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CONTAMINANT ASSESSMENT OF FISH, RANGIA CLAMS, AND SEDIMENTS IN IN THE LOWER PAMLICO RIVER, NORTH CAROLINA

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Contaminant Assessment of Fish, Rangia Clams and Sediments in the Lower Pamlico River, North Carolina

Abstract: Samples of sediment, rangia clams (Rangia cuneata), gizzard shad (Dorosoma cepedianum), and longnose gar (Lepisosteus osseus) were collected from five sites in the lower Pamlico River and analyzed for elemental contaminants, organochlorines, aliphatic hydrocarbons, and polynuclear aromatic hydrocarbons. Most sample concentrations were either beneath the detection limit or too low to be associated with biological impacts. However, sediment sample concentrations of cadmium and fluoride were observed at levels that could be associated with biological impacts at one of the sites which was located near the discharge from a large phosphate mining operation.

Key Words: Pamlico River, Fluoride, Cadmium, Sediment, Rangia Clam, Fish

Preface

This draft report addresses work funded and performed under environmental contaminants study identifier 92-4F07 and USFWS contaminants catalog number 4100003.

Questions, comments, and suggestions related to this report are encouraged. Inquires should be directed to the Service at the following address:

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INTRODUCTION

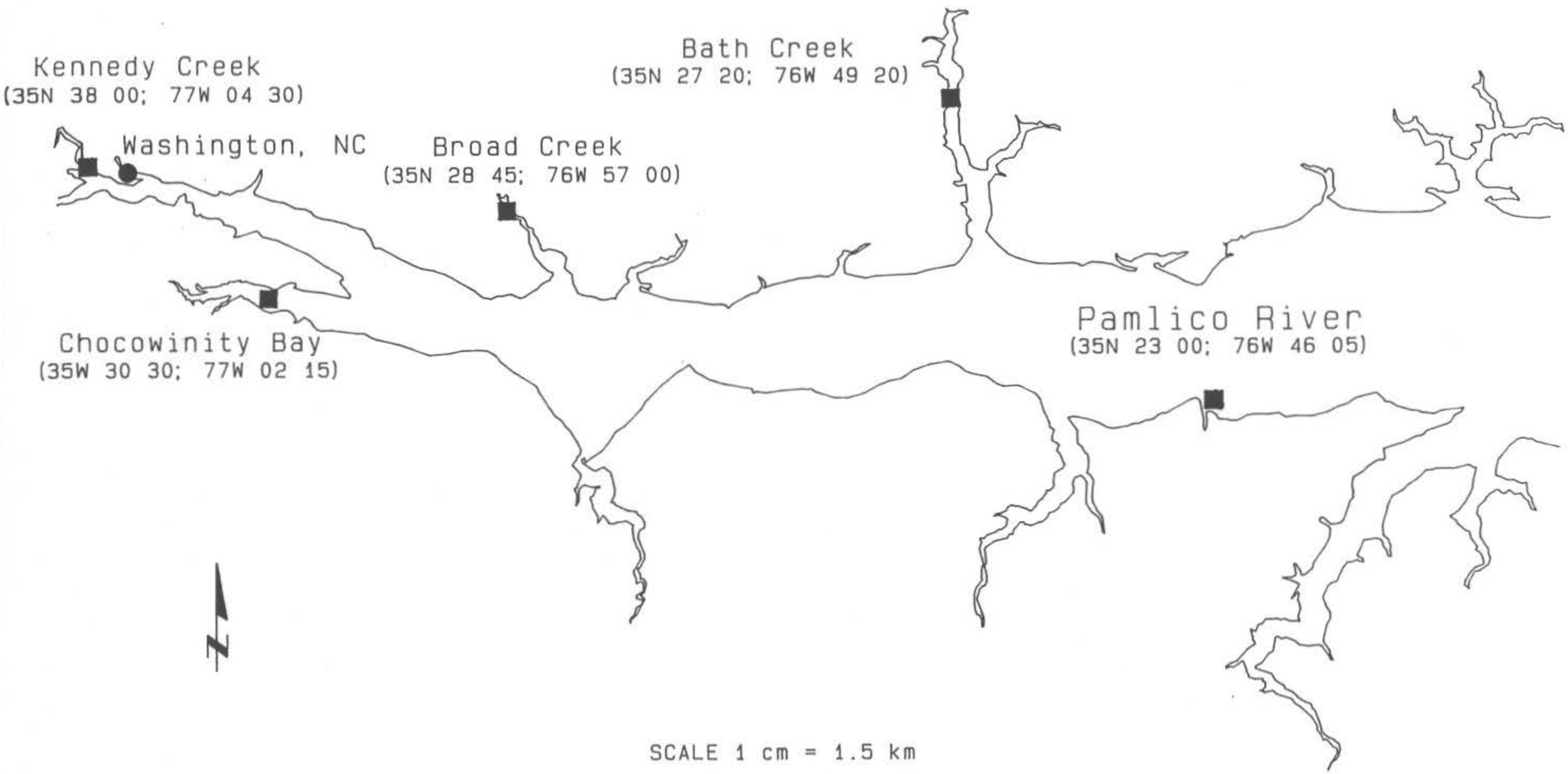
Work recently conducted has documented heavy metal pollution of sediments in areas of the Pamlico River (Riggs et al. 1989). Concern exists over the transfer of heavy metals into fish and shellfish although such mechanisms are poorly understood. The lower Pamlico River receives discharge from one of the largest phosphate mining operations in North America and has been the location of several fish kills and disease outbreaks recently (Noga et al. 1989). Contaminant data for biota in the Albemarle-Pamlico drainage are limited (NC Division of Environmental Management 1991; Benkert 1992). The objective of this study was to expand the existing contaminants baseline data in the Albemarle-Pamlico area.

METHODS

Sediment samples were collected at a depth of about three feet from October 21-23, 1991 at five sites in the lower Pamlico River. The Chocownity Bay sediment sample was collected off the northeast bank in the first embayment northwest of Fork Point. The Kennedy Creek sample was taken approximately 400 meters southwest of the water tower off the opposite bank. The Broad Creek sample was taken on the west bank approximately 3 km upstream of Mcgotter's Marina. Bath Creek was sampled approximately 15 meters offshore from the wooden bulkhead at Archbell Point, across from a group of large pine trees in the hunting preserve field. The Pamlico River sediment was taken approximately 75 meters offshore from the effluent discharge creek just east of the Texasgulf plant. Latitude and longitude coordinates were recorded for each site (Figure 1) so that sites could be accurately revisited using loran navigational equipment. Longnose gar (Lepisosteus osseus) and gizzard shad (Dorosoma cepedianum) were collected with gill nets. Rangia clams (Rangia cuneata) were collected with a clam rake or small dredge. Salinity and temperature were measured mid-water at each site. After collection fish and clams were wrapped in aluminum foil and placed in wet ice. Five core sediment samples were collected at each site with a 5 cm inside diameter PVC core sampler inserted to an approximate depth of 7.5 cm. Core samples were emptied into a stainless steel container and homogenized with a stainless steel spoon. The homogenate was split into two parts; a portion for inorganic analysis was placed in a plastic bag and a portion for organics was placed in a chemically-cleaned glass jar. All sediments were stored on wet ice in the field.

Upon returning from the field, clam soft tissues were removed, placed in a stainless steel container and homogenized with a

Pamlico River Sample Sites



stainless steel spoon. The clam sample homogenate was then split into two chemically cleaned glass jars and frozen. Clam composites consisted of 17 to 50 individuals per site. The fish were measured to the nearest mm total length and weighed to the nearest gram. Samples were stored in a freezer prior to shipment on dry ice for laboratory analysis.

Elemental contaminant analyses were conducted at the Environmental Trace Substance Research Center, Columbia, Missouri and organic analyses were conducted at the Mississippi State Chemical Laboratory, Mississippi State, Mississippi. Organochlorine scans used electron capture capillary gas chromatography, and PCB results were confirmed by mass spectrometry. Lower levels of detection were 0.01 ppm except for toxaphene and PCB's which were 0.05 ppm. Aliphatic hydrocarbons were quantified by capillary column flame ionization gas chromatography and aromatic hydrocarbon quantification utilized capillary flame ionization gas chromatography and fluorescence high pressure liquid chromatography.

Metals were extracted by strong acid digestion with a mixture of nitric and perchloric acids. This digestion yields very low results (< 50% recovery) on standard reference materials for Al, Ba, Sr, and V; low results (> 50% & < 80% recovery) for As, Cr, Fe, and Mg; and results close to the certified value (> 80% recovery) for Se, Hg, Cd, Cu, Mn, Ni, Pb, and Zn. Standard reference material was not available for B, Be or Mo, but based on spike recovery data they are probably in the low result range. Strong acid digestion gives a measure of the maximum potential bioavailablity of the metal in the sample. An inductively coupled plasma emission spectroscopy scan was used for metals. Mercury content was determined by cold vapor reduction atomic aborption spectroscopy and arsenic by graphite furnace. Fluoride samples were mixed with ion selective electrode buffer and mineral oil as a combustion aid, combused in a Parr Oxygen Bomb and analyzed with an ion selective electrode. Lower levels of detection were variable for different metal samples. Quality assurance/quality control (QA/QC) samples, including blanks, spiked samples, reference material analyses, and duplicate analyses, were performed for all analytes. Review of QA/QC samples indicated precision and accuracy were acceptable for all analytes.

RESULTS and DISCUSSION

Site salinities ranged from 0.8 ppt at Kennedy Creek to 5.8 ppt at Bath Creek. The sediment samples from Broad Creek, Bath Creek, and Pamlico River near Texasgulf had a very low percent composition of clay and total organic carbon (Table 1) which is where any contaminants are going to be. The sand and silt fractions which were the major constituents of sediment sampled from Broad Creek, Bath Creek and Pamlico River at Texasgulf are mostly inert quartz. In general elemental contaminants, organochlorines, aliphatic hydrocarbon, and polynuclear aromatic hydrocarbon sample concentrations of sediment, rangia clams, gizzard shad, and longnose gar (Tables 2-7) were either beneath the detection limit or too low to be associated with biological impacts (Long and Morgan 1990). However, sediment concentrations of cadmium and fluoride were elevated for the Texasgulf site when compared to the other sites sampled, and were observed at levels that could be associated with biological impacts (Long and Morgan 1990). Values observed for cadmium and fluoride are discussed separately in the text following the tables.

Table 1. Site water salinity, temperature and sediment composition.

Sample Site	Salinity (ppt)	Temp. (°C)	%Sand	%Silt	%Clay	%TOC
Kennedy Creek	0.8	19.9	42	44	15	16
Chocowinity Bay	2.8	22.5	30	45	25	15
Broad Creek	4.1	19.0	95	3	2	0.4
Bath Creek	5.8	18.3	88	6	6	0.2
Pamlico River (at Texasgulf)	3.8	18.0	59	34	8	0.1

Table 2. Trace element concentrations in sediment composites (ppm dry weight).

Element	Kennedy Creek	Chocow. Bay	Broad Creek	Bath Creek	Pamlico River (at Texasgulf)
Al	27800	43600	4820	7290	13300
As	2.2	3.6	0.42	<0.1	4.3
B	7.8	<20	<2	3	17
Ba	95.5	89	56.1	15.4	50.4
Be	1.4	1	0.1	0.2	0.71
Cd	<0.4	<3	<0.4	<0.3	8.8
Cr	19	<30	4	8	36
Cu	9.3	14	2.9	1.9	8.9
F	109	54.1	<8.8	11.6	475
Fe	17300	23600	1970	3760	11000
Hg	0.063	0.085	0.01	0.018	0.027
Mg	3300	5070	333	609	6430
Mn	166	160	63.8	40	204
Mo	3	<10	<1	<1	2
Ni	13	<20	2	2	8.6
Pb	21	<40	6	7	14
Se	0.4	1.1	<0.2	<0.2	0.3
Sr	62.4	78	17	6.9	311
V	40.8	62	5.4	13	37.9
Zn	34.3	23	7.4	10	98.5

Table 3. Trace element concentrations in rangia clam composites (ppm wet weight).

Element	Kennedy Creek	Chocow. Bay	Broad Creek	Bath Creek	Pamlico River (at Texasgulf)
Al	62.3	27	41	47	52.7
As	0.91	0.69	1.2	0.78	0.55
B	<0.3	<0.2	0.3	0.8	0.6
Ba	4.73	2.93	1.4	0.57	0.74
Be	0.006	0.005	<0.007	0.0060	0.007
Cd	0.026	0.01	0.062	0.07	0.17
Cr	0.1	0.09	0.62	<0.1	0.1
Cu	2.71	1.1	5.77	1.8	1.8
F	<1.00	<1.26	<1.33	1.93	13.6
Fe	82.4	70.3	92.9	61	68.7
Hg	0.036	0.014	0.036	0.0160	0.007
Mg	205	249	268	312	321
Mn	3.16	12.1	27.5	19.8	14.3
Mo	<0.1	<0.1	<0.2	<0.2	<0.1
Ni	0.86	1.1	0.56	0.68	2.1
Pb	<0.1	<0.1	<0.2	<0.2	<0.1
Se	<0.23	0.17	0.31	0.4	0.34
Sr	2.71	4.65	5.18	4.7	5.85
V	0.09	0.07	0.1	0.16	0.17
Zn	16.2	8.82	13.5	12.7	11.4

Table 4. Trace element concentrations in fish composites (ppm wet weight).

Element	Kennedy	Chocow.	Broad	Bath	Texasgulf
Al (shad)	57	6.7	5.4	318	32
(gar)	14	-----	----	----	3.5
As (shad)	0.24	0.26	0.19	0.19	0.21
(gar)	0.35	----	----	----	0.82
B (shad)	<0.5	<0.5	<0.5	<0.6	<0.6
(gar)	<0.6	----	----	----	<0.7
Ba (shad)	1.2	0.8	0.54	1.5	0.38
(gar)	4.05	----	----	----	0.85
Be (shad)	<0.01	<0.01	0.01	0.02	<0.009
(gar)	<0.01	----	----	----	<0.01
Cd (shad)	<0.02	<0.02	<0.02	<0.02	0.03
(gar)	<0.02	----	----	----	<0.02
Cr (shad)	<0.2	<0.2	0.5	2	1.3
(gar)	3.2	----	----	----	5
Cu (shad)	0.46	0.83	1.5	1.1	0.82
(gar)	0.71	----	----	----	1.2
F (shad)	2.87	2.39	<1.59	<2.90	3.56
(gar)	6.36	----	----	----	8.44
Fe (shad)	69.8	79.7	44.7	306	70.3
(gar)	50.5	----	----	----	53
Hg (shad)	0.021	0.015	0.014	0.013	0.01
(gar)	0.15	----	----	----	0.82
Mg (shad)	270	291	286	398	327
(gar)	2960	----	----	----	2840
Mn (shad)	4.9	4.3	3.3	16.6	3.2
(gar)	7.52	----	----	----	5.7
Mo (shad)	<0.3	<0.3	<0.3	<0.3	<0.3
(gar)	<0.3	----	----	----	<0.4
Ni (shad)	0.1	0.2	0.36	2	0.61
(gar)	1.6	----	----	----	2.3
Pb (shad)	<0.3	<0.3	0.5	0.88	<0.3
(gar)	<0.3	----	----	----	<0.4
Se (shad)	0.28	0.29	0.33	0.1	0.32
(gar)	0.2	----	----	----	0.2
Sr (shad)	17.6	22.2	21.4	37.1	19.9
(gar)	56.9	----	----	----	78.6
V (shad)	0.2	<0.08	<0.08	3.5	0.1
(gar)	<0.1	----	----	----	<0.1
Zn (shad)	9.14	9.39	10.2	13.4	9.83
(gar)	23.2	----	----	----	20.7

--- = less than method detection limit

Table 5. Organochlorine concentrations in gizzard shad composites (ppm wet weight).

Analyte	Kennedy	Chocow.	Broad	Bath	Texasgulf
HCB	ND[a]	ND	ND	ND	ND
α-BHC	ND	ND	ND	ND	ND
Γ-BHC	ND	0.01	ND	ND	ND
β-BHC	ND	0.01	ND	0.01	ND
δ -BHC	ND	ND	ND	ND	ND
Oxyclordane	ND	ND	ND	ND	ND
Hept. Epox.	ND	ND	0.01	ND	0.01
Γ-Chlordane	ND	0.01	ND	ND	0.01
τ-Nonachlor	0.01	0.02	0.01	0.01	0.01
Toxaphene	ND	0.45	ND	ND	ND
PCB's(total)	ND	ND	ND	ND	ND
o,p'-DDE	ND	ND	0.01	ND	ND
p,p'-DDE	0.09	0.16	0.08	0.09	0.18
Dieldrin	0.02	0.02	0.02	ND	0.02
o,p'-DDD	0.01	0.02	0.01	ND	0.01
Endrin	ND	ND	ND	ND	ND
cis-nonach.	0.01	0.02	0.01	ND	0.02
o,p'-DDT	ND	0.01	ND	ND	ND
p,p'-DDD	0.05	0.08	0.04	0.03	0.09
p,p'-DDT	0.01	0.02	0.01	0.01	0.01
Mirex	ND	0.01	ND	ND	ND

[a] ND = below method detection limit.

Table 6. Aliphatic hydrocarbon concentrations in sediment and rangia clam composites (ppm wet weight).

Analyte	Kennedy	Chocow.	Broad	Bath	Texasgulf
n-dodecane					
sediment	0.01	ND[a]	ND	ND	0.01
clams	0.01	ND	0.01	ND	ND
n-tridecane					
sediment	ND	ND	ND	ND	0.01
clam	ND	ND	ND	ND	ND
n-tetradecane					
sediment	ND	0.01	0.32	ND	0.01
clam	0.01	ND	ND	ND	0.01
octycyclohexane					
sediment	0.01	ND	0.02	ND	0.01
clam	0.02	ND	ND	ND	0.01
n-pentadecane					
sediment	0.04	0.01	0.03	0.02	0.02
clam	0.02	0.02	0.06	0.16	0.10
nonylcyclohexane					
sediment	0.01	ND	ND	0.01	0.02
clam	0.01	ND	ND	ND	0.02
n-hexadecane					
sediment	0.03	ND	0.01	0.01	0.01
clam	0.02	0.02	0.02	0.02	0.02
n-heptadecane					
sediment	0.30	0.05	0.16	0.13	0.04
clam	0.04	0.07	0.13	0.19	0.09
pristane					
sediment	ND	0.01	ND	ND	0.03
clam	0.01	ND	ND	ND	ND
n-octadecane					
sediment	0.02	0.01	0.01	0.01	0.02
clam	0.01	0.01	0.01	ND	0.02
phytane					
sediment	0.01	0.01	ND	0.01	0.04
clam	0.01	ND	ND	ND	0.01
n-nonadecane					
sediment	0.03	0.03	0.02	0.03	0.03
clam	0.01	0.01	0.01	0.01	0.03
n-eicosane					
sediment	0.02	0.01	0.01	0.03	0.02
clam	0.02	ND	ND	ND	0.01

[a] ND = below method detection limit.

Table 7. Polynuclear aromatic hydrocarbon concentrations in sediment and rangia clam composites (ppm wet weight).

Analyte	Kennedy	Chocow.	Broad	Bath	Texasgulf
napthalene					
sediment	ND[a]	ND	ND	ND	ND
clam	0.02	ND	ND	ND	ND
fluorene					
sediment	ND	ND	ND	ND	ND
clam	ND	ND	ND	ND	ND
phenanthrene					
sediment	0.01	ND	ND	ND	ND
clam	0.06	ND	ND	ND	0.02
anthracene					
sediment	ND	ND	ND	ND	ND
clam	ND	ND	ND	ND	ND
flouranthene					
sediment	ND	ND	ND	ND	0.05
clam	ND	ND	ND	ND	ND
pyrene					
sediment	ND	ND	ND	ND	ND
clam	ND	ND	ND	ND	ND
1,2-benzanthracene					
sediment	ND	ND	ND	ND	ND
clam	ND	ND	ND	ND	ND
chrysene					
sediment	ND	ND	ND	ND	ND
clam	ND	ND	ND	ND	ND
benzo(b)fluoranthene					
sediment	0.01	ND	ND	ND	0.01
clam	ND	ND	ND	ND	ND
benzo(k)flouranthene					
sediment	ND	ND	ND	ND	0.01
clam	ND	ND	ND	ND	ND
benzo(e)pyrene					
sediment	ND	ND	ND	ND	ND
clam	ND	ND	ND	ND	ND
1,2,5,6-dibenzanthracene					
sediment	ND	ND	ND	ND	ND
clam	ND	ND	ND	ND	ND
benzo(g,h,i)perylene					
sediment	ND	ND	ND	ND	ND
clam	ND	ND	ND	ND	ND

[a] ND = below method detection limit.

Cadmium

The composite concentration of cadmium in sediment observed at the Texasgulf site was 8.8 ppm dry weight. This value is above the concentration (5 ppm) at the low end of the range in which biological effects have been observed and slightly less than the concentration (9 ppm) approximately midway in the range of reported values associated with biological effects (Long and Morgan 1990). All other site concentrations were beneath detection levels (Table 2). The potential for biological effect of cadmium at Texasgulf may be underestimated by the observed concentration because the sediment sampled at Texasgulf had a low percent total organic carbon (0.1) and thereby a low pollutant binding affinity. The highest observed cadmium concentrations for rangia clam tissue and gizzard shad tissue were also observed at the Texasgulf site although the difference between site values for gizzard shad is very small. Fertilizer production is one of several anthropogenic sources of cadmium which include smelter fumes and dust, incineration of cadmium-bearing materials and fossil fuels, and municipal wastewater and sludge discharges (Eisler 1985).

Fluoride

Sediment

Sediment fluoride concentration at the Texasgulf site appeared to be elevated when compared to other Pamlico River sample locations (Table 2), although this observation could not be tested statistically because the composite sample methodology utilized did not allow for an estimate of sample variance. Lee and Marianna (1977) observed that measures of sediment chemical concentrations were not good indicators of sediment toxicity as indicated by tests using shrimp (*Palaemonetes pugio*). DiToro (1989) believed that the chemical concentration in pore water was a better indicator of toxicity, as it was more associated with bioavailability. Texasgulf discharged about 1 million pounds of fluoride each year in 1989-1990 (Cunningham et al. 1992) but is moving towards a recycling program which is expected to reduce fluoride discharges by 75 percent.

Tissue

We sampled rangia clam, gizzard shad and longnose gar tissues to determine if fluoride present in the sediments was also evident in biota, and to complement the area database for tissue concentrations of other contaminants (Riggs et al. 1989, North Carolina Division of Environmental Management 1991, Benkert 1992, Gemperline et al. 1992, Weinstein et al. 1992). These samples constitute the first assessment of fluorides in tissue for this area and subsequently should provide a reference for future comparisons. Rangia clams collected from the Pamlico River at Texasgulf had a fluoride concentration approximately seven times as high as that observed at Bath Creek which was the only other site at which fluoride occurred above the lower limit of quantification. The rangia clam composite samples utilized from 17 to 50 clams per sample so that the observed concentration represented an average for several organisms. Rangia clams are non-selective filter feeders that transform plant detritus and phytoplankton into clam biomass. They move very little compared to fish and crabs and are suitable indicators of site quality.

Rangia clams are eaten by fish, crustacea, and waterfowl (La Salle and de la Cruz 1985). Fish that eat rangia include important commercial species such as spot (Leiostomus xanthurus), Atlantic croaker (Micropogon undulatus), and southern flounder (Paralichthys lethostigma). Ring-necked duck (Aythya collaris), greater scaup (Aythya marila), lesser scaup (Aythya affinis), black duck (Anas rubripes), mallard (Anas platyrhynchos), and ruddy duck (Oxyura jamaicensis) eat rangia. However, a review of the literature does not indicate that the levels of fluoride found in tissues of rangia clam should present any threat to waterfowl that consume them[1]. Crustacea that consume rangia clams include blue crab (Callinectes sapidus) and white shrimp (Peneaus setiferus). Blue crab consumption of rangia clams and other benthic food organisms with body burdens of fluorides and other contaminantes may be associated with the frequency of shell lesions (approximately 10% and regionally up to 90%, Weinstein et al. 1992) in blue crabs taken from the Pamlico River.

Gemperline et al. (1992) analyzed trace and minor element concentrations in blue crab gills, muscle, and hepatopancreas tissues using principle component analysis. They concluded that diseased and non-diseased crabs had distinctly different concentrations of minor and trace elements. The authors speculated that since many of the elements observed in the tissue samples are normally not soluble, fluoride or phosphate concentrations in the Pamlico River may be causing enhanced solubility of these elements. Weinstein et al. (1992) stated that toxic concentrations of metals or trace elements could potentially cause shell disease through physical degradation of the hypodermis which secretes the shell or through interference of wound repair. Another more subtle mechanism that could cause shell disease would involve disruption of copper regulation which would result in stress from reduced oxygen transport to the tissues (Engel 1987, Engel and Brouwer 1984, Depledge and Bjerregaard 1989). Noga et al. (1990) observed that Pamlico

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[1] Jim Fleming, National Biological Survey, Raleigh, NC, personal communication.

River blue crabs had reduced hemocyanin levels. Hemocyanin is the respiratory pigment which contains 50-60 % of the copper in blue crabs. Pamlico River blue crabs were described as unhealthy in terms of behavior, survival, hemocyte levels, and wound repair capability (Weinstein 1991). Healthy blue crabs in tanks develop lesions when exposed to Pamlico River water (Noga et al. 1990).

The North Carolina Division of Environmental Management (DEM) conducted surveys in the Pamlico River at sites that have contaminated sediments. Samples of midge (Chironomus sp.) larvae which inhabit the sediment and the midges were examined for the presence of tooth deformities. Larva midge tooth deformity is being evaluated by DEM as a potential biological indicator of contaminant loading in North Carolina (Lenat 1993). Tooth deformities in midge larvae are associated with contaminated sediments (Hamilton and Saether 1971, Warwick 1985, 1988, 1990, Warwick and Tisdale 1988). Midge tooth deformities were observed at levels above background in Kennedy Creek, but midges were not present in the sandy sediment around Texasgulf which precluded this technique from being used there. These findings are especially interesting because blue crabs and midge are both arthropods, both undergo growth molts, and both midge teeth and blue crab shells are composed of chitin. It can be assumed that midge larvae developed on the sites at which they were collected in contrast to blue crabs, which are less sedentary.

Gizzard shad are planktivorous and longnose gar are piscivorous. Both are considered freshwater fish although they frequent brackish sites, such as those we sampled, along with some of the more typical estuarine species. They were selected for sampling because they represented a diversity of trophic levels and because they could be easily collected at all sites. Although both gizzard shad and longnose gar sampled at Texasgulf had the highest fluoride concentrations of any of the sites sampled, the difference in concentrations between Texasgulf and the other sites appeared too small to be meaningful. Fluoride accumulates in calcium rich tissues, such as bone. Therefore differences in fluoride concentrations in whole fish may have disguised the potentially major differences in fluoride exposure among sites that was indicated by sediment and rangia clam samples.

SUMMARY

Samples of sediment, rangia clams, gizzard shad, and longnose gar were collected from five sites in the lower Pamlico River and analyzed for elemental contaminants, organochlorines, aliphatic hydrocarbon, and polynuclear aromatic hydrocarbons. In general sample concentrations were either beneath the detection limit or too low to be associated with biological impacts. However, sediment concentrations of cadmium and fluoride were elevated for the Texasgulf site when compared to the other sites sampled, and were observed at levels that could be associated with biological impacts. Concentrations of fluoride in fish and clam tissue in this report are the first available for this area and subsequently should provide a reference for future comparisons.

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