Evaluation of the Toxicity of an Effluent Entering the Deep Fork River, OK

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Study problem

An effluent from a local manufacturing plant enters the Deep Fork River near the City of Okmulgee, OK

The discharge point is <1 mile upstream of the boundary of the Deep Fork National Wildlife Refuge

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Two mussel kills (August 2005 and September 2011) downstream of the effluent discharge in the Deep Fork River

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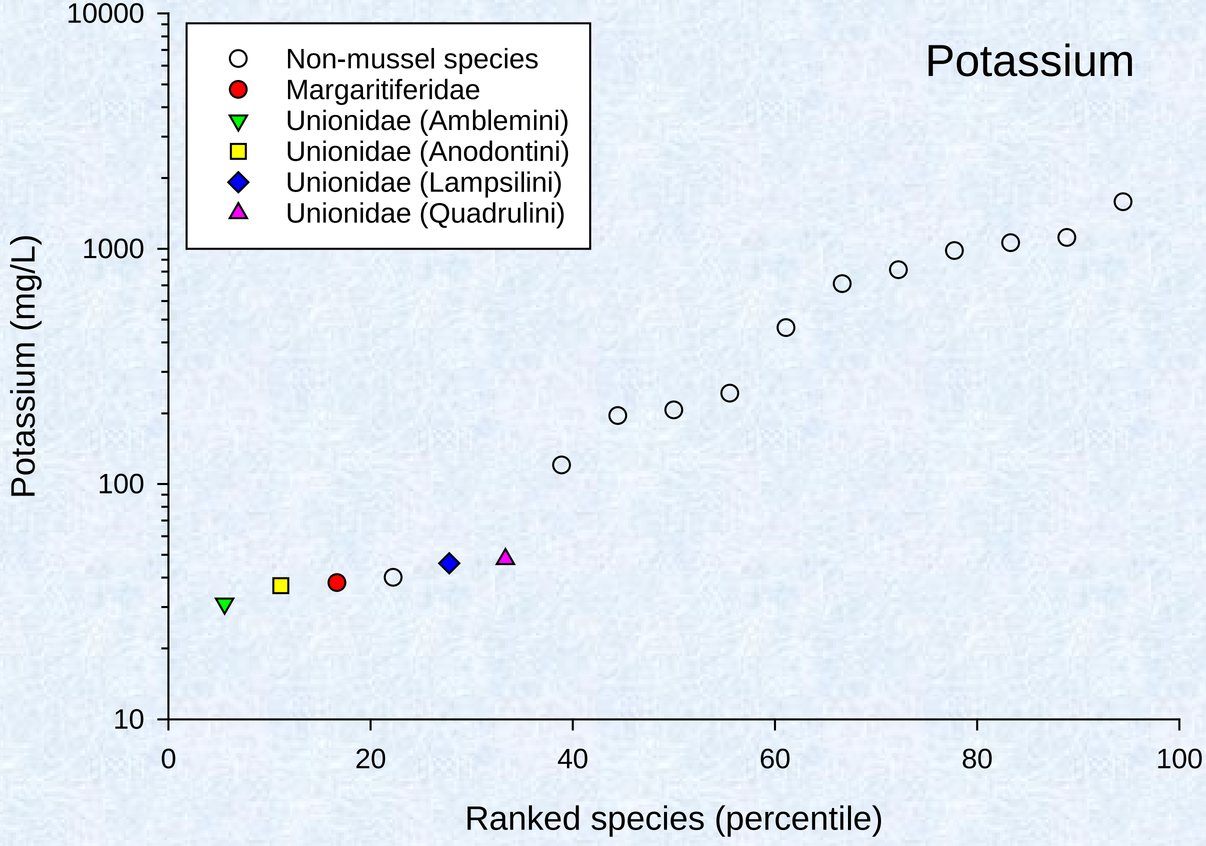
Ranked freshwater mussel species mean acute value in potassium database (Wang et al. 2017)



Previous studies reported elevated potassium of up to 80 mg/L in water contaminated by the effluent.

Study objectives

- Assess the potential toxicity of the effluent to a unionid mussel (*Lampsilis siliquoidea*) and two commonly tested species (*Ceriodaphnia dubia*, *Pimephales promelas*) in short-term (7-day) effluent test (Task 1)
- Evaluate the relative sensitivity of the three species to potassium in 7-day toxicity test (Task 2)
- Determine the influence of water quality characteristics on the acute toxicity of potassium to mussels and cladocerans (Task 3)

Fatmucket
Lampsilis siliquoidea

Cladoceran
Ceriodaphnia dubia

Fathead Minnow
Pimephales promelas

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Tasks 1 & 2: Test conditions for 7-day effluent or potassium toxicity tests in basic accordance with USEPA (2002) and ASTM (20016a)

Test species: *L. siliquoidea*, *C. dubia, P. promelas*

Toxicant: Effluent (effluent test); KCl (K test)

Test type: Static renewal

Temperature: 25±1°C

Age of organism: ~7 d (mussels), <24 h (cladocerans), <48 h (minnows)

Organism #/chamber: 10 (mussels), 1 (cladocerans), 10 (minnows)

Replicate #: 4 (mussels), 10 (cladocerans), 4 (minnows)

Feeding: algae (mussels), YCT+algae (cladocerans), brine shrimp (minnows)

Dilution water: Upstream water of the effluent outfall

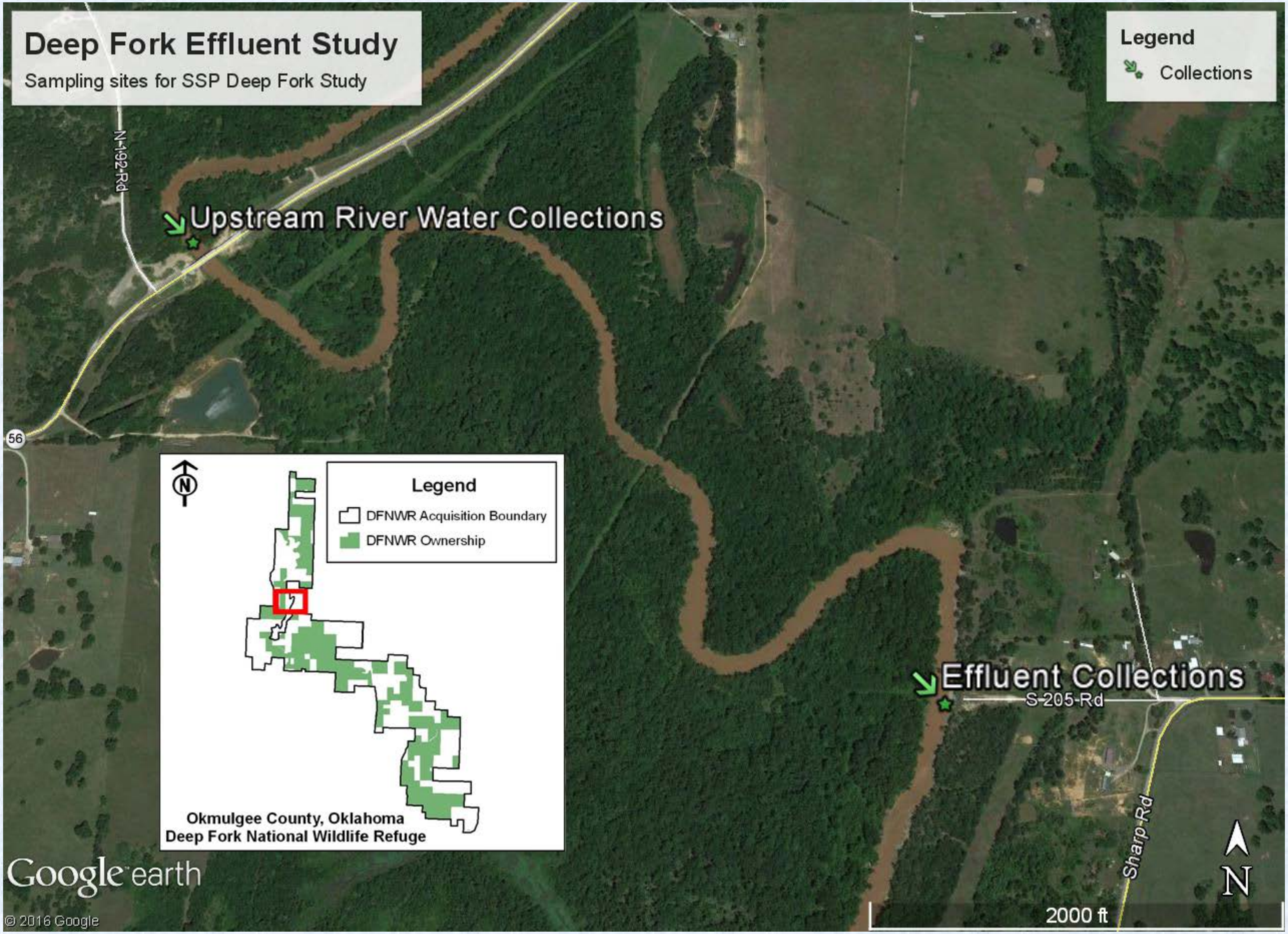
Test concentrations: 5 concentrations plus a control (50% dilution series)

Chemical analyses: Major cations and anions

Endpoint: Survival, length (mussels), reproduction (cladocerans), biomass (minnows)

Test acceptability criterion: ≥80% control survival

Water collection sites in the Deep Fork River



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Does the effluent contain elevated major ions? **Water quality measurements of water samples collected from the upstream sampling site and effluent discharge outfall, July 26, 2016**

Water	pH	Conductivity (µS/cm)	Alkalinity (mg/L as $CaCO_3$)	Hardness (mg/L as $CaCO_3$)	Ammonia (mg N/L)	Major cations and anions (mg/L)							
						Ca	Mg	Na	K	Cl	NO_3	SO_4	PO_4
Upstream water	8.2	521	134	158	0.02	32	19	43	4.1	79	1.3	16	<1.5
Effluent	8.6	1853	492	80	0.11	19	8.7	258	195*	175	38	28	119

* The potassium value is >4-fold greater than EC50s (50% effect concentrations) from acute 96-h potassium exposures with 5 mussel species in 100 mg/L hardness water (Wang et al. 2017)

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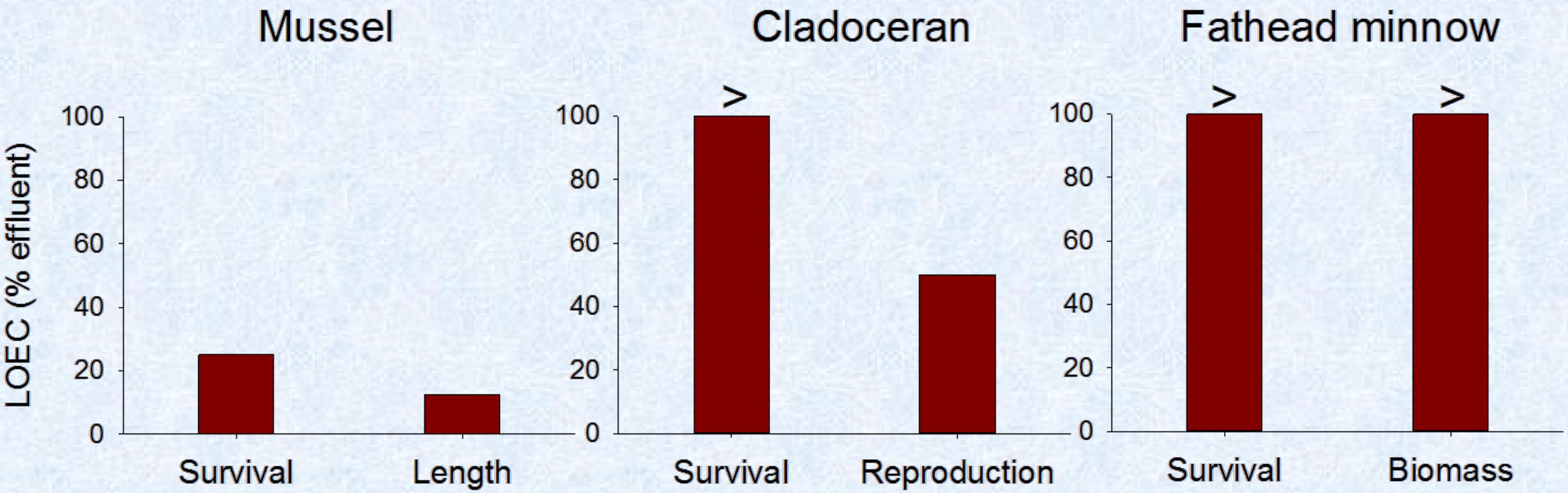
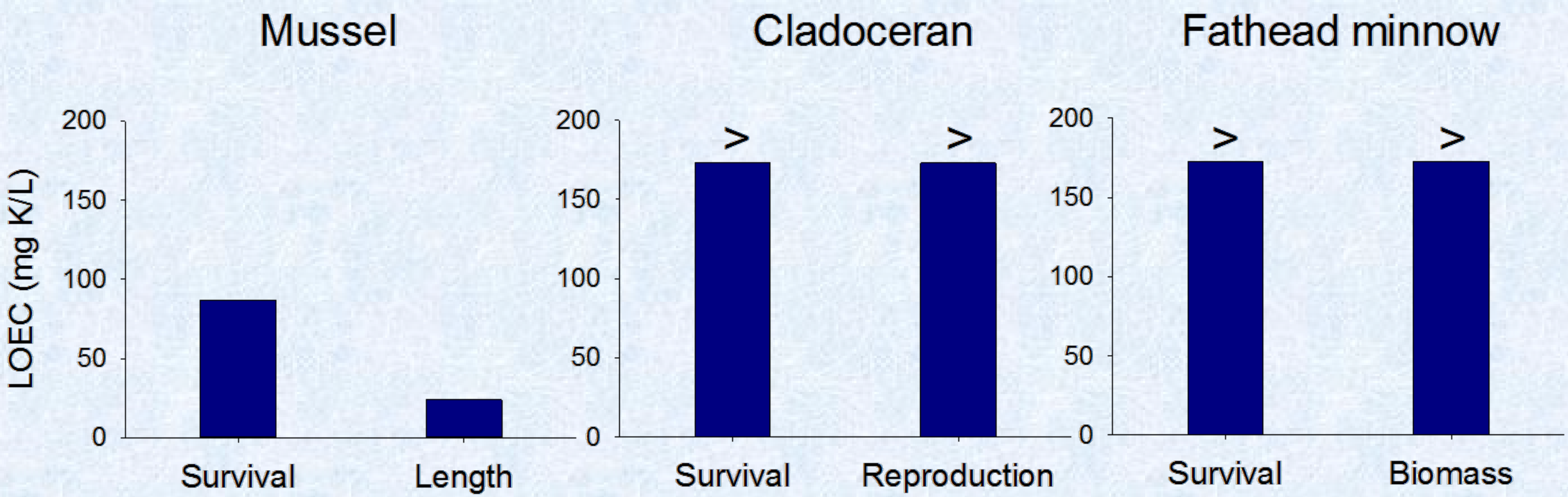
Is the effluent toxic? The lowest-observed-effect concentration (LOEC) of the effluent in 7-d effluent tests with the 3 species



LOEC: The lowest concentration causes a significant reduction in an endpoint relative to the control (Dunnett's test or Steel's many-one test, p<0.05)

>: Greater-than value (i.e., no effect with 100% effluent)

How sensitive are the mussels to potassium, compared to commonly tested species? The LOEC for potassium in <u>7-d potassium toxicity tests</u> with the 3 species



Dilution water characteristics:

Dilution water	pH	Conductivity (µS/cm)	Alkalinity	Hardness	Ammonia (mg N/L)	Major cations and anions (mg/L)							
			(mg/L as $CaCO_3$)			Ca	Mg	Na	K	Cl	NO_3	SO_4	PO_4
Upstream water	8.3	822	246	294	0.01	57	34	74	3.4	140	<1	39	<1.5

Task 3: Test conditions for acute potassium toxicity test in 4 reconstituted waters and 2 temperatures following ASTM (2016ab)

Test species: *L. siliquoidea*, *C. dubia*
Toxicant: KCl
Test duration: 96 h (mussels), 48 h (cladocerans)
Test type: Static renewal
Temperature: 23±1°C (and 28±1°C in a test water)
Age of organism: ~7 d (mussels), <24 h (cladocerans)
Organism #/chamber: 5
Replicate #: 4
Feeding: None
Dilution water: 4 reconstituted waters representing water quality (major ions) in the Deep Fork River
Test concentrations: 5 concentrations plus a control (30% dilution series)
Chemical analyses: Major cations and anions
Endpoint: Survival
Test acceptability criterion: ≥90% control survival

Water quality characteristics in samples from the upstream site in the Deep Fork River

Upstream water	Sampling date	pH	Conductivity (µS/cm)	Alkalinity (mg/L as $CaCO_3$)	Hardness (mg/L as $CaCO_3$)	Major cations and anions (mg/L)							
						Ca	Mg	Na	K	Cl	NO_3	SO_4	PO_4
1	4/18/2016	8.2	859	240	290	68	36	92	4	154	<1	40	<1.5
2	5/18/2016	8.3	822	246	294	57	34	74	3.4	140	<1	39	<1.5
3	6/27/2016	8.0	755	192	242	52	28	73	4.1	148	1.3	31	<1.5
4	7/26/2016	8.2	521	134	158	32	19	43	4.1	79	1.3	16	<1.5
5	8/1/2016[a]	7.6	278	68	60	17	8.3	16	3.9	27	2.1	12	<3.0

[a] A high flow period due to heavy rain.

Water quality characteristics in 4 reconstituted waters representing water quality range in the Deep Fork River

Dilution water	pH	Conductivity (µS/cm)	Alkalinity (mg/L as $CaCO_3$)	Hardness (mg/L as $CaCO_3$)	Major cations and anions (mg/L)					
					Ca	Mg	Na	K	Cl	SO_4
300 hard water	8.4	1008	170	310	62	31	87	4.0	174	56
150 hard water	8.2	555	130	160	38	16	44	2.1	88	27
75 hard water	8.0	290	73	84	18	8.0	22	1.0	35	14
35 hard water	7.9	151	36	40	9.4	4.2	11	0.5	21	7.5

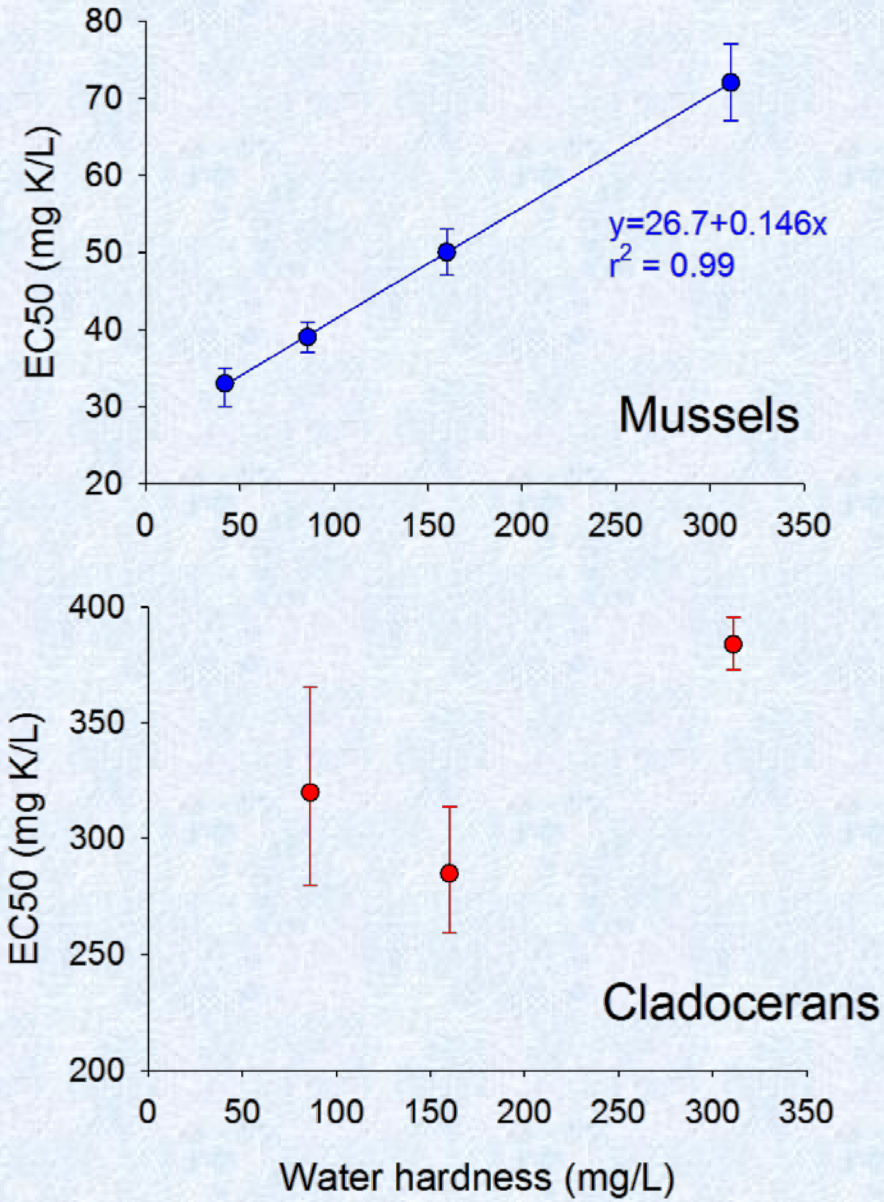
Reconstituted waters were prepared by matching the water-quality characteristics of the upstream waters: (1) diluting CERC well water (hardness ~300 mg/L) with deionized water; and (2) adding reagent grade salts into the water to match the concentrations of major ions in the upstream water samples.

Water quality characteristics in 4 reconstituted waters, compared to the upstream water samples in the Deep Fork River

Dilution water	pH	Conductivity (µS/cm)	Alkalinity (mg/L as $CaCO_3$)	Hardness (mg/L as $CaCO_3$)	Major cations and anions (mg/L) Ca	Mg	Na	K	Cl	SO_4
300 hard water	8.4	1008	170	310	62	31	87	4.0	174	56
Upstream water #1	8.2	859	240	290	68	36	92	4.0	154	40
150 hard water	8.2	555	130	160	38	16	44	2.1	88	27
Upstream water #4	8.2	521	134	158	32	19	43	4.1	79	16
75 hard water	8.0	290	73	84	18	8.0	22	1.0	35	14
Upstream water #5	7.6	278	68	60	17	8.3	16	3.9	27	12
35 hard water	7.9	151	36	40	9.4	4.2	11	0.5	21	7.5

Does water quality influence potassium toxicity?

Acute 50% effect concentrations (EC50s) for potassium in 4 reconstituted waters with different hardness at 23°C



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Significant correlation between EC50s and hardness with mussels, but not cladocerans

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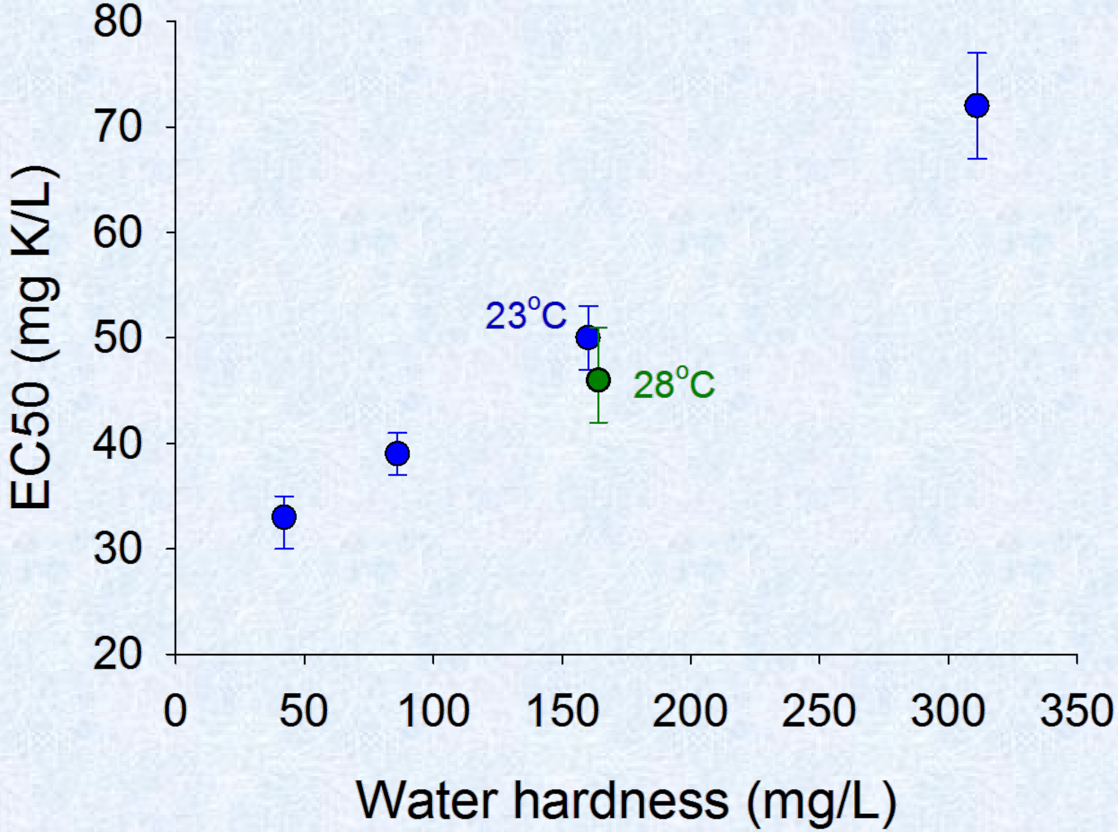
Does temperature influence potassium toxicity?

Acute EC50s for mussels tested at 23°C and 28°C



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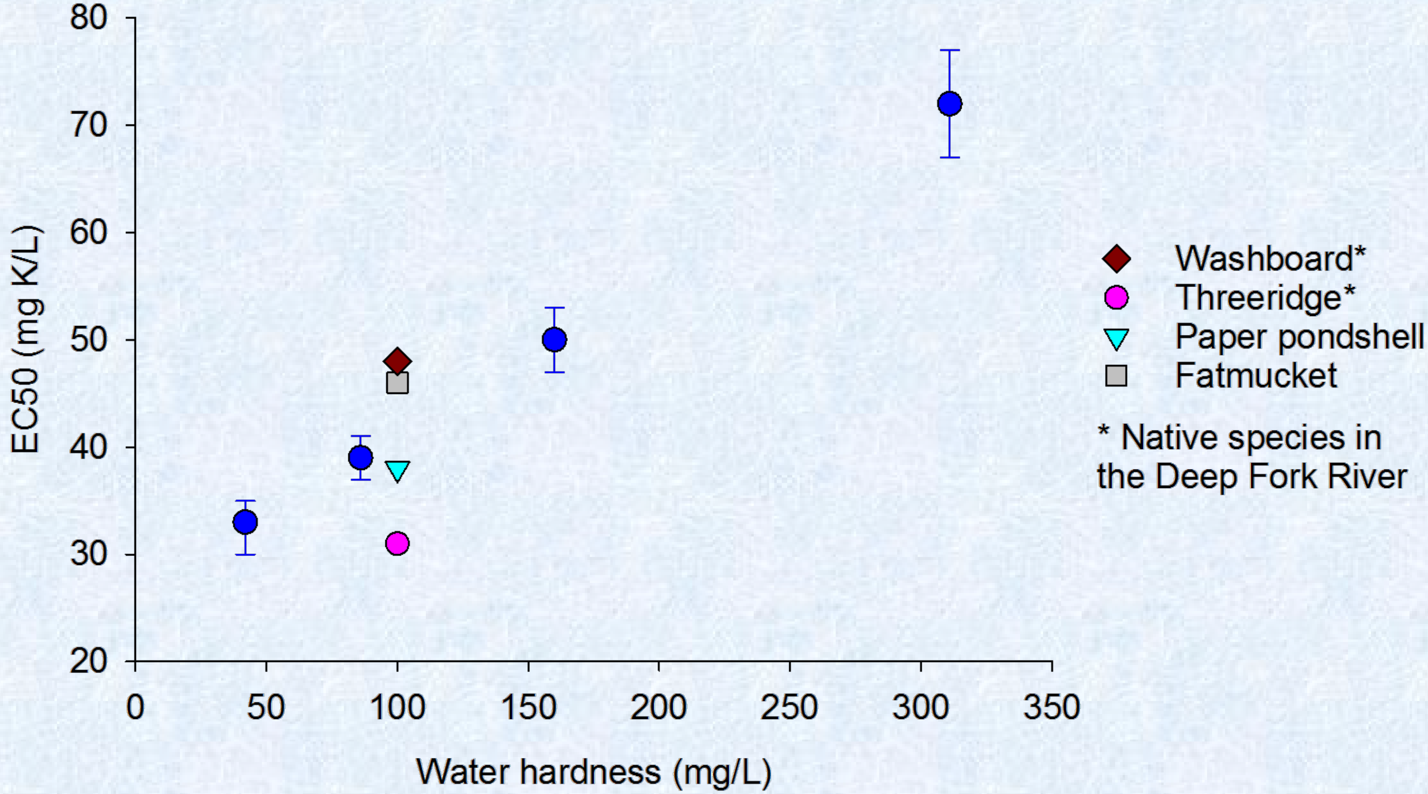
Acute toxicity of potassium to fatmucket (this study) and 4 mussel species tested previously under similar conditions (hardness 100 mg/L, 23°C; Wang et al. 2017)



All EC50s were lower than the concentrations of potassium in the effluent collected at or downstream of the effluent discharge outfall:

- 80 mg K/L at hardness 170 mg/L (ODEQ 2011)
- 195 mg K/L at hardness 80 mg/L (this study)

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Conclusions:

- The effluent contained elevated conductivity, major cations and anions, and was toxic to mussels and cladocerans tested
- The mussel was more sensitive than the cladoceran and fathead minnow to the effluent or potassium, and thus, the two commonly tested species do not represent the sensitivity of mussels
- Mussel testing should be included in effluent permitting process

Conclusions (Continued):

- Water hardness influenced the acute toxicity of potassium to the mussel; however, the toxicity decreased only by a factor of 2 with increasing water hardness from 40 to 300 mg/L as $CaCO_3$
- The potassium concentrations in the effluents from previous and present studies were above the acute EC50s and short-term LOECs for mussels

Future study:

- Evaluate the influences of water quality and temperature on potassium toxicity in longer-term (e.g., 28 d) exposures
- Evaluate the toxicity of sediments contaminated by the effluent to mussels and other benthic invertebrates
- Evaluate the effect of the mussel kill events on mussel populations