Metals Concentrations in Periphyton and Sediments of the Embarras River and Brushy Fork, Douglas County, Illinois

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ABSTRACT

The Embarras River is used as a water supply by several municipalities in east-central Illinois. This study was initiated because industrialization in the area could potentially contribute to the metal burden of Brushy Fork and the Embarras River - streams already subject to agricultural runoff, wastewater plant effluent, and landfill leachate. Our purpose was to evaluate the potential use of attached algae (periphyton) as a biological monitor of heavy metal pollution in streams and to establish baseline concentrations of metals in periphyton of the Embarras River Basin for future comparisons. Periphyton was collected using artificial substrates which were deployed for successive 2-week intervals from 30 May 1990 through 22 September 1990. Sampling sites were located in the Embarras River, upstream and downstream of the mouth of Brushy Fork, and in Brushy Fork, upstream and downstream of the mouth of Newman Drain #2. Analysis of periphyton and stream sediments by inductively coupled plasma-atomic emission spectroscopy revealed the presence of several potentially toxic metals (Al, Ba, Co, Cr, Fe, Mn, Ni, V and Zn). Comparison of these data with previously reported aqueous concentrations of these metals in the Embarras River suggests that monitoring protocols which involve sediment and periphyton may facilitate detection of metals pollution in streams and that the Embarras River drainage may be subject to chronic but relatively low levels metals pollution.

INTRODUCTION

Metals released into terrestrial and aquatic systems from natural and anthropogenic sources may alter the structure of biotic communities as well as pose a threat to public health. Algae and aquatic macrophytes are potentially useful as biological monitors of metal pollution because they concentrate metals from their surroundings such that metals content of their tissues may be measured even when ambient concentrations are so low as to be undetectable by routine analyses (Friant and Koerner, 1981; Bailey and Stokes, 1985; Smith and Kwan, 1989). In addition, biomonitors provide an integrated picture of metals pollution within a given system, particularly if the system is subject only to intermittent or low-level contamination (Whitton, 1985).

Algae which grow attached to various substrata in aquatic systems have been referred to as periphyton, and because of the immediacy of their association with the aqueous medium they may serve as indicators of perturbations in lakes and streams. Metal concentrations in periphyton have been found to be directly related to aqueous concentrations of zinc (Cushing and Rose, 1970), vanadium, chromium, selenium, and nickel (Patrick *et al.*, 1975). Subsequently, Friant and Koerner (1981) found chromium concentrations to be higher in periphyton collected at a site impacted by industrial discharge than at an upstream reference site. Uptake of metals by periphyton also may be a function of sediment metals concentrations (Bailey and Stokes, 1985).

Although ambient concentrations of metals in the waters of the Embarras River drainage were determined in 1987 (Ettinger, 1989), no detailed studies had been undertaken to determine baseline metals content of the periphyton in this drainage. Since current land use in the watershed is primarily agricultural, we believed it necessary to obtain these data before the potential for metals pollution increases. This paper reports on concentrations of metals present in periphyton and sediments of the Embarras River and its tributary, Brushy Fork, and evaluates the potential for use of periphyton as a biological monitor for assessment of water quality in the Embarras River drainage.

MATERIALS AND METHODS

Study Site

The Embarras River drains an area of approximately 4500 km² in east-central Illinois, USA (Fig. 1), receiving largely agricultural runoff along with some municipal and industrial waste effluents (Ettinger, 1987). Sampling sites were established in the Embarras River, 3.7 km upstream (UPEMB) and 4.7 km downstream (DNEMB) of the mouth of Brushy Fork, and in Brushy Fork, 3.0 km upstream (UPBFK) and 1.1 km downstream (DNBFK) of the mouth of Newman Drain #2 (Fig. 1). These sites were selected based upon the siting of a heavy metals reclamation facility in Newman, Illinois and its potential to impact water quality in the Embarras River basin.

Sampling Regime

The periphyton community indigenous to each site was sampled using artificial substrates consisting of 35.5 X 24.4 cm plexiglas sheets. Substrates were exposed just below the stream surface by attachment to a flotation device (71 X 15 X 1.5 cm PVC-pipe frame with a styrofoam block at either end) which was anchored to maintain its position within the stream reach. Substrates were exposed at each site for successive two week periods from 30 May 1990 to 22 September 1990. At the end of each two week period, substrates were collected, air dried, and replaced with clean plexiglas sheets. Sediment samples were collected from each site on 13 June, 8 August, and 22 September and stored frozen. Field measurements of stream flow, conductivity, dissolved oxygen, temperature and pH were made on each sampling date.

Laboratory Analyses

Air-dried periphyton was removed from a 185 cm⁻² area of the upper surface of each substrate. Periphyton samples were placed into separate, preweighed, acid-washed 10 mL glass vials and net dry mass determined after drying at 105 C for 24 hr. Oven-dried periphyton samples were stored in a desiccator and sediment samples were stored frozen prior to elemental analysis.

Periphyton and sediment samples were analyzed by the Illinois Natural History Survey Laboratory in Urbana, Illinois for total cations analysis. Sediment samples were freezedried. Oven-dried periphyton and freeze-dried sediment samples were ground to pass through a 100-mesh nylon fabric sieve before being subjected to similar digestion procedures. Dry samples (sediments = 0.015 g; periphyton = 0.050 g) were placed in 150-mL round bottom flasks with 10 mL of HNO3 and 4.25 mL of 70% HClO4. Flasks were heated until the HNO₃ had volatilized and dense, white $HClO_4$ fumes appeared, at which time another 5 mL of HNO3 was added. Heating with subsequent addition of HNO₃ was repeated until only a small amount of undigested residue (silicon dioxide) remained. This residue was transferred to a PTFE-lined Parr acid digestion bomb and final digestion was accomplished by addition of 3 mL HF with heating to 140 C in a muffle furnace for 2 hr. Individual samples were brought to an appropriate volume with deionized water and aliquots were analyzed for silver, aluminum, arsenic, boron, barium, beryllium, calcium, cadmium, cobalt, chromium, copper, iron, potassium, magnesium, manganese, molybdenum, sodium, nickel, phosphorus, lead, antimony, selenium, silicon, tin, vanadium, and zinc by inductively coupled plasma-atomic emission spectroscopy (ICP-AES) using a Jarrell-Ash 975 Plasma AtomComp spectrophotometer (Sue Wood, personal communication).

Statistical Analyses

Two-factor analysis of variance (ANOVA) without replication (Sokal and Rohlf, 1995) was used to determine significant differences (p < 0.05) among sampling dates and sites. Metals concentrations which did not show significant differences by site were considered replicates, and a single factor (sample date) ANOVA was performed along with the Scheffe method to test for significant differences between individual means by date (Sokal and Rohlf, 1995). Because substrates were not replicated at sampling sites, it was not possible to test for interaction of independent variables. Correlation analysis was used to relate periphyton metals concentrations with sediment metals content, as well as with stream physical and chemical variables (Sokal and Rohlf, 1995).

RESULTS

Artificial substrates were recovered from all four sampling sites every two weeks from 13 June through 22 September 1990, with the exception of 27 June. Substrates deployed on 13 June were lost, apparently due to flooding, at all but the UPEMB site. Previously, it was determined that material removed from these substrates consisted primarily of diatoms, but also contained other algae, bacteria, protozoans, aquatic insect larvae, and trapped sediments (Vaultonburg and Pederson, 1994). However, material removed from the artificial substrates will be referred to consistently as periphyton for the sake of clarity.

Seven metals which are either essential plant nutrients or which are generally not toxic that were detectible in sediments and periphyton included boron, calcium, potassium, magnesium, sodium, phosphorus, and silicon. Because these nutrient elements were not the focus of our study, they have been omitted from statistical analyses and are not considered further in this paper. Concentrations of ten metals in sediments ([S]) and periphyton ([P]), including some of the more toxic metals, were at or below the detection limits of ICP-AES at all four sampling sites on all dates. These were silver ([S] < 33.0 ppm; [P] < 10.0 ppm), arsenic ([S] < 100 ppm; [P] < 30.0 ppm), beryllium ([S] < 3.33 ppm; [P] < 1.00 ppm), cadmium ([S] < 13.3 ppm; [P] < 4.00 ppm), copper ([S] < 13.3 ppm; [P] < 4.00 ppm), nolybdenum ([S] < 20.0 ppm; [P] < 6.00 ppm), lead ([S] < 50.0 ppm; [P] < 15.0 ppm), antimony ([S] < 43.3 ppm; [P] < 13.00 ppm), selenium ([S] < 103 ppm; [P] < 31.0 ppm), and tin ([S] < 110 ppm; [P] < 33.00 ppm).

Aluminum (Al), barium (Ba), chromium (Cr), iron (Fe), manganese (Mn), nickel (Ni), and zinc (Zn) are potentially toxic and were present in measurable concentrations in periphyton (Table 1) as well as sediments (Table 2). Cobalt (Co) and vanadium (V) were detected consistently in periphyton (Table 1) but not in sediments (Table 2). Periphyton concentrations of these nine metals were subjected to two-factor ANOVA (sample date, sample site). Nickel concentrations did not vary significantly by date or site over the course of the study. Therefore, the mean concentrations detected significant differences between site and date. However, if data are grouped such that UPEMB and DNEMB are considered replicates as are UPBFK and DNBFK, then the mean Cr concentration in periphyton of the Embarras River (51.7 ppm, n=14) is greater than in periphyton of Brushy Fork (41.7 ppm, n=14).

Concentrations of Al, Ba, Co, Fe, Mn, V, and Zn in periphyton did not differ by site but did differ by date. On this basis, sampling sites were considered to be replicates and data were subjected to one-factor ANOVA (sample date) which identified significant variation between sampling dates for all of these metals. Concentrations increased temporally, with periphyton collected in September bearing a significantly higher metals concentration than periphyton collected earlier in the sampling season (Table 3). Periphyton concentrations of Al, Ba, Cr and Ni were not correlated with any of the physical and chemical variables, while significant negative correlations were observed between stream flow and concentrations Co, Fe, Mn, V, and Zn.

Sediment concentrations of Al, Ba, Cr, Fe, Mn, and Zn did not differ by site or date. Nickel concentrations in sediment differed significantly by site but not date, although Scheffe's test identified differences only between UPBFK and DNEMB. While V was present in measurable amounts in some sediment samples collected from both Embarras River sites as well as DNBFK, Co was not detected in any sediment samples (Table 2). Of the nine potentially toxic metals identified in periphyton, only Ni was always present in greater concentrations in sediment (Table 4). Barium and Zn were detected at slightly higher maximum concentrations in sediment than in periphyton, however values did not vary by orders of magnitude. Otherwise, metals were present in equal or greater concentrations in periphyton when compared to concentrations in sediments. No correlation was detected between periphyton and sediment concentrations of these nine metals.

DISCUSSION

Detection of metals pollution in streams may be facilitated by using periphyton as a biological monitor, rather than by attempting to directly assess metals concentrations of water or sediments (Friant and Koerner, 1981). Concentrations of heavy metals in water may be below the analytical limit of detection as exemplified in the data obtained during an intensive study of the Embarras River basin (Ettinger, 1989). Potentially toxic metals such as Co, Cr, Ni, V, and Zn often were not detected or were found only in very low aqueous concentration, leading to the conclusion that water quality in the Embarras River was very good or even excellent with regard to metals content. However, even if aqueous concentrations are high enough to permit detection, one time "grab" samples may not intercept pulses of pollution which can be considerably greater than background levels.

Our work suggests that metals can be detected more reliably through monitoring of sediments and periphyton as compared to monitoring of aqueous concentrations alone. Of the toxic metals not detected by water analysis in 1987 (Ettinger, 1989), only Co remained undetected in sediments during 1990 (Table 4). Furthermore, Bailey and Stokes (1985) found sediment metals concentrations of 1,000-10,000 times concentrations found in water. If aqueous metals concentrations in the Embarras River are presumed to have changed little since 1987, then our data suggest sediment concentration factors ranging from nearly 500 times the probable aqueous concentration of Mn in water to more than 8000 times that for Al (Table 4).

Since periphyton metals concentrations provide an integrated picture of metals pollution over time while demonstrating bioavailability, our data indicate that the Embarras River drainage may be subject to chronic but relatively low level metals pollution. This is supported by the fact that we found concentrations of some potentially toxic metals in sediments of the Embarras River and Brushy Fork to be greater than those found in other regions of the United States. Concentrations of Al in sediments of the Embarras drainage exceed those found in sediments of Clark Fork River in Montana which is heavily contaminated with Cu mining and smelting wastes (Moore et al., 1989). Barium, Cr, Fe, Mn, and Ni are more concentrated in Embarras River and Brushy Fork sediments than in those of the South Dry Sac and Little Sac Rivers in southwest Missouri which are subject to leachate from landfills and wastewater treatment plant effluent (Mantei and Foster, 1991), and Cr concentrations were higher than the average reported for sediments in 63 Illinois lakes (Kelly and Hite, 1981). In addition, concentrations of Al, Cr, Fe, Mn, Ni and Zn in Embarras River and Brushy Fork sediments tended to be higher than the average concentrations in 1318 soil samples collected throughout the United States (Shacklette and Boerngen, 1984).

Although pH has been found to be inversely correlated with metals concentrations in water and algal material (Bailey and Stokes, 1985), pH tended to be higher on September sampling dates during our study. Thus, our observation of a temporal increase in periphyton metals concentration is somewhat difficult to explain. Possible explanations include seasonal changes in algal community structure (Vaultonburg and Pederson, 1994) since concentrations of metals in algae vary between genera and within a genus (Trollope and Evans, 1976). Alternatively, qualitative changes in sediment accumulated along with

periphyton on artificial substrates may have resulted in perception of increased metals concentrations. Metal concentrations are affected by the size and composition of sediment particles (Combest, 1991) as well as the type of other metal sequestering materials (e.g., organics) which are attached to those particles (Mantei and Foster, 1991). Work by Bailey and Stokes (1985) demonstrated sediment metals to be correlated with organic content. Combest (1991) found a near linear relationship between the percentage of clay present in sediment and the quantity of Cu, Cr, and Zn detected. He also observed an inverse relationship between the quantity of metal present and the percent of sand. However, it is worth noting that metal concentration does not always increase in proportion to decreasing particle size and, on occasion, higher metal concentrations are found in association with larger particles (Gibbs, 1977; Feltz, 1980). Seasonal changes in community structure and quality of sediment associated with the periphyton may ultimately have been determined by flow regime.

We believe that use of artificial substrates to collect periphyton for monitoring metals pollution in streams provides a viable alternative to monitoring protocols which include analysis of only water or sediments. In our study, not only were more metals detected in periphyton, but concentrations were generally much higher than aqueous concentrations reported by Ettinger (1989). Furthermore, concentrations in periphyton were at least equal to those of sediments. Although we detected a greater number of metals in sediments than were detected in water by Ettinger (1989), Friant and Koerner (1981) point out that physical and chemical processes are required to sequester metals into sediments. In contrast, algae (periphyton) have been demonstrated to concentrate metals by active as well as passive uptake. Therefore, periphyton provides a more realistic representation of spatial and temporal variation in ambient metals concentrations while demonstrating which metals are available for uptake by the aquatic and riparian plants that form the basis of freshwater food webs.

The metals burden of Brushy Fork and the Embarras River already may be substantial with sources including agricultural runoff, municipal and industrial effluent, and landfill leachate. Our data provide a valuable baseline for assessing future impacts on the Embarras River system while demonstrating a method with potential for broad application. However, further research is required regarding the influence of various physical and chemical factors on metals uptake by periphyton.

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<u>a.</u>	Elements								
Site ExposureDates	Al	Ba	Со	Cr	Fe	Mn	Ni	V	Zn
UPEMB									
5/30-6/13	40000	472	14.3	51.6	14200	406	27.5	54.6	51.2
6/27-7/11	34900	419	19.0	53.8	16600	826	72.8	95.0	77.0
7/11-7/25	57800	418	16.2	50.0	14400	546	32.2	38.8	45.0
7/25-8/08	46200	493	24.8	41.6	18200	808	33.8	40.2	70.2
*8/08-8/25	39700	476	20.6	51.2	15800	984	45.6	68.0	59.0
8/25-9/08	41000	430	36.2	66.6	29300	1670	47.2	108	108
9/08-9/22	56800	530	31.4	62.0	29400	1790	45.8	104	114
DNEMB									
5/30-6/13	33500	402	11.4	58.6	12800	391	27.9	54.5	49.7
6/27-7/11	29300	272	11.0	38.2	11000	405	24.8	29.5	48.2
7/11-7/25	38500	422	15.2	42.6	14100	799	37.2	60.1	61.2
7/25-8/08	39000	476	13.8	48.3	18000	889	38.3	63.6	73.0
*8/08-8/25	38400	460	22.6	54.8	16700	1840	48.2	34.8	68.6
8/25-9/08	49600	502	35.2	48.6	25400	1380	41.2	83.4	98.0
9/08-9/22	48800	529	41.0	55.7	25800	1760	42.0	94.0	101
UPBFK									
5/30-6/13	33600	334	16.2	41.3	16800	596	33.7	53.5	87.6
6/27-7/11	32900	345	18.1	38.8	16100	642	31.6	68.2	64.2
7/11-7/25	44400	473	19.0	44.6	17900	684	41.2	63.0	76.0
7/25-8/08	38400	424	21.4	33.6	17400	833	37.0	82.2	76.0
*8/08-8/25	44900	501	32.7	42.4	23400	1270	39.8	86.6	97.6
8/25-9/08	35800	449	33.3	36.4	18600	2010	35.0	74.5	82.1
9/08-9/22	50800	533	35.2	42.9	25800	4260	49.4	95.4	95.4
DNBFK									
5/30-6/13	35000	386	14.8	43.1	15200	628	34.7	42.5	73.8
6/27-7/11	28800	463	15.1	31.4	10400	554	55.4	53.4	64.8
7/11-7/25	44600	460	15.2	46.9	19100	956	29.0	66.0	73.4
7/25-8/08	43300	458	28.1	33.4	19000	1380	33.8	61.5	80.3
*8/08-8/25	41200	476	24.4	41.4	17200	1220	29.9	56.6	63.0
8/25-9/08	49600	479	38.1	54.2	24400	1100	38.4	87.4	97.5
9/08-9/22	44800	477	48.1	52.8	27800	2690	40.6	104	125

Table 1. Metals concentrations (ppm) in periphyton removed from artificial substrates exposed for successive two-week periods in the Embarras River and Brushy Fork during 1990.

* Substrates deployed on 8/08/90 were recovered after a 17-day incubation period rather than the standard two week period due to logistical problems.

		Elements							
Site ExposureDates	Al	Ba	Со	Cr	Fe	Mn	Ni	V	Zn
UPEMB									
6/13	19800	395	<6.7	62.2	11400	760	57.5	26.0	50.2
8/08	22100	400	<6.7	57.2	15600	424	84.6	46.8	142
9/22	17200	290	<6.7	32.8	8460	377	65.3	<23.3	38.1
DNEMB									
6/13	14700	318	<6.7	44.0	17000	1090	52.6	59.0	82.6
8/08	15400	240	<6.7	48.0	4530	220	51.0	44.0	25.5
9/22	18100	320	<6.7	40.4	10100	488	40.6	64.8	49.2
UPBFK									
6/13	21300	390	<6.7	39.4	8980	446	67.2	<23.3	32.8
8/08	15300	353	<6.7	49.0	18700	2170	84.6	<23.3	114
9/22	18600	383	<6.7	37.0	17000	1010	75.8	<23.3	51.8
DNBFK									
6/13	24900	590	<6.7	43.4	22000	1140	58.0	34.0	53.4
8/08	19900	360	<6.7	39.2	11600	456	55.6	24.4	51.0
9/22	16900	352	<6.7	29.8	13200	467	53.4	<23.3	67.4

Table 2.Metals concentrations (ppm) in sediments of the Embarras River and Brushy
Fork during 1990.

< Indicates metal concentrations below detection limits of ICP-AES.

F		Elements								
(Period) Date	Al	Ba Co		Fe N	In	V	Zn			
(1) 5/30-6/13	35500 (7)	399	14.2 (6,7)	14800 (6,7)	505 (7)	51.3 (7)	65.6 (7)			
(2) 6/27-7/11	31500 (3,7)	375 (7)	15.8 (6,7)	13500 (6,7)	607 (7)	61.5	63.6 (7)			
(3) 7/11-7/25	46300 (2)	443	16.4 (6,7)	16400 (6,7)	746 (7)	57.0	63.9 (7)			
(4) 7/25-8/08	41700	463	22.0 (6,7)	18200 (7)	978 (7)	61.9	74.9			
(5) 8/08-8/25	41100	478	25.1 (7)	18300 (7)	1330	61.5	72.1			
(6) 8/25-9/08	44000	465	35.7 (1,2,3,4)	24400 (1,2,3)	1540	88.3	96.4			
(7) 9/08-9/22	50300 (1,2)	517 (2)	38.9 (1,2,3,4,5)	27200 (1,2,3,4)	2630 (1,2,3,4)	99.4 (1,2,3)	109.0 (1,2,3)			

Table 3. Mean concentrations (ppm) of potentially toxic metals in periphyton collected from 4 sites in the Embarras River and Brushy Fork during 1990. Mean concentrations for exposure periods which differ significantly (p < 0.05) from a given value are shown in parentheses.

Table 4. Minimum and maximum metals concentrations (ppm) observed in sediments and periphyton of the Embarras River and Brushy Fork during 1990. Minimum and maximum metals concentrations observed in 1987 in water samples collected from Brushy Fork and the Embarras River near the mouth of Brushy Fork are from Ettinger (1989).

Element	Aqu Concer (19	eous ntration 87)	Sedi Conce (19	iment ntration 990)	Periphyton Concentration (1990)		
Element	Minimum	wiaximum	WIIIIIIIIII	Maximum	wiininum	Maximum	
Al	0.46	2.91	14700	24900	28800	57800	
Ba	0.041	0.074	240	590	272	533	
Co			<6.7	<6.7	11.4	48.1	
Cr	0.005	0.006	29.8	62.2	31.4	66.6	
Fe	0.57	3.47	4530	22000	10400	29400	
Mn	0.082	0.464	220	2170	391	4260	
Ni	< 0.005	0.020	40.5	84.6	24.8	72.8	
V	< 0.005	0.007	<23.3	64.8	29.5	108	
Zn	< 0.005	< 0.005	25.5	142	45.0	125	

--- Indicates metals for which concentrations were not determined

< Indicates metal concentrations which were below detection limits with regard to the material tested

Figure 1. Embarras River and Brushy Fork drainage in Champaign, Edgar and Douglas Counties showing the locations of sampling sites in the Embarras River, upstream (UPEMB) and downstream (DNEMB) of the mouth of Brushy Fork, and in Brushy Fork, upstream (UPBFK) and downstream (DNBFK) of the mouth of Newman Drain #2.

