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Bio-physicochemical effects of gamma irradiation treatment for naphthenic acids in oil sands fluid fine tailings

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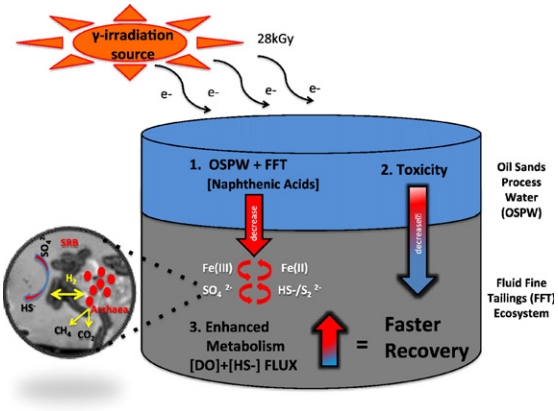
HIGHLIGHTS

- Gamma irradiation substantially reduced concentrations of ecotoxic naphthenic acids
- Acute toxicity was reduced in gamma irradiated process water
- Gamma irradiated tailings exhibited increased rates of microbial respiration

GRAPHICAL ABSTRACT



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ABSTRACT

Naphthenic acids (NAs) are persistent compounds that are components of most petroleum, including those found in the Athabasca oil sands. Their presence in freshly processed tailings is of significant environmental concern due to their toxicity to aquatic organisms. Gamma irradiation (GI) was used to reduce the toxicity and concentration of NAs in oil sands process water (OSPW) and fluid fine tailings (FFT). This investigation systematically studied the impact of GI on the biogeochemical development and progressive reduction of toxicity using laboratory incubations of fresh and aged tailings under anoxic and oxic conditions. GI reduced NA concentrations in OSPW by up to 97% in OSPW and in FFT by 85%. The GI-treated FFT exhibited increased rates of biogeochemical change, dependent on the age of the tailings source. Dissolved oxygen (DO) flux was enhanced in GI-treated FFT from fresh and aged source materials, whereas hydrogen sulfide (HS^-) flux was stimulated only in the fresh FFT. Acute toxicity to *Vibrio fischeri* was immediately reduced following GI treatment of fresh OSPW. GI treatment followed by 4-week incubation reduced toxicity of aged OSPW to *V. fischeri*.

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1. Introduction

Extraction of bitumen to produce oil from the Athabasca oil sands in northern Alberta, Canada has created large volumes of waste materials termed fluid fine tailings (FFT), which consist of a mixture of water, dissolved salts, sand, clay, residual bitumen and naphtha products (Allen, 2008). To comply with a zero discharge policy, oil sands process materials (OSPM) are stored in large tailings ponds to promote the separation of the oil sands process-affected water (OSPW) and FFT before the tailings containment area is eventually reclaimed (Quagraine et al., 2005). The OSPW or the overlying surface water is recycled and re-used in the bitumen extraction process, which reduces freshwater demand; however, various organic and salt constituents become concentrated, further increasing FFT's toxicity (Hwang et al., 2013). Tailings ponds are stratified ecosystems that harbor a diverse microbial community (Chi Fru et al., 2013; Penner and Foght, 2010), involved in the aerobic and anaerobic degradation of organic compounds (Siddique et al., 2011) and elemental reduction/oxidation (redox) cycling within the ponds (Chen et al., 2013; Stasik et al., 2014). The acute and chronic toxicity of OSPW to aquatic organisms is mainly attributed to a refractory mixture of naturally occurring acid extractable organics known as Naphthenic Acids (NAs), which are comprised of alkyl substituted, acyclic and cyclo-aliphatic saturated carboxylic acids with/without S and/or N atoms as impurities (Grewer et al., 2010; Kannel and Gan, 2012).

In tailing ponds, NA concentrations range from 20 to 120 mg L^{-1} (Holowenko et al., 2002). Natural biodegradation can reduce NA concentrations. However, concentrations seldom decline below 20 mg L^{-1} because of the presence of refractory NAs in the mixture (Quagraine et al., 2005). Biodegradation studies determined that NAs with greater alkyl side chain branching are more refractory than short chain linear compounds. Findings suggest the more refractory NAs found in OSPW contain larger, alkyl branched side chains that are less bioavailable due to a greater degree of intramolecular hydrogen bonding (Johnson et al., 2010). Although toxicity declines through time in the environment, the refractory isomers of the NA mixture can persist for many years. To address these aspects, various treatment options have been explored to promote or accelerate the degradation process. These range from chemical treatments to bioremediation using microbial enrichments and phytoremediation (Biryukova et al., 2007; Scott et al., 2008; Toor et al., 2013; Quagraine et al., 2005).

Several of these treatments rely on the production of hydroxyl radicals ($OH^{\bullet}$) to oxidize NAs; these include ultraviolet photolysis (McMartin et al., 2004), photo-catalysis with TiO_2 (Mishra et al., 2010), and ozonation (Martin et al., 2010; Scott et al., 2008), which have been proven effective in selectively degrading NAs, leading to increased bioavailability for further microbial bioremediation, and reducing the toxicity of OSPW. One drawback of these treatments is that they are limited to recycled OSPW from which particulate matter has settled or has been filtered and therefore cannot be used on turbid fluids such as FFT (Martin et al., 2010).

A less explored treatment option is gamma irradiation (GI), which is well developed for the treatment of complex refractory organic compounds in other industrial wastewaters, including contaminated groundwater and sewage sludge (Wang and Wang, 2007). The radiolysis reactions from GI produce very reactive species (Eq. (1)), which have the energy to oxidize organic compounds.

$$GI + H_2O \rightarrow OH^{\bullet} + e^{-} + H^{\bullet} + H_3O^{+} + H_2O_2 + H_2 \quad (1)$$

Previous studies have shown GI to be effective at degrading refractory organic compounds such as cyclohexanebutyric acid (a model NA) (Jia et al., 2015), phenols (Chitose et al., 2003), tetrachloroethane and trichloroethane (Gerhinger and Matschiner, 1998), polychlorinated byphenyls (Arbon et al., 1996) and trinitrotoluene (Lee and Lee, 2005). Unlike photolytic or ozonation technologies, GI can penetrate turbid fluids and sediments, providing the potential to treat large volumes of OSPM as a means of degrading and eliminating NAs.

To date, there has been no investigation of the impacts of GI on the biogeochemical function and the resulting toxicity associated with OSPM upon treatment - particularly, the potential for biotransformation of NAs following GI-treatment within redox gradients and the resulting flux of dissolved species. The diffusive boundary layer (DBL) is the epibenthic layer of water overlying the sediment surface of aqueous environments, through which molecular diffusion is the dominant transport mechanism of dissolved species. The flux of dissolved constituents across the DBL and into or from the sediment is a measure of the rate of mineralization of organic matter within the sediment and the dissolution or precipitation of minerals (Glud et al., 2007). These expressed fluxes influence sediment oxygen demand (SOD) and will determine the FFT's influence on the overall quality of the water cap (Chen et al., 2013; Matzinger et al., 2010). If GI treatment alters the composition of NA isomers, a clear understanding of the resulting biogeochemical processes controlling the generation of metabolites from either organic or inorganic materials must be assessed.

It is hypothesized that the ionizing effects of GI degrade NA isomers within OSPM into a more bioavailable carbon source, stimulating the biological (kinetic) responses of microorganisms inoculated into GI-treated process materials. GI-treated OSPM are expected to show markedly reduced toxicity and greater microbial respiration rates, measurable as altered fluxes and quantities of microbial respiratory products in GI-treated materials. The objectives of this study were to (1) assess the biological and physicochemical responses to irradiated OSPW and FFT from fresh and aged sources; (2) determine the efficacy of GI-treatment in reducing NA concentrations and; (3) assess the toxicity of GI-treated and untreated OSPW to *Vibrio fischeri*. The hypotheses were tested by Incubating GI-treated materials inoculated with their native microbial consortium using laboratory microcosms and determining changes in biogeochemical processes through time. Exposure to GI eliminates all biota without affecting sediments' geochemistry (Bank et al., 2008). Post-treatment inoculation re-establishes the native microbial community, allowing assessment of the bio-physicochemical responses to an altered organic carbon source in a relatively unchanged geophysical environment.

2. Material and methods

2.1. Microcosm design

Fresh and Aged OSPW and FFT source material was collected from South Tailings Pond (STP) and Pond 1A (P1A), respectively, operated by Suncor Energy Inc., located in the Athabasca region, Alberta. P1A is one of the oldest tailing basins in the Suncor lease area, created in 1968 (Fedorak et al., 2003). It does not receive freshly processed FFT but is used for the recycling of recently processed OSPW on the surface. From a chemical, biological and treatment perspective, aged material from P1A represents a source of stable, consolidated material containing refractory NA compounds remaining in the FFT, which have undergone, or been exposed to natural biodegradation. In contrast, the STP pond is 9 years old and routinely receives freshly processed FFT, which is expected to contain a full suite of both labile and refractory NA isomers. The STP represents a tailings basin early in development from the perspective of treatment, chemistry and biology (e.g. microbial community succession).

The study design was as follows: Each sample material comprised 2 treatments: untreated (100% untreated) and GI-treated (90% gamma-irradiated material into which was mixed 10% by volume untreated inoculum from the pond source) × 2 sources of material × 2 atmospheric conditions, × 6 time points, with n = 2 replicates per treatment. The 10% inoculum added to the GI-treatments served as a source of microbial biomass to promote microbial colonization in a GI-treated FFT parent material.

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