Field Toxicity Tests in Three North Florida Rivers

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Abstract: Larval fathead minnows (*Pimephales promelas*), <48 hours old, were exposed to water from the Apalachicola, Choctawhatchee, and Ochlockonee rivers in northern Florida during field toxicity tests in November 1985 and April 1986. The fish were exposed for 6 or 7 days in a flow-through system to control water; full-strength river water; and 50%, 25%, and 12.5% dilutions of river water. Mortality in full-strength Choctawhatchee River water was significantly higher (40% mortality) than in control water or in the 3 other dilutions of the river water (<15% mortality). Mortalities of fathead minnows in the different dilutions of the Apalachicola and Ochlockonee rivers did not differ significantly from those in the controls. Further study is needed to determine the cause of the toxicity of Choctawhatchee River water to larval fish.

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Declines in sport and commercial fisheries in rivers and coastal waters of the Gulf of Mexico in recent years have been attributed to habitat loss, habitat alteration and manipulation, sedimentation, and inflow of contaminants from industrial and agricultural sources (Rulifson et al. 1982, Goodyear 1985). The fish and wildlife affected by these actions are aesthetically important and basic to the economy of coastal areas, particularly regarding the growing tourist industry along the Gulf Coast.

Water quality of coastal rivers is of particular concern because these rivers support indigenous fish assemblages as well as anadromous species (Bain and Bain 1982). Also, water quality of coastal rivers influences the quality of estuarine environments, which serve as critical habitats for many estuarine and marine species (Lindall 1973, Bozeman and Dean 1980).

The U.S. Fish and Wildlife Service and the Gulf Coastal States have recently taken steps to enhance populations of the Gulf Coast race of striped bass (*Morone saxatilis*) where they still exist and to reestablish them in waters within their historical ranges (Rulifson et al. 1982). Because stocks are diminished, the recruitment of striped bass is limited and the fishery depends on Federal and State stocking programs (Lukens 1988). Only a few striped bass of the Gulf Coast race of striped bass are

collected each year for the stocking program; consequently, the supply of hatchery-spawned fish of this race is generally limited and stocking demands are high.

The objective of this study was to determine the water quality in 3 rivers in northwestern Florida by using field toxicity tests (on-site assessments conducted near the study area).

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Methods

Toxicity tests were conducted on water from 2 sites on the Apalachicola River and from 1 site on the Choctawhatchee River in November 1985 and on water from 1 site each on the Apalachicola, Choctawhatchee, and Ochlockonee rivers in April 1986. The rivers are in northwestern Florida and receive drainage from Alabama, Georgia, and the coastal plain of Florida.

The 2 sites sampled on the Apalachicola River in 1985 were near Blountstown (Calhoun County), Florida; they were 1.6 km upstream and downstream from State Highway 20. Water from the Choctawhatchee River was collected at the bridge on State Highway 20 near Ebro (Walton County), Florida. Grab-samples of water were collected daily during the 6-day testing period from each of the sample sites for the 1985 toxicity tests.

In 1986, samples from the Apalachicola River were collected 100 m downstream from the end of State Highway 22A near Wewahatchka (Gulf County), Florida; those from the Ochlockonee River were taken on the east side of the stream, 150 m downstream from the U.S. Highway 90 bridge in Leon County, Florida; and those from Choctawhatchee River were collected 300 m north of the State Highway 20 bridge at Howell Bluff (Walton County), Florida. Water samples for the 1986 toxicity tests were collected from each site with battery-powered ISCO[1] Samplers (Model 1680). The samplers were programmed to collect water at 30-minute intervals and provide 24-hour composite samples that were collected daily during the 7-day tests and transported in 20-liter glass containers that had been cleaned with acid and acetone.

The toxicity tests were similar to those developed by Norberg and Mount (1985), except that a flow-through system similar to that described by Finger and Bulak (1988) was used. For each treatment (full-strength river water, river water dilution, or controls), water flowed from a 3.6-liter glass headtank through a series of 10 borosilicate glass beakers (250 ml) placed on a stair-step platform. Exposure

[1]References to trade names or manufacturers do not imply government endorsement of commercial products.

water passed from one beaker to the next in the series through a small-bore glass siphon (3 mm inside diameter). Flow rate was maintained at 2 to 3 ml/minute, which replaced the water in each beaker approximately every 2 to 2.5 hours and provided suitable dissolved oxygen concentrations (>7 mg/liter) in all the breakers.

Fish used in the tests were obtained from the Environmental Protection Agency, Athens, Georgia, for the 1985 tests and from the National Fisheries Contaminant Research Center, Columbia, Missouri, for the 1986 tests. Fathead minnows were exposed to full-strength river water and serial dilutions of 50%, 25%, and 12.5% of the river water. The dilutions were made with control water, which consisted of a 50:50 mixture of deionized water and water from a local well. The alkalinity, hardness, pH, and specific conductance of the control water were similar to those of the river waters being tested. Fish were also exposed to waters from fish-culture facilities; U.S. Environmental Protection Agency, Athens, Georgia (1985 toxicity tests) and the Richmond Hill Fish Hatchery, Richmond Hill, Georgia (1986 toxicity tests). These test groups were designated hatchery controls.

Two larval (<48 hours old) fathead minnows were placed in each beaker (N = 20 larvae/treatment) at the start of the 6-day toxicity tests in 1985, and 3 larval fish (<24 hours old) were stocked in each beaker (N = 30 larvae/treatment) for the 7-day tests in 1986. Room and test water temperatures were maintained between 23° and 26° C. Fish were fed freshly hatched brine shrimp (*Artemia salina*) twice a day during the test. Dead fish were removed from the beakers daily.

Water samples collected from the rivers near the beginning and end of each test were analyzed for organochlorine pesticides, PCBs, petroleum hydrocarbons (aliphatic and aromatic), and the elemental contaminants Al, As, Cd, Cr, Cu, Hg, Pb, Se, and Zn. Samples for elemental analyses were acidified to pH 2 with ultra-pure HNO_3. Samples for organic analyses were placed in 1-liter amber bottles and stored at 4° C. Alkalinity, hardness, pH, specific conductance, salinity, dissolved oxygen, and temperature of the treatment waters were noted daily.

Water samples for organochlorine and PCB analyses were extracted with hexane and partitioned with Ch_3CN and petroleum ether. Florisil adsorption chromatography was used for cleanup and initial fractionation of the sample extracts. PCBs were separated and further cleaned up by silica gel chromatography. Residues were measured by packed column electron capture-gas chromatography with a Varian 6000/6500 Gas Chromatograph and Varian 402 Chromatography Data Station. About 10% of the analytical results were confirmed by gas chromatography-mass spectrometry with a DB–5 bonded phase quartz capillary column and helium carrier.

Samples for petroleum hydrocarbon analyses were extracted with methylene chloride and fractionated with petroleum ether and methylene chloride by silica gel chromatography. Elutriates were cleaned up with a Florisil column and quantified with high pressure liquid chromatography.

All elemental analyses except those for As, Hg, and Se were performed by inductively coupled argon plasma emission spectrophotometry with a Jarrell-Ash Model 1100, Mark III Inductively Coupled Plasma DEC 11/23. Arsenic and Se were determined by hydride generation atomic absorption spectrophotometry on a

Perkin-Elmer Model 603 atomic absorption spectrophotometer. Mercury was analyzed by cold-vapor atomic absorption with a Perkin Elmer Model 403 atomic absorption instrument.

Blank samples, replicates, and spiked samples were used in the analyses for quality control. Limits of detection for organochlorines, polychlorinated biphenyls, and petroleum hydrocarbons were 5 μg/liter. Detection limits for the inorganic contaminants were: 0.1 μg/liter for CD; 0.2 μg/liter for Hg; 0.5 μg/liter for As, Cu, and Se; 0.9 μg/liter for Pb, 1.0 μg/liter for Al and Cr; 5.0 μg/liter for Sn; and 10 μg/liter for Zn. Percent recovery from spiked samples and percent deviation between duplicate samples were within acceptable ranges. No contaminants were found at measurable levels in the procedural blank samples.

Prior to statistical analyses, percentages of mortality were normalized by the angular transformation (Snedecor and Cochran 1973). Significant differences ($P \leq 0.05$) among treatment means for each river were determined by analysis of variance, and differences between means were determined with Tukey's studentized range test (SAS Institute Inc. 1985).

Results

The mortality of fathead minnows was significantly higher in full-strength Choctawhatchee River water than in the controls and the other dilutions of the Choctawhatchee water, except for the 25% dilution in the 1985 toxicity tests (Table 1). The high mortality in the 25% dilution was due to loss of fish from 3 beakers when the siphons became clogged and the beakers overflowed. Mortality did not differ significantly among or between river water dilutions and controls in the toxicity tests of water from the Apalachicola and Ochlockonee river waters.

Water quality was similar at the 3 test sites. Alkalinities ranged from 20 to 60 mg/liter $CaCO_3$ and specific conductance from 90 to 160 μmhos/cm. Values for the control water and hatchery control water were slightly higher than those in the river waters: Alkalinity, 20–100 mg/liter; hardness, 80–100 mg/liter; and specific conductance, 100–195 μmhos/cm. The pH of the rivers ranged from 7.0 to 7.6, control water from 7.1 to 7.4, and hatchery control water from 7.6 to 7.9.

No organochlorine pesticides, PCBs, or petroleum hydrocarbons were detected in the water samples from the 3 rivers. The concentrations of elemental contaminants were low, ranging only from the lower limits of detection to normal background levels (Table 2). The elemental composition was similar among rivers and control waters, except for zinc, which was higher in the control water than in the river waters and hatchery control waters, and aluminum, which was higher in the Apalachicola and Ochlockonee rivers than in the controls and Choctawhatchee River.

Discussion

Testing early-life stages of fish has been shown by Benoit et al. (1982) and Van Leeuwen et al. (1985) to be a reliable method for predicting long-term chronic

Table 1. Mean percent mortality and standard deviation (in parentheses) of fathead minnows exposed in field toxicity tests to water from Gulf Coast rivers in November 1985 ($N = 20$) and April 1986 ($N = 30$). Means with the same superscript within a toxicity test (vertical column) are not significantly different ($P \leq 0.05$).

Date and Test water	Toxicity Test: Apalachicola (upper)	Apalachicola (lower)	Choctawhatchee
1985			
River water			
100%	20[a]	15[a]	40[a]
	(24)	(22)	(29)
50%	15[a]	0[a]	15[b]
	(15)	(0)	(22)
25%	5[a]	10[a]	45[a]
	(15)	(20)	(41)
12.5%	0[a]	15[a]	15[a]
	(0)	(22)	(22)
Control	10[a]	5[a]	0[b]
	(20)	(15)	(0)
Hatchery control	15[a]	0[a]	10[b]
	(22)	(0)	(20)
	Apalachicola	Ochlockonee	Choctawhatchee
1986			
River water			
100%	20[a]	13[a]	40[a]
	(16)	(16)	(29)
50%	13[a]	6[a]	6[b]
	(16)	(13)	(19)
25%	10[a]	6[a]	0[b]
	(15)	(13)	(0)
12.5%	3[a]	3[a]	0[b]
	(9)	(9)	(0)
Control	3[a]	3[a]	6[b]
	(9)	(9)	(13)
Hatchery control	3[a]	0[a]	3[b]
	(9)	(0)	(9)

effects of contaminants. Larval fish have also been used to assess the toxicity of natural river systems (Hall et al. 1985, Finger and Bulak 1988). Results of this study indicate that water from the Choctawhatchee River was more toxic to larval fathead minnows than control water and would likely produce similar mortalities in naturally spawned and hatchery-stocked fishes such as striped bass.

Contaminants (but not necessarily those that were measured on the water from the study sites) are a probable cause of this poor water quality, and a more thorough evaluation of contaminants in the Choctawhatchee River is warranted to identify the cause of the increased fathead minnow mortality. Contaminants not measured in

Table 2. Elemental contaminants (μg/liter) measured in water used on field toxicity tests from the Apalachicola, Choctawhatchee, and Ochlockonee rivers in November 1985 and April 1986. All values were below the limits of detection for Hg (<0.20), Se (<0.50), and Sn (<5.0).

Date and Test water	Al	Cd	Cr	Cu	Pb	Zn
1985						
Apalachicola (upper)	—[a]	0.4	3.0	1.9	<1.0	20.0
Apalachicola (lower)	—	<0.1	3.0	<0.6	<1.0	20.0
Choctawhatchee	—	0.5	2.0	2.9	1.0	80.0
Control	—	0.1	2.0	2.5	<1.0	350.0
Hatchery control	—	0.3	2.0	<0.6	<1.0	30.0
1986						
Apalachicola	440	0.4	<1.0	1.0	1.0	10.0
Ochlockonee	440	0.2	13.0	0.5	2.9	20.0
Choctawhatchee	70	0.4	<1.0	0.5	<0.9	20.0
Control	20	0.2	<1.0	<0.5	0.9	240.0
Hatchery control	10	0.2	2.0	0.7	<0.9	10.0

[a]Not measured.

the test waters or combinations of contaminants at or below the limits of detection may have been responsible for the mortality of larval fathead minnows in these studies. Low concentrations of organic and inorganic contaminant mixtures in river and laboratory systems have been shown to reduce survival of juvenile striped bass in other studies (Hall et al. 1984, Palawski et al. 1985, Mehrle et al. 1987).

Although concentrations of specific elemental contaminants in water samples from the study sites were not considered to be high enough to adversely affect fathead minnows (Table 2), aluminum concentrations were higher in the Apalachicola and Ochlockonee river waters (440 μg/liter) than in the Choctawhatchee River (70 μg/liter). Buckler et al. (1987) determined that concentrations as low as 100 μg/liter were toxic to young striped bass, particularly at reduced pH. Similarly, Hall et al. (1985) attributed mortality of juvenile striped bass in a tributary of the Chesapeake Bay to aluminum at concentrations of 480 to 4,100 μg/liter. Juvenile striped bass may thus be susceptible to mortality from aluminum toxicity in Gulf Coast rivers, and future assessments of the suitability of these systems for striped bass should investigate aluminum levels.

Reduced survival of fathead minnows in the Choctawhatchee River water supports the concern for striped bass inhabiting the system. State and Federal fishery managers should be cautious in their efforts to enhance the striped bass fishery in the Choctawhatchee River. Mortality of fathead minnows exposed to Choctawhatchee River water was probably a conservative estimate of the mortality of larval striped bass in the river, because striped bass are more sensitive than fathead

minnows to most contaminants (Palawski et al. 1985). Their survival in the Choctawhatchee River would probably be substantially lower than that of fathead minnows in the present study.

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