

Fate of Heavy Metal Contaminants in a Former Sewage Treatment Lagoon, Hancock County, Ohio

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A Former sewage sludge lagoon, thoroughly mixed and abandoned in 1999, was analyzed to determine the subsequent mobility of heavy metals and their relationship to the dominant plant species at the site. ICP analyses of soils taken with respect to depth relative to the root zone and association with either common ragweed or stinging nettles revealed noticeable differences. In the five years since abandonment, the heavy metals have been stratified due to leaching and plant uptake, with the majority of metals showing higher concentrations at more shallow depths. Although stinging nettles can uptake higher concentrations of heavy metals into their plant parts, the soils do not have an associated reduction in heavy metal content. As the plants die in the fall, the heavy metals are returning to the soil as vegetation decays. Ragweed is less capable of incorporating the heavy metals into its biomass and is found associated with overall higher heavy metal concentrations in the soil around its roots. Thus, some other process is causing the retention of heavy metals in these soils. The lower concentrations in the soils beneath the root zones, which were reported to have been initially identical in composition to the shallower soils, are likely due to leaching.

Keywords Stinging nettles, common ragweed, heavy metal contamination, sewage lagoon

Introduction

Heavy metal pollution is one of the most pervasive and serious environmental problems facing society. As a result of urban, agricultural, industrial, and military activities, the world is faced with a legacy of contaminated soil and water. The largest sources of metal pollution are mining and smelting, but substantial quantities of trace metals also are released as the result of coal combustion, refuse and sewage sludge disposal, and as waste products of electronics or plating industries. Many areas in Ohio have been contaminated with trace metals presenting serious environmental problems that are impediments to economic development.

Remediation of soils contaminated with heavy metals is complicated and expensive, partly because the contamination is often diffuse and covers large areas. Recently, plants have been used to remediate contaminated sites. Certain native and traditional crop plants have been identified that concentrate metals to levels that are toxic to most species. These

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relationships include copper uptake by sunflower (*Helianthus annuus*) and Indian mustard (*Brassica juncea*); and zinc uptake by fescue (*Festuca rubra*), barley (*Hordeum vulgare*), oat (*Avena sativa*), alfalfa (*Medicago sativa*) and *Brassica* spp. (Dushenkov et al., 1995; Ebbs and Kochian, 1997; Salt et al., 1997; Ebbs and Kochian, 1998; Gardea-Torresdey et al., 1998). However, other research has shown that high metal concentrations can inhibit the growth of some plants. For example, high copper inhibited the growth of *Thlaspi caerulescens* (Lombi et al., 2001). High concentrations of zinc and copper inhibited the uptake of iron and manganese in certain *Brassica* species, resulting in poor growth (Ebbs and Kochian, 1997).

The Fostoria Waste Water Treatment Plant, located in Hancock County in northwest Ohio, has a 3.5-hectare lagoon containing digested sewage sludge from an aerobic digester, filled from 1950 through 1994 (Figure 1). Any industry could dump into the septic system when the lagoon was first opened. During the 1960s and 1970s, the industries included those using heavy metals, manufacturers of spark plugs, and a foundry. Also, excess concrete was dumped into the lagoon. The wastes were mixed several times with bulldozers in subsequent years, although prior to mixing the southeast quadrant was reported to have the highest metal concentrations, specifically copper and manganese. In 1999, the city of Fostoria had soil samples analyzed by two consultants to determine whether the site could be used as an extension to the neighboring park system and as an outdoor lab for local schools. However, field concentrations exceeded 1999 CFR 503 regulations (Table 1). Since 1999, the site has been allowed to revert to nature.

A study of metal concentrations in the soil horizons and indigenous plants was conducted from 2000 to 2005. Herein, we report soil results from 1999 through 2005. In an

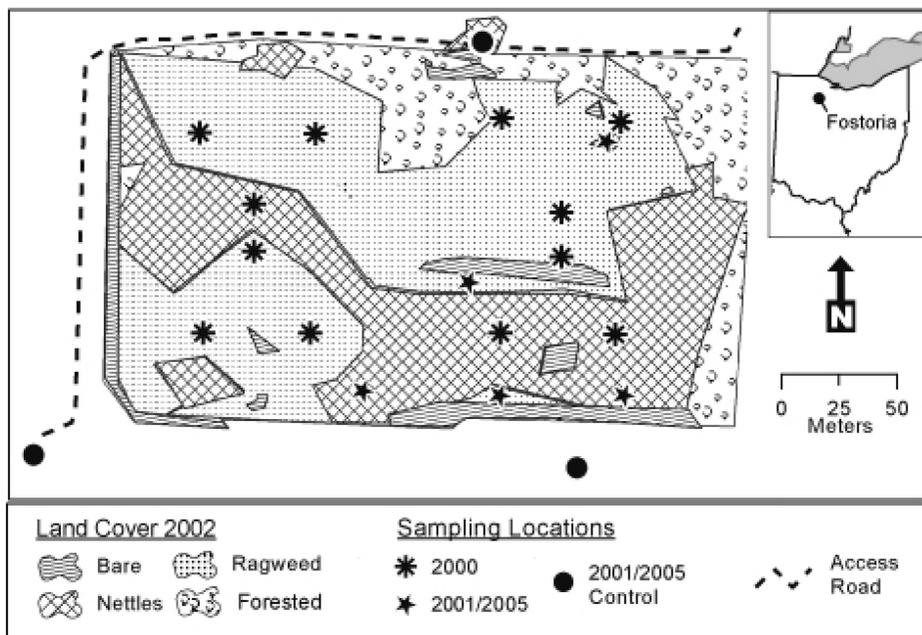


Figure 1. Location map of the Fostoria Waste Water Treatment Plant, Hancock County, Ohio, showing sample locations used throughout this study (modified from the original by Dr. Donald Stierman to include only these dominant species).

Table 1

External consultant data for Fostoria Sludge Lagoon analytical comparison—1999, with current EPA disposal regulations

Parameter	1999 CFR 503.23 Subpart C Limit for Surface Disposal	Average value reported by two labs*	2005 Compliance parameters Table 3 of 40 CFR 503.13**
	(mg/kg dry weight)		
Arsenic	39	2.87	41
Cadmium	Not listed	2.14	39
Chromium	260	43.41	Not listed
Copper	Not listed	2554	1500
Lead	Not listed	132	300
Manganese	Not listed	431	Not listed
Mercury	Not listed	0.19	17
Molybdenum	Not listed	8.30	75
Nickel	270	73.7	420
Selenium	Not listed	0.10	100
Zinc	Not listed	753	2800

*Data were obtained from Northwest Ohio EPA Solid Waste Division files. Data were compiled by EPA from MidWest Environmental Consultants, Inc. (1999) and Ginosko Laboratories, Inc. (1999). Total number of samples averaged is unknown but is at least eighteen. Standard Deviations are not given in report.

**40 CFR 503.13 Current Regulations for metal concentration in compressed cake (~25% solids) for land application.

initial evaluation, soils from the four quadrants were tested to provide an overall picture of heavy metal contamination at the site. During the subsequent summers, soils and plants were sampled repeatedly, focusing on soil horization, rooting depth and associations between dominant plant species and surrounding soil. Mobilization and redistribution of the once spatially uniform metal concentrations and their association with the indigenous plant populations was investigated.

Materials and Methods

The site is organized as quadrants over a total land area of about 3.5 hectares. All quadrants were built at the same time. Although original plans for the lagoons were destroyed by a previous owner, it is believed that dikes were created between the four cells. In 2000, three equally spaced cores from each quadrant were obtained by hand augur and/or a Mobile B-30S trailer-mounted drill rig with 1.5 meter solid stem augurs (Figure 1). Individual cores up to 3.0 m in length were sub-sampled at 0.25 m intervals for analysis of basic soil properties and heavy metal concentration (Table 2). These soils range from sandy loam to silty loam, have a neutral pH ranging from 6.5 to 7.6, and cation exchange capacities ranging from 14 to 24 milliequivalents/100 g. Two control sites within the same soil series were selected based on the presence of dominant plant species. These soils were processed identically to the samples collected from the soils.

An inventory of plant species found on site was conducted in 2000 Spatial distribution of plant species were mapped using GIS locations input into ArcGIS Arcinfo 9.1. Figure 1

Table 2

Basic soil properties* from the Fostoria Waste Water Treatment Plant sludge lagoons (2000)

Depth (m)	N	CEC		pH		Organic matter		Texture
		meq/100 g	St. Dev.	pH	St. Dev.	%	St. Dev.	
0.25	7	19.81	6.18	6.91	0.31	7.84	1.28	sandy loam
0.50	8	21.89	1.91	6.70	0.42	8.00	0.97	loamy sand
0.75	7	23.87	4.52	6.53	0.74	5.97	2.34	loam
1.00	8	23.83	4.21	6.70	0.63	6.45	4.24	silt/clay loam
1.25	8	21.38	3.25	6.90	0.62	5.38	3.62	clay loam
1.50	7	19.86	3.25	6.97	0.46	4.49	3.46	silt/clay loam
1.75	1	20.50		7.20		5.50		silt loam
2.00	1	19.30		7.40		2.70		silt loam
2.25	1	14.30		7.60		1.50		clay
2.50	1	18.10		7.40		1.90		silt loam

N = number of samples.

*Analyses were performed by Spectral Analytical Laboratories, Washington Courthouse, Ohio

is a modified version of the more detailed map. Based on the dominance of stinging nettles (*Urtica dioica*) and common ragweed (*Ambrosia artemisiifolia*), these plants were sampled for heavy metal analysis. Plants were collected; extraneous soil was removed prior to air-drying and separation into leaves, roots and shoots.

Samples (0.1 – 0.5 g) of air-dried, sieved (2mm) soils and/or plants were processed for heavy metal analysis using a modification of USEPA Method 3051. The samples are digested in concentrated nitric acid in a microwave digester programmed with a 15 min. ramp, 200°C, and 105 min. hold (US EPA, 1986) and analyzed by ICP-OES (IRIS Intrepid 2-XSP, ThermoElectron Corp., Boston, MA). ICP analyses were performed gratis at the USDA-ARS research facility in Toledo, Ohio. Metals chosen for analysis were based on earlier reports by private consultants and included chromium, zinc, cadmium, lead, nickel, manganese, molybdenum, and copper.

National Bureau of Standards reference soil samples and tissue samples of tomato and spinach leaves (National Institute of Standards and Technology) were used for quality control as both internal spiking and external references. Solutions of known concentrations in like matrices were used to calibrate instrumentation and quantify sample concentrations.

All statistical analyses were performed using SigmaStat for Windows Version 3.11 software, developed by Systat Software, Inc. The dependent variable (metal concentration in the 1999 soil samples) was analyzed using two-way analysis of variance with quadrant and depth as main effects and the interaction of the two terms. Comparisons of means were analyzed for significance (at $p < 0.05$) using the Holm-Sidak Pair-wise Multiple Comparison Procedure. Soil data from 2000–2005 were analyzed to determine whether control sites were significantly different from contaminated sites. For the purposes of analyses, all depths greater than 1.5 meters, which is well below the root zone, were pooled. A two-way analysis of variance on the significance of heavy metal content with respect to quadrant and depth (within the root zone and at least 10 centimeters beneath the root zone) and dominant plant type (stinging nettles or ragweed) for the contaminated samples (no controls are included) are presented in Table 3. P-values < 0.05 are significant at the 95% confidence level. The dominant plant type at our reported sampling locations remained fairly stable as mapped in

Table 3
ANOVA Statistical P-values for significance of quadrant, depth and interaction for heavy metal concentration in soil at the Fostoria Waste Water Treatment lagoon

Metal	Factor	Degrees of Freedom	Sum of squares	P-value
Chromium	Quadrant	3	99744.986	0.566
	Depth	5	808757.554	0.013
	Interaction	15	2038074.469	0.769
Zinc	Quadrant	3	9126305.842	0.003
	Depth	5	17586098.798	<0.001
	Interaction	15	23005138.131	0.006
Lead	Quadrant	3	279285.911	0.055
	Depth	5	997299.374	<0.001
	Interaction	15	503844.243	0.484
Nickel	Quadrant	3	17251.928	0.606
	Depth	5	172989.968	0.007
	Interaction	15	88130.749	0.830
Manganese	Quadrant	3	3644454.939	<0.001
	Depth	5	527654.831	0.070
	Interaction	15	1690410.948	0.014
Copper	Quadrant	3	122640570.702	0.018
	Depth	5	235464101.626	0.003
	Interaction	15	102077496.881	0.841

Data were analyzed using Two-way ANOVA in SigmaStat for Windows Version 3.11.

Figure 1 over the five years of this study. Furthermore, because the plant type was not noted at the time of the initial 1999 sampling, those data are not included in this final statistical analysis.

Results and Discussion

Concentration of heavy metals in soils sampled in 1999 (Figures 1 and 2, Table 1) exceeded current EPA 503 regulations for concentrations allowed in compressed cake (sludge) applied to land. Copper was the most concentrated heavy metal exceeding standards at the Fostoria site, whereas lead, nickel and zinc exceeded standards less frequently. Cadmium was absent in most samples, and no trend with depth was observed when present. Typically, concentrations of zinc, copper, chromium, and nickel decreased, and manganese increased, with increasing depth.

Concentration of metal content varied among quadrants and with depth (Table 3). The interaction term between quadrant and depth is only significant ($p < 0.05$) for zinc and manganese, implying that the differences in concentration in any quadrant are dependent upon the depth for these metals. For both metals, concentrations are greater in the southeast than the other three quadrants. Concentrations of zinc were greater in shallow than deep sediments whereas manganese occurred in greatest concentrations in the more chemically reduced deeper horizons.

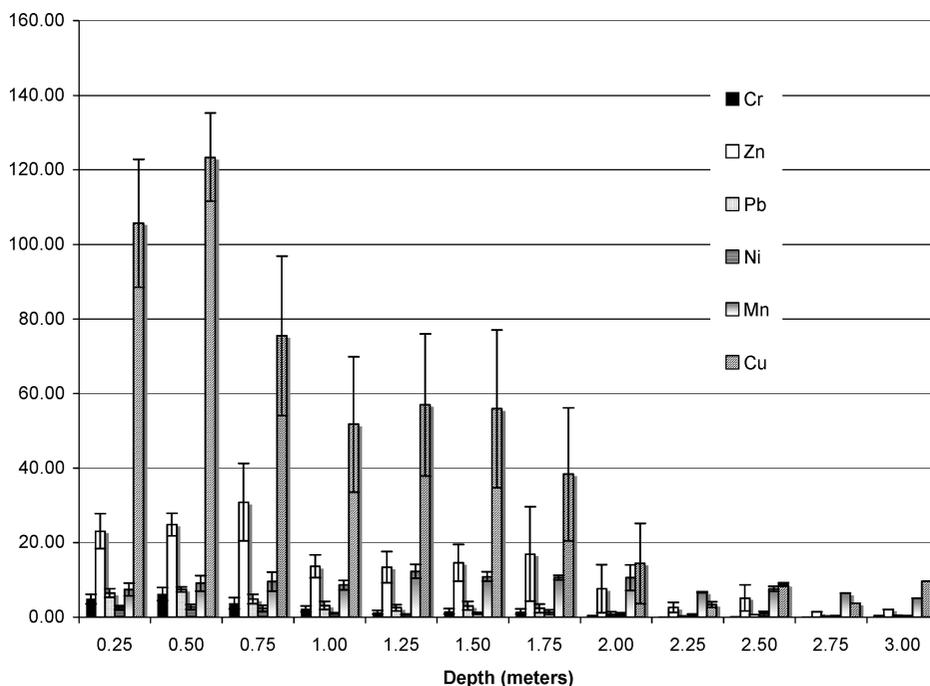


Figure 2. Average heavy metal concentrations (mg/kg) in soil with depth at the Fostoria waste water treatment plant, Hancock County, Ohio. Samples were taken in 1999.

Distributions of chromium, lead, nickel and copper vary by depth. Concentrations of copper and lead also varied by quadrant but not nickel and chromium. Considering that the sediments have been mixed numerous times prior to 1999, metal mobility must have occurred since that time. Quadrant was only significant for copper, with lead showing significance at $p = 0.055$, and the northeast quadrant showing the highest concentrations for both metals. However, only the NW and SW quadrants are significantly distinct for copper.

In general, the initial impression from field personnel that the southeast quadrant would show the highest concentrations was only partially confirmed, assuming that no loss from the profile had occurred within this one year since previous analysis. However, and most importantly, metal concentrations within an area that had been uniform throughout its thickness less than one year previous to our sampling (all soil mixed at once, from one source) were found to be non-uniform, especially with depth.

Plant distribution at the field site is heterogeneous and dominated by two major species: stinging nettles (*Urtica dioica*) and common ragweed (*Ambrosia artemisiifolia*) (Figure 1). Heavy metal concentrations in these plants are presented in Table 4. For all three plant parts, a significant difference is found between plants on the contaminated sites versus those on the control sites, while there is no difference between the contaminated sites ($p > 0.05$). Stinging nettles accumulated the highest concentrations of heavy metals compared to the other plants at the site and seemed to accumulate all of the metals analyzed at a relatively high concentration. This compares favorably with similar analyses by Tack and Verloo (1996), who noticed that heavy metal concentrations in stinging nettles were due to high soil concentrations and unrelated to soil physical parameters. However, ragweed was found to accumulate heavy metals in only average to below average levels, as compared to other

Table 4

Heavy metal concentrations in Stinging nettles (*Urtica dioica* L.) and Common Ragweed (*Ambrosia artemisiifolia*) at the Fostoria Waste Water Treatment Plant. Data are means given in mg/kg dried weight for samplings taken in 1999

Location	Plant Part	Chromium	Copper	Manganese	Molybdenum	Nickel	Lead	Zinc
Stinging Nettle (<i>Urtica dioica</i> L.)								
Control Site	Stem	1.49	19.72	33.99		1.07	1.58	68.3
	Root	5.29	91.18	11.18	1.95	2.55	19.38	80.9
	Leaf	1.17	43.68	51.36		3.37	1.69	65.4
Contaminated Site SN1	Stem	17.06	60.19	37.39		3.45	0.61	50.3
	Root	35.59	431.42	179.31		18.68	15.80	226.3
	Leaf	15.17	138.33	86.45	0.00	12.52	0.47	45.5
Contaminated Site SN2	Stem	21.42	34.03	0.00		2.56	0.98	48.5
	Root	37.79	562.84	96.77		17.94	32.38	458.4
	Leaf	14.39	26.92	49.04		1.07	0.93	130.1
Contaminated Site SN3	Stem	22.52	37.63	22.05	1.22	3.33	4.32	73.2
	Root	24.25	241.60	36.26		9.44	12.57	195.5
	Leaf	21.63	52.59	39.67		5.83	1.45	56.4
Ragweed (<i>Ambrosia artemisiifolia</i>)								
Control Site	Stem	15.02	12.29	29.46		3.59	0.72	98.8
	Root	57.40	12.77			1.75	7.87	59.7
	Leaf	26.19	19.18	21.89	2.20	4.72	1.26	134.7
Contaminated Site RW1	Stem	12.52	93.37	247.67		6.78	4.66	181.3
	Root	35.68	1471.11	103.39		76.95	41.56	274.8
	Leaf	29.19	248.89	548.10		24.32	10.86	468.7
Contaminated Site RW2	Stem	7.00	8.54	46.44		5.83	0.93	348.0
	Root	33.56	1311.60	107.87		51.30	58.63	827.6
	Leaf	10.49	21.87	36.15		19.91	2.15	592.3

plants at our study site. Therefore, soil at the field site was re-sampled during June of the following five summers and samples were grouped with respect to the presence of either stinging nettles or ragweed.

Control sites where no wastes had been applied but with the dominant plant species being either stinging nettle or ragweed were also sampled at this time. Unfortunately, the only sites with these plants outside the lagoon areas were located right along the road where the contaminated waste was transported from the treatment plant to the lagoons. Therefore, the first test was to determine whether the controls were, indeed, unique from the contaminated sites (Tables 4 and 5). Content of all heavy metals in soil with ragweed was significantly greater in contaminated than control sites. However, observed differences

Table 5
Heavy metal concentrations in sediment collected from 2001–2005 at the Fostoria Waste Water Treatment Plant sorted by Plant type

	N	Chromium	Copper	Manganese	Molybdenum	Nickel	Lead	Zinc
(in mg/kg dried sediment)								
Control Sites								
Stinging Nettles								
Shallow								
Average	6	111.12	1517	589	12.65	94.93	887	2061
S.D.		78.01	1033	459	5.19	48.16	808	1092
Deep								
Average	4	47.01	760	224	12.73	66.23	152	1238
S.D.		17.51	442	123	2.20	11.40	52	233
Ragweed								
Shallow								
Average	8	87.45	252	1179	7.99	70.72	191	850
S.D.		52.40	255	541	5.80	14.18	49	1155
Deep								
Average	6	272.60	1305	562	4.42	126.96	269	2793
S.D.		116.11	887	110	3.81	89.16	70	167
Treated Sites								
Stinging Nettles								
Shallow								
Average	18	886.63	19271	1792	15.98	657.42	1320	10081
S.D.		290.08	3508	477	6.61	119.00	308	2009
Deep								
Average	9	898.11	15501	2106	5.74	394.75	1182	5338
S.D.		475.18	10577	395	1.58	178.36	570	3735
Ragweed								
Shallow								
Average	16	1416.92	30614	1435	7.07	676.46	1678	6320
S.D.		454.93	4969	973	2.36	147.09	341	2387
Deep								
Average	10	1190.73	26941	1096	13.59	351.83	1922	6500
S.D.		569.88	17012	405	7.80	82.88	357	1369

Table 6

Statistical analyses of metal concentration data as a function of plant type (stinging nettle and ragweed), depth of soil sample (root zone and deeper), and their interaction term

	P value for plant type	P value for depth	P value for interaction term
Chromium	0.002	0.394	0.345
Copper	<0.001	0.161	0.985
Manganese	<0.001	0.947	0.087
Molybdenum	0.731	0.231	<0.001
Nickel	0.758	<0.001	0.425
Lead	<0.001	0.63	0.086
Zinc	0.065	0.002	<0.001

were not always between contaminated and control sites. For example, molybdenum and manganese show a significant difference between the contaminated sites, but not between the control site and one of the contaminated sites. In contrast, concentrations of chromium and nickel were greater in contaminated than control soil with stinging nettles, but not for molybdenum or lead. There is no clear distinction between contaminated and control stinging nettle sites for copper, zinc, or manganese. Perhaps these control sites became contaminated during transport of sludge from the treatment plant, or leaching of excess metals from the lagoon since their initial deposition on site. We are unable to confirm this hypothesis because the 1999 contractor data was obtained on composited samples that were not discrete with respect to depth.

Heavy metal concentrations in soil within and below the plant maximum root depth were investigated (Tables 5 and 6). The root depths for both plant groups were generally less than the most shallow sampling depth (0.25m) in the original sediment sampling design. Average rooting depth for ragweed at peak season was 15 cm, while nettles grew to about 18 cm depth at the site. The greater concentration of heavy metals in the shallow samples is likely due to absorption of metals by the plants, translocation into their biomass during the growing season, and redeposition at the soil surface upon their death. Metal that is leached below the rooting depth can either be held due to sorption onto the soil or organic particles and/or lost from the profile through leaching (Hartley et al., 2002).

The interaction term between depth and plant type was significant for zinc and molybdenum. In comparison, concentrations of chromium, copper, lead and manganese varied by plant type only (Table 6). Soils with ragweed contained greater concentrations of chromium, copper, and lead than soils associated with nettles (Table 5). Chromium and nickel concentration could not be predicted based on quadrant in the first study; however, plant type helps predict the differences in chromium but not nickel concentrations. Chromium, copper and lead are found in significantly higher concentrations in soils associated with ragweed, even though stinging nettles accumulate a greater concentration of these metals into their leaves (Table 4). Surprisingly, nickel is the only metal that shows only depth as being significant. The ability to uptake these metals into plant biomass and subsequently re-release them into the soil solution could greatly affect the bioavailability of the heavy metals (Edwards et al., 1998).

Soil characteristics, pH and water content are uniform throughout the entire landfill. Initial composition of the waste pits is reported to be identical in all quadrants. Therefore, significant differences in vertical distribution of metal concentrations may be an indication that the plants are able to mobilize some metals, while insignificant differences may indicate that the metals are either “ignored” or indistinguishable by the plant or are affected equally by both plant types. Also, this may indicate that even though the ragweed does not absorb as much heavy metal as the stinging nettles, the soils are not prone to leaching, either. The metals seem to be immobilized in the stinging nettles through plant uptake, and in the ragweed plots through some other process. This aspect of the study will be addressed in further studies.

Summary and Conclusions

The Fostoria Waste Water Treatment Facility is an excellent location to study metal mobility as a function of plant uptake and/or leaching. Sediments were homogenized in 1999 and have been untouched since that time. In the five subsequent years, the heavy metals have been stratified due to these processes, with the majority of metals showing higher concentrations at more shallow depths. Thus, some process is responsible for retaining the metals in the sediment by inhibiting leaching. Although stinging nettles accumulate more metal into biomass as compared to common ragweed (the two dominant plant species), this does not affect the overall soil concentrations. Based on our observations, stinging nettles are efficient in translocating many heavy metals into their aboveground biomass. Nonetheless, the overall heavy metal content of the sediment through time has not been reduced. As the plants die in the fall, the heavy metals are evidently returned to the soil as vegetation decays. In contrast, ragweed is less capable of incorporating the heavy metals into its biomass and is found associated with overall higher heavy metal concentrations in the soil around its roots. Thus, some other process is causing the retention of heavy metals in these soils. The lower concentrations in the soils beneath the root zones, which were reported to have been initially identical in composition to the shallower soils, are likely due to leaching.

Acknowledgements

The authors would like to thank the Ohio Plant Biotechnology Consortium and the United States Department of Agriculture (Grant number 2002-06143) and the NSF REU program for funding this project. We would also like to acknowledge the assistance of Lloyd Jones for plant identification, Donald Stierman for GIS mapping of spatial distribution of plants, technical support by research technicians Brian S. Marlow, Thomas R. Weicht, Greg Taylor, and NSF Research Experience for Undergraduate students (Catherine Buchanan, Natalie Gottschall, Kathryn Lanz). We would like to thank the USDA ARS and Drs. Jonathan Frantz and Charles Krause and Doug Sturtz in Toledo, Ohio, who provided the ICP analyses.

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