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# Oil sands tailings pond sediment toxicity to early life stages of northern pike (*Esox lucius*)

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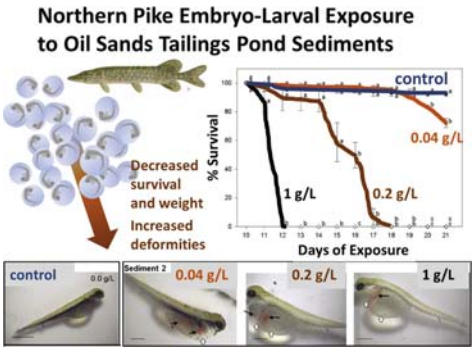
## HIGHLIGHTS

- Northern pike embryos were exposed to oil sands tailings pond sediments.
- A mixture of PAHs and alkylated PAHs were found in the sediments.
- Decreased survival, weight, increased deformities accompanied increasing PAH levels.
- These results can aid evaluation of ecological effects if accidental release occurs.

## GRAPHICAL ABSTRACT

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## ABSTRACT

The Athabasca River in Alberta flows through natural sources of eroding oil sands bitumen and oil sands mining operations that may result in low level contamination of surface waters. Northern pike (*Esox lucius*) are apex predators and important food and game fish species native to the Athabasca River system. This species has the potential to be exposed to both natural and anthropogenic sources of contamination from oil sands related materials throughout its life cycle. Pike are difficult to rear in the laboratory and little information exists on the toxicity of oil sands related materials to this key indigenous fish species. In this study, the potential effects of two sediment samples collected from different areas of one tailings pond in the Athabasca oil sands area are assessed in a daily renewal bioassay on early life stages of northern pike. Gametes were collected from spawning wild pike captured from a reference site outside of the oil sands area. Fertilized eggs were exposed to control water or increasing concentrations of tailings pond sediments for 21 days, coinciding with initiation of exogenous feeding and completion of yolk absorption. Developing fish were examined for survival and changes in body weight, length, and development. Embryos exhibited increased developmental abnormalities and decreased growth and survival with increasing sediment concentration. Both sediment samples had similar levels of naphthenic acids and similar types of PAHs, with alkylated PAHs dominating. However, concentrations of total and alkylated PAHs differed between sediment samples and were related to increasing developmental abnormalities and decreased growth and survival. This is consistent with developmental changes observed with exposure to PAHs in other fish

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species. These results provide information on the effects of tailings pond sediments comprising mixtures of PAHs and alkylated PAHs on the development and survival of a key species in the northern aquatic ecosystem.



## 1. Introduction

The Athabasca River and its tributaries in the Athabasca oil sands region flow through areas of exposed, naturally-occurring bitumen, and erosion results in the various oil sands related compounds (OSRCs) entering the aquatic ecosystem. Polycyclic aromatic hydrocarbons (PAHs) and naphthenic acids (NAs) and other dissolved organic compounds are naturally occurring constituents of bitumen, and the sediment and surface waters of the Athabasca River basin contain these chemicals (Headley et al., 2001; Kavanagh et al., 2009; Yergeau et al., 2012). Little information exists concerning the effects of these compounds in aquatic ecosystems, but there is some evidence that fish populations found within areas of naturally occurring oil sands come into contact with these compounds (Tetreault et al., 2003a, 2003b). Increased liver enzyme activity and decreased steroid hormone production have been shown in fish species indigenous to the oil sands region of the Athabasca River, including slimy sculpin (*Cottus cognatus*) and pearl dace (*Semotilus margarita*) (Tetreault et al., 2003a, 2003b). Similarly, pearl dace and brook stickleback (*Culaea inconstans*) collected from tributaries of the Athabasca River within the exposed oil sands area exhibited differences in parasitism and gill histology relative to these same species collected from a reference site, suggesting differences in the health of fish populations between these areas (Raine et al., 2017a).

A predominant constituent of tributary sediments and oil sands tailings are alkylated PAHs (Madill et al., 2001; Headley et al., 2001; Colavecchia et al., 2004). Exposure to sediments from tailings ponds and natural river sediments increased embryo-larval mortality and the incidence of abnormalities in early life stages of white sucker (*Catostomus commersoni*) and fathead minnows (*Pimephales promelas*) was associated with alkylated PAHs (Colavecchia et al., 2004, 2006). Many studies show that early life stages of fish are especially sensitive to the effects of alkylated PAHs and PAHs in weathered crude oil and oil sands related samples (Guiney et al., 1997; Marty et al., 1997a, 1997b; Carls et al., 1999; Couillard, 2002; Rhodes et al., 2005; Sorhus et al., 2017). Early life stages of fish exposed to alkylated PAH mixtures in crude oil show similar developmental malformations (such as craniofacial deformities, hemorrhages and edema) as fish exposed to oil sands sediments (Marty et al., 1997a, 1997b; Carls et al., 1999; Couillard, 2002; Colavecchia et al., 2004, 2006; Raine et al., 2017b; Sorhus et al., 2017).

Abnormal development of early life stages of walleye (*Sander vitreus*) and fathead minnows have been observed with exposure to the organic fraction of oil sands process water (He et al., 2012; Marentette et al. 2015). This organic fraction contains NAs and a number of other dissolved organic chemicals, which are another possible source of toxicity (Wiseman et al., 2013). Spinal malformations, yolk sac and cardiac edema have been shown in early life stages of zebrafish exposed to an extract of NAs (Wang et al., 2015). Similar effects were seen after embryo-larval exposure of fathead minnows and walleye to the oil sands process water organic fraction (He et al., 2012; Marentette et al. 2015). It has been suggested that toxicity of the organic fraction of oil sands process water to walleye and fathead minnow early developmental stages is due to the generation of reactive oxygen species and oxidative stress (Wiseman et al., 2013; Marentette et al., 2017).

Northern pike (*Esox lucius*) are top predators and popular food and game fish indigenous to the Athabasca River (Tripp and McCart, 1979; Nelson and Paetz, 1992). Pike are spring spawners and increases in water levels during this time could increase oil sands related constituents in Athabasca surface waters from erosion of exposed bitumen, and anthropogenic sources of contamination arising from atmospheric deposition or tailings pond seepage (Kelly et al., 2009; Hall et al., 2012; Frank et al., 2014). Early life stages of these fish would be most at risk to oil sands related constituents during this sensitive developmental period.

The purpose of the present study was to expose early life stages of northern pike to sediments from an oil sands tailings pond, to provide information on the effects oil sands related constituents on a key apex species in the local aquatic ecosystem. A semi-static exposure assay was developed using sediment samples from two different sites in a single tailings pond. Oil sands related constituents can vary spatially within tailings ponds (Frank et al., 2016). This study will provide information that can be used to compare the toxicity of oil sands related constituents between lab species of fish and those indigenous to the Athabasca River basin. The toxicity data generated from this investigation will be essential for evaluating the possible ecological effects of accidental release of tailings pond sediments.

## 2. Material and methods

### 2.1. Tailings pond sediment collection

In September 2009, two 2 L sediment samples were collected from two different sites within one tailings pond (Mildred Lake Settling Basin, Syncrude) in Fort McMurray, Alberta. Samples of sediment were collected using a stainless steel saucepan connected to a 2 M wooden pole. Samples were collected at a depth of 1.2 M, and pooled in food grade polyethylene bags inside 20 L white plastic pails with lids. Sampling procedures were standardized between sites. Pictures of the sampling equipment are in Supplemental Data Fig. S1. These sediment samples were enclosed in airtight plastic bags and held at 4 °C in darkness during shipping and storage, until the pike assay was implemented.

### 2.2. Collection of gametes

Spawning wild northern pike (*Esox lucius*) were caught using trap nets from Lake Diefenbaker, SK, by staff of the Saskatchewan Fish Culture Station (Fort Qu'Appelle, SK) in spring 2011. Lake Diefenbaker was chosen as a reference site for fish collection as it has excellent water quality and the possibility of contamination from oil or PAHs was expected to be very low. Gametes were collected from one female and three male pike, and the milt was pooled prior to fertilization. Fertilized eggs were maintained in 10 °C lake water and transported to the Aquatic Toxicology Research Facility (Toxicology Centre, University of Saskatchewan, Saskatoon, SK Canada). Once in the aquatic facility, the pike eggs were sorted to remove unfertilized eggs and allocated to treatment beakers. The sediments were introduced to the treatment beakers and the exposure assay began 48 h post-fertilization.

The Animal Research Ethics Board, University of Saskatchewan approved this study (Animal Utilization Protocol # 20100042), and all work adhered to Canadian Council on Animal Care guidelines.