Kängsepp, personal communication, 2006).

The behaviour of nitrogen compounds in table 2 is quite different in the different treatment procedures. This is illustrated in figure 2, which shows the concentrations of total nitrogen, ammonium, and nitrate in the different treatments.



Figure 2. Concentrations of nitrogen, ammonium, and nitrate analysed after the different treatment steps (abbreviations as in section 2).

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The highest treatment efficiency in figure 2 is obtained by using bioremediation, which has reduced almost all ammonium and increased nitrate. This is of course expected, since this biological treatment is mainly focused on denitrification. Very interesting, however, is the reduction of ammonium in GeoCO and corresponding nitrate increase in the same treatment step. The changes in this study were more pronounced the later the samples were taken, indicating that the biological activity increases with time. Thus, a geofilter can obviously act as a combined filter—biodegradation step. The microbial activity using geofilters also seems to lead to a decomposition of organic matter. An indication of this is that TOC and BOD values decrease. In GeoU, anaerobic conditions are probably achieved, reflected by the low NO_3 -N value due to denitrification processes.

3.2.2 Metals. The values of the chromium and lead concentrations in table 2 are lower after pre-treatment than in the raw leachate at the 95% significance level. This indicates some co-precipitation with iron. The concentrations of other metals are hardly influenced by the different treatment steps. In some cases, the concentrations were even significantly increased. The concentrations of zinc and copper increased in the Bio and Ozone steps, respectively, which probably depends on their release from materials in the tank construction. The increase in arsenic in GeoCO is most probably due to contamination from the bottom ash, but the value represents a moderate risk according to Swedish environmental quality criteria [19, 20] for this type of water. However, all metal values are still very low in this landfill leachate after any treatment procedure, and in this respect leachate does not present any threat to the surroundings.

3.2.3 Organic compounds. Ozone-treatment or chemical oxidation gives the best reduction of phenolic compounds and non-polar compounds, but also a geofilter with peat and carbon containing ash is quite efficient. The lower treatment efficiencies of geofilters with only peat show that a more efficient adsorbent than peat is needed for organic compounds. The reason for lower efficiency in the bioremediation step seems to be that phenolic compounds are only partially degraded, the final product being phenol, which appears at a high concentration in the outlet stream from this treatment step. The low level of PCBs (a few ppt) in the raw leachate decreases a further 20–50% in the pilot plant. The pre-treatment procedure contributes significantly at the 95% level to this decrease, but all the other subsequent steps, except Bio, contribute to this reduction For Chem. Ox., Ozone and GeoU, the difference is significant at the 90% level. For the 10 unidentified non-polar compounds, there is a significant reduction at the 95% level in the pretreatment step to ca 50% of the values in the raw leachate and a further reduction to ca 20% of the original value in the subsequent step. For the Chem. Ox., Bio, and GeoCO steps, the reductions are significant at the 95% level.

3.2.4 Acute toxicity. Table 3 shows the results from acute toxicity measurements using *A. salina* on unfiltered leachate water.

In general, the toxicity for the raw water was low using the *A. salina* test. A significant reduction in acute toxicity (higher LOEC) was noted using the bioreactor and geofilter with peat and carbon ash, but also the chemical oxidative treatment procedures indicate a lower toxicity after treatment. Changes in toxicity of the leachate

	30/05/2000	06/06/2000	13/06/2000	19/06/2000	27/06/2000	04/07/2000
Raw	68	79	75	75	75	75
Pre-tr.	68	68	75	75	75	75
Chem. Ox.	79	79	n.a. ^b	79	79	79
Ozone	79	79	75	79	75	75
Bio	n.a.	n.a.	n.a.	n.a.	91	91
GeoU	79	79	75	75	75	75
GeoO	79	79	75	79	79	75
GeoCO	86	79	79	86	86	>91

Table 3. Acute toxicity measured using the A. salina test system.^a

^aThe response of the test organisms are given, according to the dilution of leachate water in steps of 45, 68, 75, 79, 86, and 91%, as lowest observed effect concentration (LOEC).

^bn.a. not analysed.

during the measurement period (6 weeks) were small. The variability of the values in time in table 3 is small. The RSD values vary between 3 and 6% for all treatments. The variability within samples was determined from the reference solution. The relative standard error of the mean from 10 observations was 5%. Thus, fairly small deviations in toxicity can be measured with the methodology used.

3.2.5 General observations. After the first pre-treatment step (aeration), the leachate had turned brown depending on oxidation of iron (II) to iron (III), and contained large amounts of particles. However, a large number of these particles precipitated during pre-treatment in the sedimentation tanks, where the residence time was ca 11 h. Typical for the ozone degradation steps was that the leachate water leaving the ozone degradation tank was clear and light yellow with a green tone and with a reduced smell compared with the inflow leachate water. The choice of materials for equipment used in the treatment procedure might be important. For example, higher values were found at the outlet than at the inlet for zinc after bioremediation, and copper after ozone treatment (in tanks and with valves which contained zinc or copper). In our case, the increase in concentrations is not expected to give toxic problems, but one should always be alert to the possibility of toxic material effects. In general, there is a concentration reduction of organic compounds in the leachate after the different treatment steps. This reduction is significant in all steps for the sum of polar phenolic compounds. For non-polar compounds (both PCBs and 10 unidentified compounds), there is a significant decrease in the pre-treatment step, in the chemical oxidation steps (Chem. Ox. and Ozone), and in the geofilter with a mixture of peat and carbon containing ash.

4. Discussion

Previous studies have shown that for efficient treatment results of municipal solid-waste leachate with its complex mixture of nutrients, heavy metals, and both degradable and persistent organic substances, a combination of different treatment methods is necessary. Thus, access to a pilot plant in which different treatment steps and combinations of such steps can be investigated is essential to obtain the information

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needed for construction of efficient local treatment systems that consider both sitespecific requirements and cost-effective aspects. In our study, we have shown that a good evaluation of what happens during different treatment procedures can be obtained by measuring the analytical parameters given in the LAQUA protocol. The chosen markers give a good overall picture of changes in the concentrations of pollutants. The appropriate use of model compounds with a wide range of properties is expected to give a good picture of the general behaviour of other similar and unknown substances. In the case of organic pollutants, a treatment plant that can reduce the concentrations of such model compounds would in most cases be expected to efficiently reduce the concentrations of other unidentified organic pollutants to a safe level as well. The choice of model compounds is not easy and should be based on information obtained from characterization of the raw leachate.

Our results show that a simple pre-treatment with aeration and sedimentation should always be included in a treatment plant. In spite of a relatively low retention time, most of the parameter values and compound concentrations decreased significantly. The same performance has also been found to be even more accentuated in a full-scale plant [21], based on natural treatment with serially connected retention ponds followed by an area with fast-growing willow, irrigated by the effluent from the last pond in the series. The retention time in this system is about 1 year.

Considering the costs, the combination of pre-treatment with a step based on a geofilter with peat and carbon containing ash seems to be a good choice for leachate treatment. The two oxidation methods (Chem. Ox. and Ozone) yield good reduction of all kinds of organic pollutants and are in this respect more efficient than the suggested approach. However, both these methods are more expensive to use, technically more advanced and demand a continuous process control to prevent an excess of oxidation agents from entering the environment. The use of Bio does not seem to be efficient for very persistent compounds such as PCBs and may lead to the formation of unwanted toxic degradation products. However, the use of Bio as a subsequent final step after the main treatment to reduce ammonia to nitrate is certainly a good approach. Interestingly, as found in this investigation, the geofilter might also reduce ammonia to nitrate. This has also been observed in a full-scale plant based on a similar geofilter (P. Kängsepp, personal communication, 2006).

The pilot plant in our study was built on the actual landfill site. This approach is better when setting up soil-plant irrigation steps, since it is relatively easy to establish vegetation in this way with willow or reed beds, for example. However, a more versatile approach could be to design and construct a movable pilot plant. This has been tested with good results at an industrial landfill site (P. Kängsepp, personal communication, 2006). The possibility of running a pilot plant in parallel with a later constructed full-scale plant based on the same concept is valuable, and this may help to foresee problems before they occur in the full-scale plant.

Acknowledgements

The authors acknowledge SweBaltcop for the financial support and Kristianstad Renhållnings AB (KRAB) for financial support and interest. Dahn Rosenqvist,

Rosenqvist Mekaniska AB, Margaretha Svensson, and Ulo Päbo are acknowledged for technical assistance.

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