

Development of a Novel Engineered Bioprocess for Oil Sands Process-Affected Water and Tailings Fines/Bitumen/Water Separation

Kerry N. McPhedran, Md. Shahinoor Islam, and Mohamed Gamal El-Din
Department of Civil and Environmental Engineering, University of Alberta

December 2014



Oil Sands Research and Information Network

The Oil Sands Research and Information Network (OSRIN) is a university-based, independent organization that compiles, interprets and analyses available knowledge about managing the environmental impacts to landscapes and water affected by oil sands mining and gets that knowledge into the hands of those who can use it to drive breakthrough improvements in regulations and practices. OSRIN is a project of the University of Alberta's School of Energy and the Environment (SEE). OSRIN was launched with a start-up grant of \$4.5 million from Alberta Environment and a \$250,000 grant from the Canada School of Energy and Environment Ltd.

OSRIN provides:

- **Governments** with the independent, objective, and credible information and analysis required to put appropriate regulatory and policy frameworks in place
- **Media, opinion leaders and the general public** with the facts about oil sands development, its environmental and social impacts, and landscape/water reclamation activities – so that public dialogue and policy is informed by solid evidence
- **Industry** with ready access to an integrated view of research that will help them make and execute environmental management plans – a view that crosses disciplines and organizational boundaries

OSRIN recognizes that much research has been done in these areas by a variety of players over 40 years of oil sands development. OSRIN synthesizes this collective knowledge and presents it in a form that allows others to use it to solve pressing problems.

Citation

This report may be cited as:

McPhedran, K., M.S. Islam and M. Gamal El-Din, 2014. Development of a Novel Engineered Bioprocess for Oil Sands Process-Affected Water and Tailings Fines/Bitumen/Water Separation. Oil Sands Research and Information Network, University of Alberta, School of Energy and the Environment, Edmonton, Alberta. OSRIN Report No. TR-63. 28 pp.

Copies of this report may be obtained from OSRIN at osrin@ualberta.ca or through the OSRIN website at <http://www.osrin.ualberta.ca/en/OSRINPublications.aspx> or directly from the University of Alberta's Education & Research Archive at <http://hdl.handle.net/10402/era.17507>.

Table of Contents

LIST OF TABLES	iv
LIST OF FIGURES	iv
REPORT SUMMARY	v
ACKNOWLEDGEMENTS	vi
1 INTRODUCTION	1
1.1 Motivation.....	2
1.2 Advantages of BioTiger™	2
1.3 Objectives	3
2 METHODOLOGY	4
2.1 BioTiger™ Culture	4
2.2 Experimental Scheme	4
2.3 Oil Sands Process-Affected Water Experiments	6
2.4 Toxicity Quantification using Microtox®	7
2.5 Acid-Extractable Fraction Quantification.....	8
2.6 Naphthenic Acids Quantification by HRMS	8
2.7 Mature Fine Tailings Experiments.....	8
2.7.1 Methane and Carbon Dioxide Quantification by GC-FID	11
2.7.2 MFT Solids Consolidation	11
3 OSPW RESULTS AND DISCUSSION.....	11
3.1 Microtox® Results	11
3.2 Acid Extractable Fraction Results	13
3.3 Naphthenic Acid Results.....	14
4 CONCLUSIONS AND RECOMMENDATIONS	18
4.1 Conclusions for Oil Sands Process-Affected Water	18
4.2 Recommendations for Oil Sands Process-Affected Water	18
5 REFERENCES	18
6 GLOSSARY	21
6.1 Terms	21
6.2 Acronyms.....	21

6.3	Chemicals.....	22
	LIST OF OSRIN REPORTS	23

LIST OF TABLES

Table 1.	OSPW and MFT sample sources.	5
Table 2.	Microtox [®] results after 5 and 15 minutes for each of the four OSPW samples....	12

LIST OF FIGURES

Figure 1.	Schematic of OSPW and MFT experiments at three temperatures (8 °C, 22 °C and 35 °C).....	6
Figure 2.	Experimental shaker setup for OSPW treatments at three temperatures (8 °C, 22 °C and 35 °C).....	7
Figure 3.	MFT treatments at three temperatures (8 °C, ~22 °C and 35 °C) for 2 L graduated cylinders including schematic of column dimensions.	9
Figure 4.	Example large settling column (20 L of MFT) image and schematic for the room temperature (~22 °C) settling columns.	10
Figure 5.	Toxicity to <i>V. fischeri</i> for each OSPW before (control) and after treatment at various temperatures.	13
Figure 6.	The AEF for each OSPW sample (CNRL, SHLL, SUNC, and SYCD) before (control) and after treatment at various temperatures.	14
Figure 7.	The naphthenic acid concentrations for each OSPW sample (CNRL, SHLL, SUNC, and SYCD) before (initial control) and after (final) treatment at 8 °C. ...	15
Figure 8.	The naphthenic acid concentrations for each OSPW sample (CNRL, SHLL, SUNC, and SYCD) before (initial control) and after (final) treatment at 22 °C. .	16
Figure 9.	The naphthenic acid concentrations for each OSPW sample (CNRL, SHLL, SUNC, and SYCD) before (initial control) and after (final) treatment at 35 °C. .	17

REPORT SUMMARY

The oil sands bitumen extraction process results in the creation of waste products including oil sands process-affected water (OSPW) and mature fine tailings (MFT). Many technologies are currently under investigation to treat these waste products that are currently contained in vast storage ponds. Biodegradation is a promising treatment method, however, the current biodegradation rates for indigenous bacteria in storage ponds are very slow and need to be enhanced for this process to be considered viable. The BioTiger™ consortium has been successfully used for the treatment of oil contaminated soils making it a potentially useful bacterial assemblage for the treatment of both OSPW and MFT.

In this study, BioTiger™ was not successful for treatment of OSPW after 24 h experiments at 8, 22 and 35 °C. Results for toxicity to *V. fischeri* were inconclusive, while there was no reduction in either the acid extractable fraction (AEF) or the naphthenic acid (NA) contents. The MFT experiments have not commenced as of yet due to the unavailability of some samples. These experiments will start in January 2015 and run for approximately four months. It is expected that the longer duration will allow the BioTiger™ to biodegrade organics in the MFT.

Although the current OSPW experiments did not produce anticipated results, further research is planned to better assess the ability of BioTiger™ to degrade OSPW organics. These experiments will include longer experimental durations, higher initial bacterial concentrations and/or amendment with easily degradable organics. These new conditions should aid the consortium to better acclimate to, and degrade, recalcitrant OSPW organics.

ACKNOWLEDGEMENTS

The Oil Sands Research and Information Network (OSRIN), School of Energy and the Environment (SEE), University of Alberta provided funding for this project.

1 INTRODUCTION

Alberta is the global leader in the development of oil sands. Extensive work has been done by industry, government and communities towards an environmentally sustainable oil sands development. The Alberta Energy Strategy (Government of Alberta 2008) illustrates the government's commitment to a responsible development of energy resources that balances economic growth and environmental protection. The Water Management Framework (Alberta Environment and Fisheries and Oceans Canada 2007) limits water withdrawal from the Athabasca River, especially during low flow periods, and encourages the industry to reduce water demand through recycling. Environmental operating approvals require that water produced from oil sands operations cannot be discharged directly into the environment unless authorized by the approval¹, thus requiring storage in tailing ponds. Tailings generated from oil sands processing are mainly composed of suspended and dissolved particles, natural organic matter, unrecovered hydrocarbons, trace metals and many other toxic compounds such as naphthenic acids (NAs) which could have significant environmental and social impact in the vicinity of discharge areas.

The oil sands regions in northern Alberta contain the world's third largest proven oil reserves of over 170 billion barrels that in 2010 were extracted at a rate of over 2 million barrels per day (Giesy et al. 2010). This production is expected to surpass 3.5 million barrels per day by 2020. Overall, the bitumen extraction process associated with mining operations produces over 1 million m³ per day of fluid fine tailings that is stored in vast tailings ponds which eventually need to be treated (Siddique et al. 2014). The reuse of this oil sands process-affected water (OSPW) leads to the reduction of fresh water needed, while consolidating the mature fine tailings (MFT) in tailings ponds both increase the available storage and further reclamation efforts for the over 182 km² of tailings ponds (Alberta Environment and Sustainable Resource Development 2013). The Energy Resources Conservation Board (2009) requires oil sands mining companies to significantly reduce fine particles in liquid tailings and make tailings solids ready for reclamation in the near future.

The treatment of OSPW has been investigated using various methods including advance oxidation processes, biodegradation, membranes, and adsorption; these treatments are aimed at reducing NAs which have been attributed to OSPW toxicity (Drzewicz et al. 2012, Gamal El-Din et al. 2011, Pereira et al. 2013, Wang et al. 2013). In addition, natural MFT consolidation is a very slow process that needs to be accelerated. Accelerated dewatering and detoxification has been investigated by evaluating the biodegradation of various organics by microbial communities found in tailings ponds (Fedorak et al. 2002, Holowenko et al. 2000, Penner and Foght 2010, Siddique et al. 2007) and promoting their activity using methods including addition of larger organic size fractions, binders such as gypsum, and addition of labile organic substrates (Siddique et al. 2012, 2014, Voordouw 2012). The ability to use a single biological consortium,

¹ See, for example, Suncor approval clauses 4.2.1 and 4.2.8 <https://avw.alberta.ca/pdf/00000094-02-00.pdf>

such as the proposed BioTiger™² community, would be beneficial for its ability to simultaneously treat both OSPW and MFT.

1.1 Motivation

BioTiger™, and associated extracellular polymeric substances created by this consortium, have the potential to degrade OSPW and MFT organic compounds and coagulate/flocculate fine clays and adsorb cations from OSPW and tailings, thereby reducing tailings volumes and facilitating existing OSPW and tailings treatment. Aeration, mixing and bulk transport that traditionally enhance bitumen recovery and tailings capture can also promote microbial activity in bioreactors. By regulating these bioprocesses, a continuous production of desired by-products and extended durations for microbial activity can be achieved without the continued renewal of microbial biomass. This outcome can be achieved through an in-line bioreactor that has microbial recycling. Should the microbial consortium be released from the bioreactor they may continue to actively participate in bioremediation of the OSPW and tailings solids in dedicated disposal areas.

Thus, the proposed OSPW and tailings bioprocessing technology can help provide cleaner energy production using natural microbial processes to potentially replace and/or minimize chemical additives, and allow bioremediation and soil reclamation. Along with the potential of having enhanced tailings capture, this integrated approach addresses environmental issues such as reducing water use, improving water quality (reduced fines, organics and cation content), facilitating tailing reclamation and minimizing the impact on local ecosystems.

1.2 Advantages of BioTiger™

BioTiger™ shows rapid and complete degradation of aliphatic and aromatic hydrocarbons, produces copious amounts of novel surfactants, is tolerant of both chemical and metal toxicity and has demonstrated high metabolic activity at temperature and pH extremes. Although originally developed and used by the U.S. Department of Energy for the bioremediation of oil-contaminated soils, recent efforts have proven that BioTiger™ can also be used to increase hydrocarbon recovery and degradation from oil sands tailings, as well as effectively treat OSPW. This enhanced *ex situ* oil recovery process utilizes BioTiger™ to optimize bitumen separation. For example, a floatation test protocol with oil sands (Shell Canada) from Fort McMurray was used for initial BioTiger™ evaluation. A comparison of hot water extraction/floatation test of the oil sands performed with and without the addition of BioTiger™ demonstrated a 50% improvement in separation as measured by gravimetric analysis in 4 h and a five-fold increase at 25 hrs. Since BioTiger™ performs well at high temperatures, it can potentially be applied in wastewaters at elevated temperatures to enhance recovery of hydrocarbons from oil sands, process tailings, or other complex recalcitrant matrices. Advantages of natural bacterial products are their biodegradability, synergistic activity, and negligible toxicity.

² See <http://techportal.eere.energy.gov/technology.do/techID=326>

Moreover, BioTiger™ can degrade toxic compounds in the tailings as proven with other complex matrices (Berry et al. 2006). Although mechanisms for some of these processes have been investigated in the laboratory, the information on precise conditions operating in the environment is limited. Moreover, intrinsic microbial activity in the oil sands setting, for example tailing ponds, is known as a long-term, and to certain extent uncontrolled, event. We believe that the application of the well-designed microbial consortium organized in an engineered bioreactor may improve *in situ* activity.

1.3 Objectives

This research program evaluated the use of BioTiger™ for an innovative engineered bioprocess using microbial activity for the treatment of oil sands process-affected water (OSPW). The team included established researchers from the University of Alberta and the Savannah River National Laboratory (SRNL) and combined expertise in petroleum engineering, petroleum and environmental microbiology, chemical and environmental engineering, and biotechnology to conduct interdisciplinary research to improve our fundamental understanding of OSPW and MFT treatment using a biological consortium (BioTiger™).

Current oil sands processing technologies result in the generation of stable dispersions of fine solids which impact bitumen recovery and tailings reclamation. Typically, bitumen and other toxic compounds remain chemically bound and become long-term sources of soil, air, and water pollution. We proposed a novel environmentally friendly biotechnology that can be used to improve bitumen recovery, tailings dewatering and sedimentation through natural bioprocesses. The core element of this technology is the BioTiger™ multispecies microbial consortium (US Patents No. 7,472,747 B1 – Brigmon and Berry (2009) and No. 7,473,546 B2 – Brigmon et al. (2009)) isolated from a century-old oil refinery in Europe. This bacterial consortium produces a variety of enzymes and metabolites (surfactants, solvents, and polymers) that provide the potential ability to remediate recalcitrant polycyclic aromatic hydrocarbons (PAHs), naphthenic acids (NAs) and heavy metals; flocculate/coagulate clay fines and cations; and alter interfacial forces between bitumen, solids and water. BioTiger™ can thrive in extreme environments (e.g., temperature) during bitumen and tailings processing, and maintain sufficient microbial activity to remove hydrocarbons bound to solids in tailings, therefore, allowing settling and separation of solids.

The objective of this project was the understanding of the bioremediation parameters of BioTiger™ for OSPW and MFT and how they can be optimized for more efficient treatment. We applied BioTiger™ for the treatment of OSPW (and will be doing so for MFT) from different oil sands companies with simultaneous tailings settling, hydrocarbon recovery/degradation, and reuse and/or remediation of the water. The specific objectives were as follows:

1. Develop BioTiger™-based bioprocess for OSPW treatment, naphthenic acid and hydrocarbon recovery/degradation, and tailings capture.
2. Optimize BioTiger™-based bioprocess to environmental conditions (e.g., temperature) observed during OSPW and MFT processing and treatment.

Objective 1

Oil sands process-affected water from four oil sands companies (Syncrude Canada Ltd., Suncor Energy Inc., Shell Canada, and Canadian Natural Resources Ltd.) was used to assess the efficacy of the BioTiger™ community. The MFT was treated with microbial cultures in column tests and then collected and analyzed for:

1. bacteria interaction with tailing compounds,
2. bitumen content and floatation,
3. liquid/solids separation (i.e., densification), and
4. quality and quantity of solid deposit and recovered water.

The toxicity of the samples was determined before and after the bioprocess. The OSPW was tested for reduction of the acid extractable fraction (AEF), naphthenic acids and toxicity.

Objective 2. A variety of environmental conditions associated with oil sands operations, including temperature and viscosity variation were addressed. The characterization of microbial activity and tailing behavior was performed with the experimental procedure described above.

2 METHODOLOGY

2.1 BioTiger™ Culture

A stock BioTiger™ culture was prepared at the Savannah River National Laboratory using MFT mixed with a Bushnell-Haas medium³. The culture was delivered on ice to the University of Alberta in 50 mL vials and stored at 4 °C until used.

Two aliquots of a Bushnell-Haas medium were prepared by suspending 1.64 g of Bushnell-Haas broth in 500 mL of distilled water within a 1 L flask and stirring until completely dissolved. The solution was sterilized by autoclaving at 15 psi and 121 °C for 15 min. To each flask about 25 mL of the BioTiger stock solution, 0.5 g yeast extract (carbon source), and 0.5 g of MFT (hydrocarbon source) were added and the flasks placed on an orbital shaker at 150 RPM at room temperature ($20 \pm 1^\circ\text{C}$) for seven days to allow for bacterial growth. After this incubation time the bacterial cells per mL were assessed by measuring the transmittance at 600 nm with a known value for 10^8 cells/mL of 0.13 to 0.14. The cells/mL values were confirmed using plate counts. The culture (at 10^9 cells/mL) was used for seeding of OSPW experiments as outlined below (the same methods will be used to prepare the cultures for the MFT experiments).

2.2 Experimental Scheme

Information for OSPW and MFT samples along with acronyms used in this study are included in Table 1. Four individual OSPW and three MFT samples have been considered for use in the current experiments. Overall, OSPW and MFT samples show marked variability in physico-

³ See <https://www.sigmaaldrich.com/content/dam/sigma-aldrich/docs/Fluka/Datasheet/b5051dat.pdf>

chemical parameters making experiments using individual samples less applicable to the other samples (Drzewicz et al. 2012, Gamal El-Din et al. 2011, Wang et al. 2013). By using multiple samples for OSPW and MFT a true representation of the ability of BioTiger™ to treat the various matrices will be elucidated.

Table 1. OSPW and MFT sample sources.

Matrix	Sample	Acronym
OSPW	Canadian Natural Resources Limited	CNRL
	Shell Canada	SHLL
	Suncor Energy Inc.	SUNC
MFT	Syncrude Canada Ltd.	SYCD
	Shell Canada	SHLL
	Suncor Energy Inc.	SUNC
	Syncrude Canada Ltd.	SYCD

The overall experimental scheme for both OSPW and MFT samples is shown in Figure 1. Three temperatures were chosen for each set of experiments:

- The lowest temperature (8 °C) was chosen to closely simulate the lowest temperature of tailings ponds in the winter months which is most applicable to the OSPW.
- 22 °C (room temperature) was chosen given the temperatures recorded in actual tailings ponds approximate this temperature for both OSPW and MFT (Fedorak et al. 2002, Holowenko et al. 2000).
- An elevated temperature (35 °C) was chosen to approximate the temperature of freshly received tailings water coming directly from the bitumen extraction process.

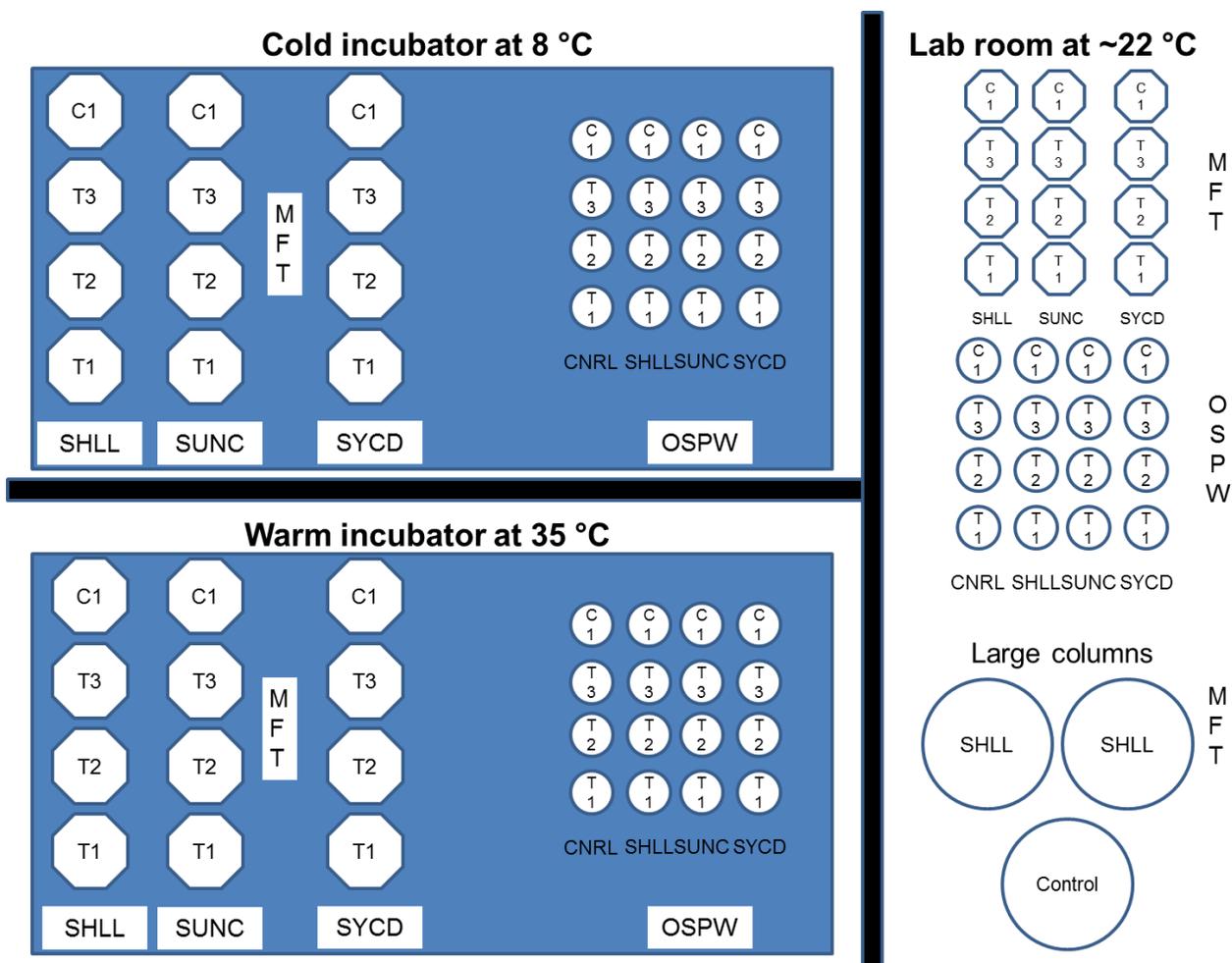


Figure 1. Schematic of OSPW and MFT experiments at three temperatures (8 °C, 22 °C and 35 °C).

2.3 Oil Sands Process-Affected Water Experiments

The OSPW experiments included control (no BioTiger™) and treatment (with BioTiger™) flasks used to determine the efficacy of the BioTiger™ consortium for reducing toxicity, AEF and NAs over 24 hours at various temperatures. The procedure for these experiments included:

1. 100 mL of OSPW samples for each of CNRL, SHLL, SUNC, and SYCD were added to four 250 mL flasks (1 control; 3 treatment replicates) for a total of 16 flasks (see Figure 1 for schematic).
2. A 1 mL aliquot of BioTiger™ at 10^9 cells/mL was added to treatment replicates for a final concentration of 10^7 cells/mL.
3. All samples were placed on an orbital shaker at 150 RPM at 8 °C for 24 hours (Figure 2).

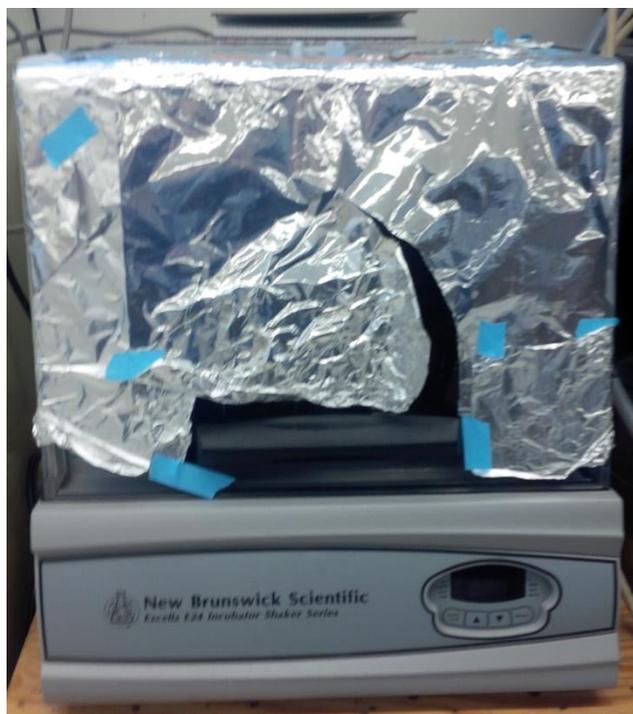


Figure 2. Experimental shaker setup for OSPW treatments at three temperatures (8 °C, 22 °C and 35 °C).

4. After 24 hours, samples were distributed for analyses (see section below) as follows:
 - a. NAs HRMS analysis → treatment samples were pooled with a 1 mL aliquot from each treatment (3 mL total) placed into a 5 mL vial and stored at 4 °C until analyzed
 - b. Microtox[®] analysis → a 5 mL aliquot was placed into a 10 mL vial and analysis completed immediately
 - c. FTIR analysis → the remaining sample (~93 mL) was filtered using a 0.45 µm nylon membrane and stored at 4 °C until extracted within 48 hrs
5. Steps #1 to #4 were repeated at 22 °C and 35 °C.

2.4 Toxicity Quantification using Microtox[®]

The toxic effects of untreated and treated OSPW and MFT samples toward *Vibrio fischeri*⁴ were measured using a Microtox 500 Analyzer (AZUR Environmental, Carlsbad, CA, USA) and the 81.9% screening test protocol (AZUR Environmental, Microtox Omni Software). All required analytical materials were purchased from Azur Environmental. The percentage of inhibition after incubation and the inhibitory volume percent concentration required to decrease the

⁴ See https://microbewiki.kenyon.edu/index.php/Vibrio_fischeri

bacterial luminescence by 50% (IC₅₀⁵) were calculated from the change in luminescence intensity at 5 minutes and 15 minutes.

2.5 Acid-Extractable Fraction Quantification

About 90 mL of OSPW for each sample was filtered through 0.45 µm Nylon filter (diam. 47 mm) (Sigma-Aldrich). A 50 mL aliquot was placed into a 100 mL beaker and the pH adjusted to 2.5 using 2M H₂SO₄. Samples were transferred into individual 250 mL separatory funnels and 25 mL of dichloromethane (DCM) was added. Samples were mixed well by shaking the separatory funnel vigorously for 2 min with frequent opening to release generated gases. The mixture was allowed to separate (~ 4 min) and the bottom layer containing the DCM extract was separated into a 50 mL glass vial. This process was repeated with a second 25 mL aliquot of DCM. The resulting 50 mL of DCM was evaporated under a gentle stream of air overnight in a fume hood until complete dryness. The dried sample and vial mass were placed on an analytical balance and tared prior to adding 5.0 to 7.0 g of DCM to re-dissolve the extracts. The exact weight of the added DCM was recorded for the calculation of the AEF concentrations. A 3 mm calibrated KBr cell was used for the FT-IR analysis using an FT-IR spectrometer (PerkinElmer: Spectrum 100, ON, CA). The absorbance of the monomeric and dimeric carbonyl stretch equivalents were measured at 1,743 and 1,706 cm⁻¹ respectively. A calibrated absorbance curve was prepared using a Fluka (Sigma-Aldrich) NA mixture. The actual concentration of AEF in the sample was calculated based on the mass of DCM optima, total height of peaks (peaks at 1,743 and 1,706 cm⁻¹) and the volume of the original sample using the standard curve produced with the Fluka NA mixture.

2.6 Naphthenic Acids Quantification by HRMS

NA samples were quantified using a high performance liquid chromatograph coupled to an ion trap mass spectrometer (HPLC/MS) (Varian500-MS) equipped with a Phenomenex C₈ column as described by Afzal et al. (2012). The separating method was: mobile phase A: 100% methanol, and mobile phase B: 4 mM ammonium acetate (aqueous) with 0.1% acetic acid, gradient elution from 45% to 15% of mobile phase A over 30 min and returned to 45% of mobile phase A for 10 min. The flow was 200 µL min⁻¹ with 20 µL injection volume, and column temperature was 40 °C. The detection limit expressed as the lowest concentration 3 times higher than the noise level for the HPLC/MS method ranged from 0.02 to 0.1 ppm.

2.7 Mature Fine Tailings Experiments

Unfortunately the MFT experiments were delayed due to the inability to acquire tailings from two of the suppliers. These experiments will commence once the MFT samples are available with an estimated start in January 2015. As with the OSPW experiments, Microtox[®], AEF and NAs results for released porewaters will be used to determine the efficacy of the BioTiger[™] consortium once adequate porewaters are available.

⁵ See <http://en.wikipedia.org/wiki/IC50>

The MFT experiments will include control (no BioTiger™) and treatment (with BioTiger™) 2 L columns to determine the efficacy of the BioTiger™ consortium for reducing toxicity, AEF and NAs at various temperatures. Larger control and treatment columns will be considered for the SHLL MFT only at room temperature. The procedure for these experiments includes:

1. A 250 mL aliquot of BioTiger™ at 10^9 cells/mL will be added to 20 L of MFT in 25 L pails for SUNC and SYCD samples and three 20 L MFT in 25 L pails for SHLL for a final concentration of 10^7 cells/mL in treatment samples.
2. Control samples will be prepared using 6 L of MFT in 10 L pails for SUNC and SYCD and 26 L in two 25 L pails for SHLL.
3. Control and treatment samples will be aerated and mixed for 24 h prior to distribution into settling columns.
4. Control and treatment samples will be distributed into 2 L graduated cylinders (Figure 3) according to the schematic shown in Figure 1 for each of SHLL, SUNC and SYCD.

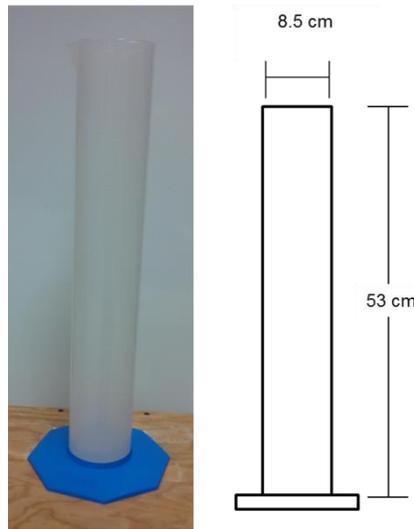


Figure 3. MFT treatments at three temperatures (8 °C, ~22 °C and 35 °C) for 2 L graduated cylinders including schematic of column dimensions.

5. Control and treatment samples will be distributed into 20 L columns (Figure 4) according to the schematic shown in Figure 1 for the SHLL sample only. The columns will be sealed on the top with a 5 L Tedlar bag attached to the top port for capture of released gases.

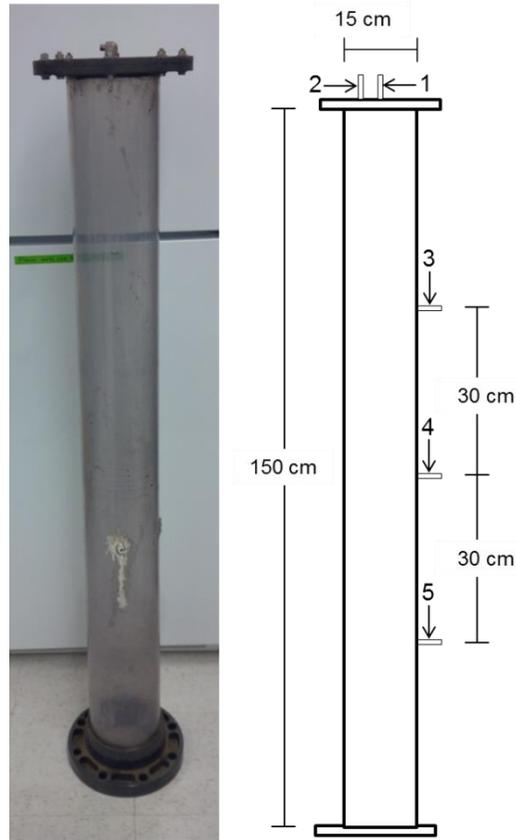


Figure 4. Example large settling column (20 L of MFT) image and schematic for the room temperature (~22 °C) settling columns.

1: Tedlar sampling bag; 2: porewater sampling port; 3,4,5: MFT sampling ports

6. The 2 L samples will be monitored weekly for settling by measurement of the mud line.
7. The 20 L columns will be monitored weekly (up to 4 months) for settling by measurement of the mud line. In addition, the gas production quantity and methane/carbon dioxide concentrations will be measured when the 5 L Tedlar bag is full (Figure 4). The bag will be replaced with a new bag when sampled and the gas production monitored until complete.
8. Once appreciable porewater is available in 2 L and 20 L columns, samples of the waters will be distributed for analysis (see section below) as follows:
 - a. NAs HRMS analysis → 2 mL aliquots from each treatment replicate were placed into a 10 mL vial and mixed. A 2 mL aliquot for each control was placed into a 5 mL vial. All samples were stored at 4 °C until analyzed
 - b. Microtox® analysis → a 5 mL aliquot was placed into a 10 mL and analysis completed immediately

- c. FTIR analysis → the remaining samples (~93 mL) was filtered using a 0.45 μm nylon membrane and stored at 4 °C until extracted within 48 hrs

2.7.1 Methane and Carbon Dioxide Quantification by GC-FID

Methane (CH₄) and carbon dioxide (CO₂) samples will be collected from each of the large columns in 5 L Tedlar bags until bags became full. The CH₄ and CO₂ concentrations will be determined using a Varian GC-FID fitted with an HP-Plot U bonded divinylbenzene/ethylene glycol dimethacrylate column (30 m X 0.32 mm ID) with helium carrier gas (30 mL/min). The oven temperature will ramp from 35 °C (5 min hold) to 225 °C at 20 °C/min.

2.7.2 MFT Solids Consolidation

The height of the mud line will be used to determine the solids consolidation and porewater recovery as per Siddique et al. (2014) according to the following set of equations:

$$V = \pi r^2 h \quad (1)$$

$$W_r = \left(\frac{V_c}{V_i} \right) \times 100 \quad (2)$$

$$\text{Consolidation (\%)} = [(V_{in} - V_{MFT})/V_{in}] \times 100 \quad (3)$$

Where

V = volume of cap water or MFT,

r = inner radius of column,

h = height of mud line,

W_r = water recovery percentage,

V_c = measured volume of cap water,

V_i = initial volume of pore water,

V_{in} = initial volume of MFT, and

V_{MFT} = measured volume of MFT.

3 OSPW RESULTS AND DISCUSSION

3.1 Microtox[®] Results

Typically greater than 50% inhibition after 15 minutes of exposure is considered to be the threshold for toxicity for *V. fischeri*. The OSPW samples showed variable impacts on *V. fischeri* with the SHLL and SYCD samples (both before and after treatment) showing inhibitions approaching or exceeding 50% for the majority of the samples (Table 2). In contrast, both the CNRL and SUNC samples had lower inhibitions that were well below the 50% threshold,

although still significant as 0% inhibition would be expected for contaminant-free water samples. Previous reports on OSPW toxicity have indicated similar toxicity ranges for OSPW samples, both above and below the 50% threshold (Klamerth et al. 2015, Nian et al. 2014, Wang et al. 2013).

Table 2. Microtox[®] results after 5 and 15 minutes for each of the four OSPW samples. Control: n = 1; Treatment: n = 3; SD = standard deviation; avg = average.

Sample		% Inhibition (5 min)			% Inhibition (15 min)		
Temperature (°C)		8	22	35	8	22	35
CNRL	Control	32.5	30.3	25.7	35.6	34.4	28.5
	Treatment (avg)	33.6	32.5	34.4	35.6	35.7	36.6
	Treatment (SD)	0.8	0.2	5.6	0.9	0.5	5.5
SHLL	Control	62.8	55.2	51.9	67.2	61.5	57.3
	Treatment (avg)	61.5	56.0	51.6	64.2	60.8	55.4
	Treatment (SD)	1.6	1.2	1.3	1.6	1.0	2.3
SUNC	Control	17.9	15.4	15.3	22.0	19.1	15.7
	Treatment (avg)	19.7	19.0	18.5	22.1	21.1	17.9
	Treatment (SD)	0.7	0.8	1.1	1.2	1.6	1.2
SYCD	Control	49.3	45.4	49.2	52.6	48.4	51.1
	Treatment (avg)	48.6	46.4	52.9	50.2	46.7	54.1
	Treatment (SD)	0.7	2.5	1.1	0.4	2.1	1.1

At 8 °C, the toxicity of the SHLL and SYCD treatments at 15 min was significantly reduced after treatment (Figure 5). This reduction in toxicity indicates that the BioTiger[™] consortium was successful in reducing the toxicity in the OSPW samples containing the highest initial toxicities, but not in the lower toxicity samples (CNRL and SUNC). Previous studies assessing toxicity reduction using *V. fischeri* following biodegradation of OSPW are not available, however, larger reductions in toxicity have been shown in ozonation studies (Gamal El-Din et al. 2011, Wang et al. 2013).

At 22 °C and 35 °C, a total of five out of eight samples showed increased toxicity of BioTiger[™] treated OSPW treatments at 15 min (Figure 5). This result was unexpected, however, it may be attributed to the degradation of OSPW compounds into more toxic by-products after treatment with the bacterial consortium. The toxicity of the control (raw OSPW) varied depending on the treatment temperature (generally decreasing with increasing temperature), thus the impact of temperature on the toxicity must be further investigated. Experiments of increased duration may be useful in determining if the toxicity will continue to show similar trends after longer time available for biodegradation by the BioTiger[™] consortium.

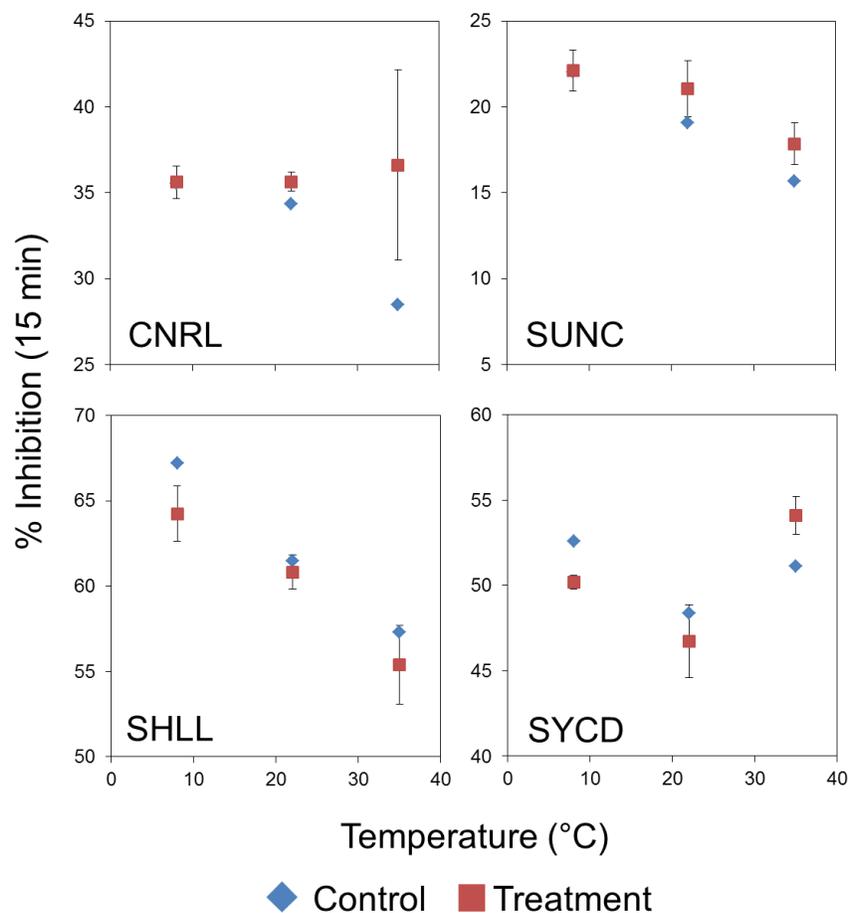


Figure 5. Toxicity to *V. fischeri* for each OSPW before (control) and after treatment at various temperatures.

Control: n = 1; Treatment: n = 3; error bars represent standard deviations.

3.2 Acid Extractable Fraction Results

The AEF varied markedly between the various OSPW samples, with treatment averages of 53.2 ± 1.1 , 48.3 ± 2.2 , 31.3 ± 2.3 , and 71.3 ± 6.3 mg/L (\pm SD) for CNRL, SHLL, SUNC, and SYCD, respectively. As the OSPW ages, the AEF decreases as the organics are slowly degraded. The SUNC samples had the lowest AEF being the oldest samples processed, while the SYCD samples were fresh OSPW having the highest AEF. It should be noted that the AEF between the OSPW samples is not expected to be constant as the samples are a result of various processing and bitumen sources. Overall, there was no impact on the AEF after treatment with BioTiger™ for 24 h (Figure 6). Given the reduction in toxicity results, reductions in the AEF were expected for the 8 °C treatment. Even if compounds are degraded, they may not be completely mineralized and may still be measured as AEF despite having reduced toxicity. It should be noted that the AEF measures all compounds with functional groups containing carboxylic acids, ketones, and aldehydes; it cannot be used as a direct measure of NA concentrations. However, AEF values are commonly used as a surrogate measure for NAs by

the oil sands industry. To determine if the bacterial consortium can degrade the AEF, further OSPW experiments of longer duration should be conducted.

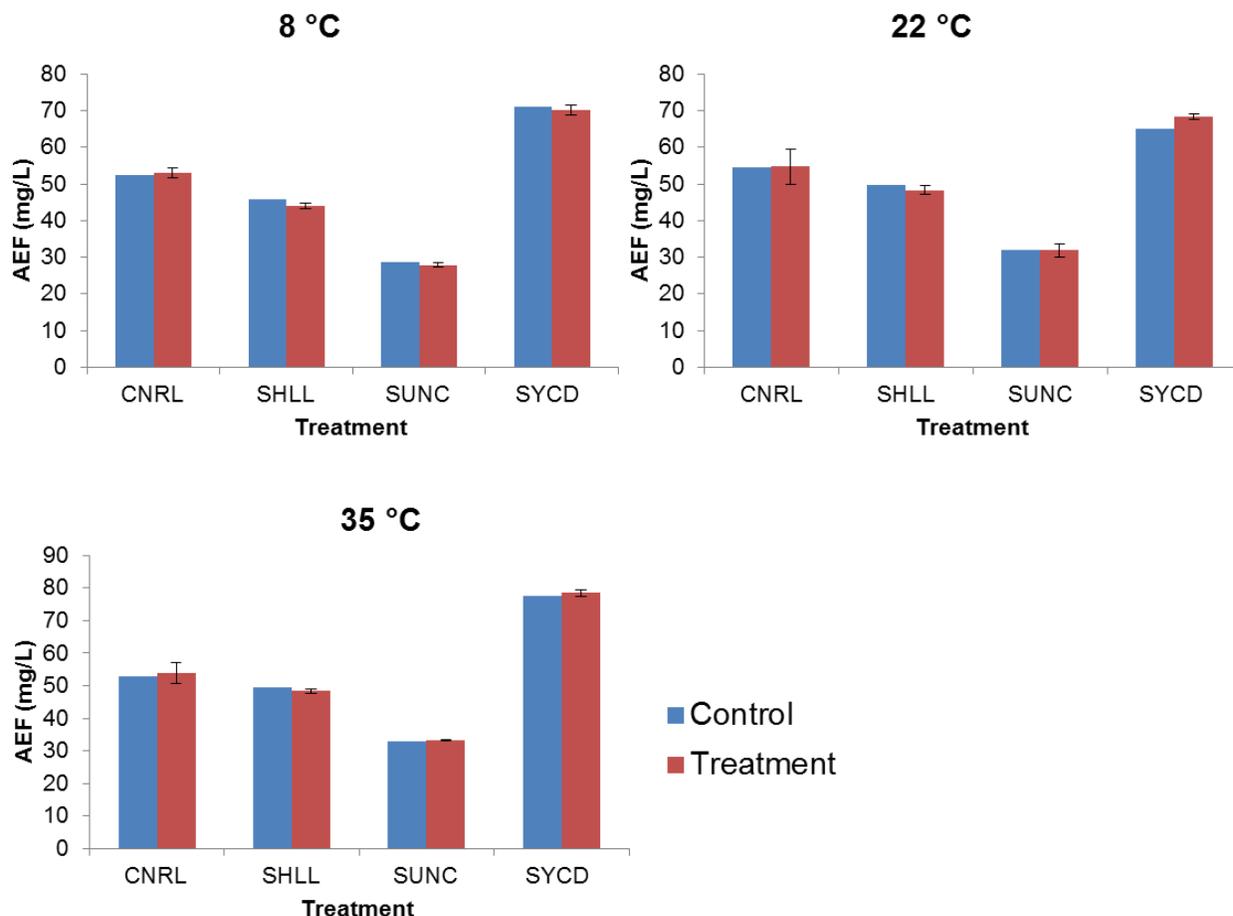


Figure 6. The AEF for each OSPW sample (CNRL, SHLL, SUNC, and SYCD) before (control) and after treatment at various temperatures. Control: n = 1; Treatment: n = 3; error bars represent standard deviations.

3.3 Naphthenic Acid Results

For the experiments conducted at 8, 22, 35 °C, the NAs varied markedly between the various OSPW samples of CNRL, SHLL, SUNC, and SYCD (Figures 7, 8 and 9). The reasons for the variation in NAs between the samples are analogous to the AEF discussed in section 3.2. Overall, there was no impact on the NAs after treatment with BioTiger™ for 24 h (Figures 7 to 9). These results were in agreement with the AEF results indicating the biological consortium was not able to degrade organics within the 24 h experiment duration. Subsequent OSPW experiments of longer duration should be conducted to determine if the bacterial consortium can degrade the NAs.

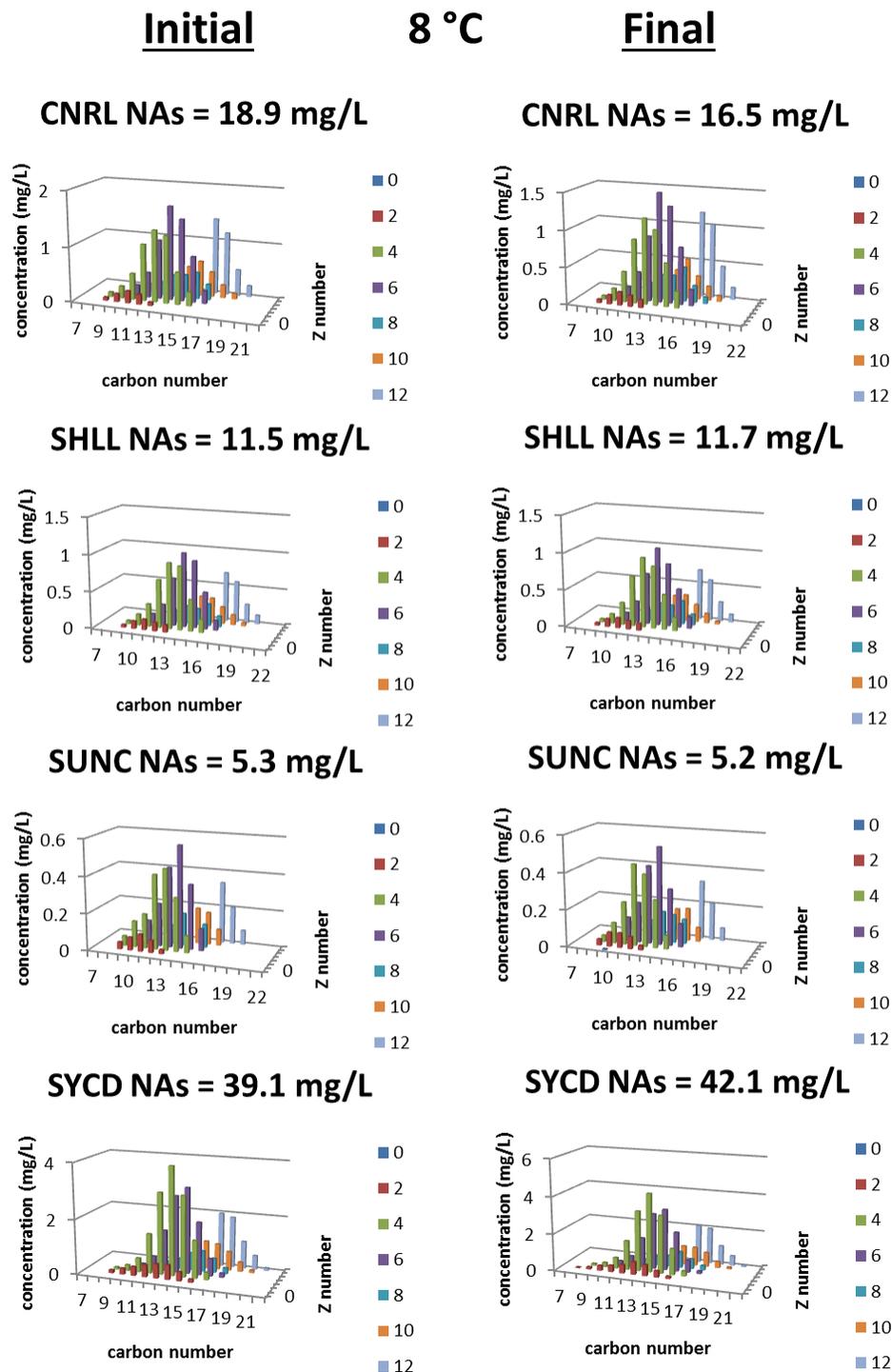


Figure 7. The naphthenic acid concentrations for each OSPW sample (CNRL, SHLL, SUNC, and SYCD) before (initial control) and after (final) treatment at 8 °C. NOTE: treatment samples were pooled for NAs analysis (see section 3.3).

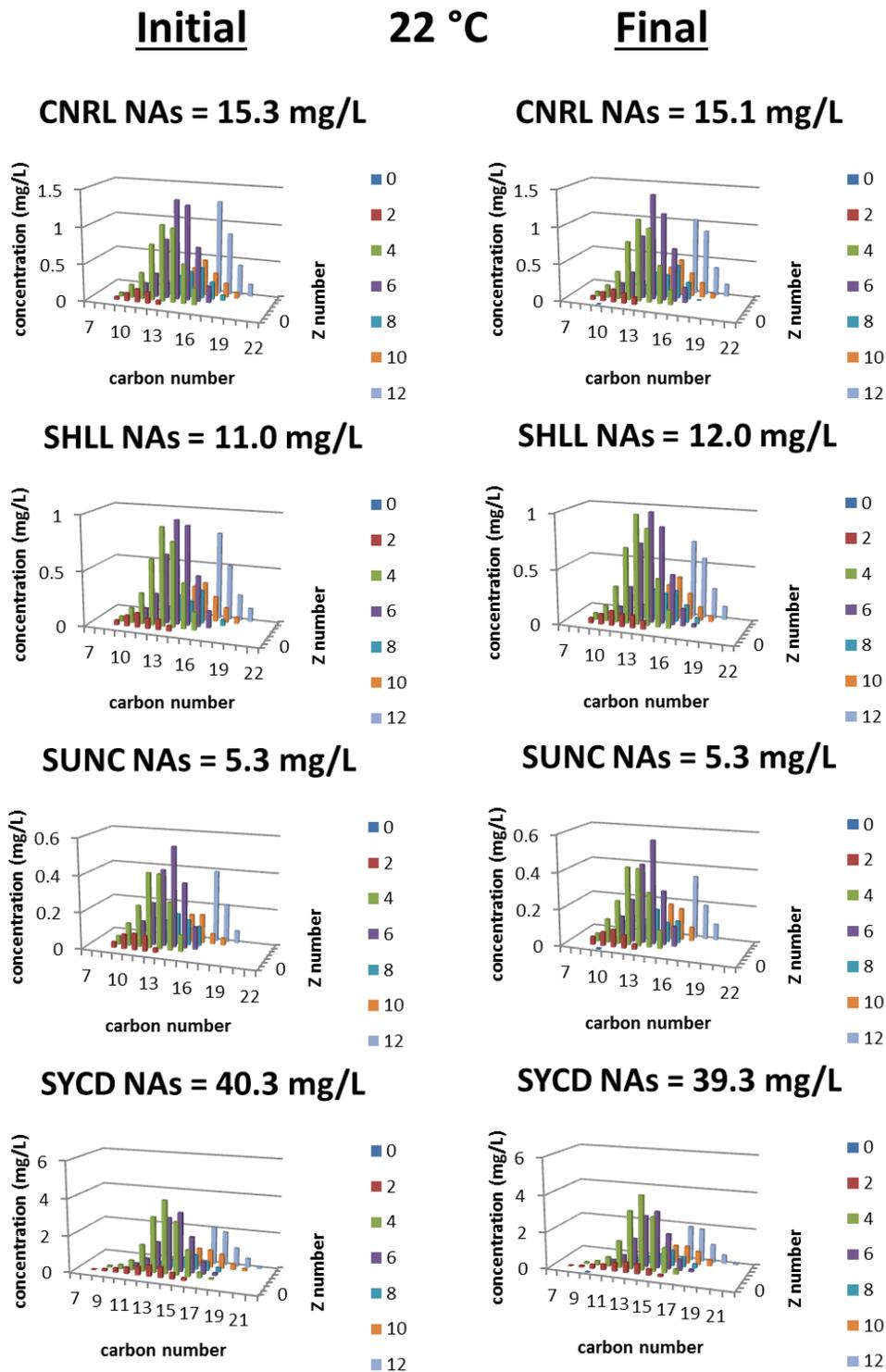


Figure 8. The naphthenic acid concentrations for each OSPW sample (CNRL, SHLL, SUNC, and SYCD) before (initial control) and after (final) treatment at 22 °C. NOTE: treatment samples were pooled for NAs analysis (see section 3.3).

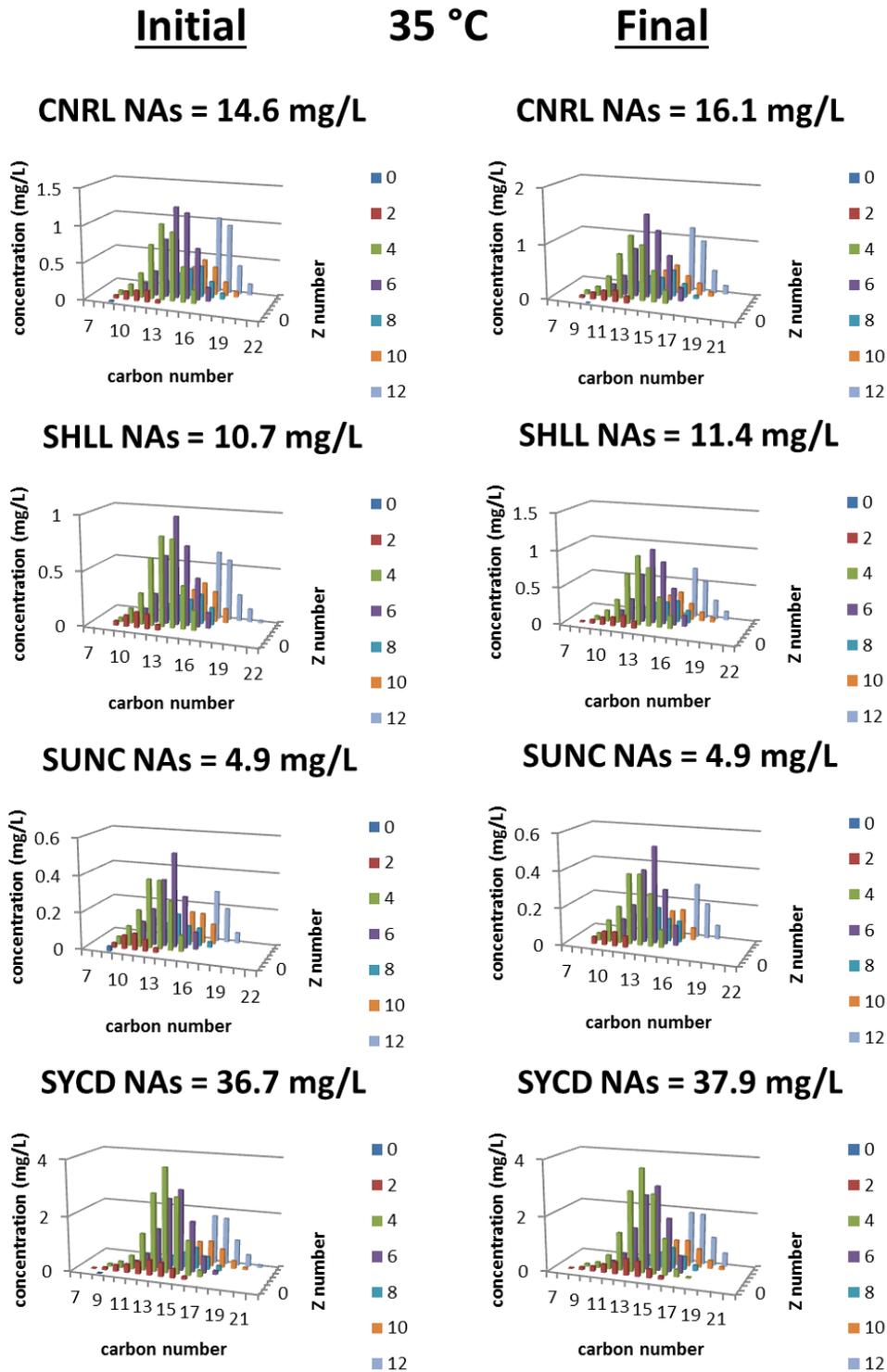


Figure 9. The naphthenic acid concentrations for each OSPW sample (CNRL, SHLL, SUNC, and SYCD) before (initial control) and after (final) treatment at 35 °C. NOTE: treatment samples were pooled for NAs analysis (see section 3.3).

4 CONCLUSIONS AND RECOMMENDATIONS

4.1 Conclusions for Oil Sands Process-Affected Water

- Overall, the OSPW samples had a wide range of toxicity to *V. fischeri* and AEF and NA concentrations. This variability of OSPW in the oil sands region confirms the need for treatment processes to be applicable to a wide range of samples. Alternatively, the treatment processes can be tailored to specific sample sites and/or ponds.
- The toxicity in some samples was higher after treatment than relevant controls. These results were unexpected and need further assessment to elucidate the efficacy of the consortium in impacting the toxicity.
- The BioTiger™ consortium had no impact on reducing AEF and NAs in OSPW at any of the experimental temperatures. Further experiments based on the recommendations set out below should be considered to determine if the consortium may be useful for OSPW treatment.

4.2 Recommendations for Oil Sands Process-Affected Water

- The current experiments can be repeated with extended treatment duration of one week to determine if BioTiger™ can degrade OSPW organics. Although the BioTiger™ was acclimated to the MFT for initial growth, the bacteria may need a longer duration to acclimate to the organics available in the individual OSPW samples.
- A higher initial concentration of BioTiger™ may be considered to potentially improve the degradation ability.
- A labile organic (e.g., acetate) can be added to aid in the synergistic degradation of the more recalcitrant organics found in OSPW. This addition would be in conjunction with extended treatment duration to improve the potential for degradation of OSPW.

5 REFERENCES

Afzal, A., P. Drzewicz, J.W. Martin and M. Gamal El-Din, 2012. Decomposition of cyclohexanoic acid by the UV/H₂O₂ process under various conditions. *Science of the Total Environment* 426: 387-392.

Alberta Environment and Fisheries and Oceans Canada, 2007. Water Management Framework: Instream flow needs and water management system for the lower Athabasca River. Alberta Environment, Edmonton, Alberta and Fisheries and Oceans Canada, Ottawa, Ontario. 37 pp. http://environment.alberta.ca/documents/Athabasca_RWMF_Technical.pdf [Last accessed December 1, 2014].

Alberta Environment and Sustainable Resource Development, 2013. Oil sands environmental management quick facts. Alberta Environment and Sustainable Resource Development, Edmonton, Alberta.

http://www.oilsands.alberta.ca/FactSheets/Quick_Facts_FSht_85x11_Nov_2013_Online.pdf [Last accessed December 8, 2014].

Berry, C.J., S. Story, D.J. Altman, R. Upchurch, W. Whitman, D. Singleton, G. Plaza and R.L. Brigmon, 2006. Biological treatment of petroleum and radiological contaminated soil. IN: Innovative approaches for the remediation of subsurface-contaminated hazardous waste sites: Bridging flask and field scales. Clark, C. and A. Lindner (Editors). Oxford Press. ACS Symposium Series. pp. 87-104.

Brigmon, R.L. and C. Berry, 2009. Biological enhancement of hydrocarbon extraction. US Patent No. 7,472,747 B1.

Brigmon, R.L., S. Story, C. Berry and D. Altman, 2009. Surfactant biocatalyst for remediation of recalcitrant organics and heavy metals. WSR-54 (SRS 02-019). US Patent 7,473,546 B2.

Drzewicz, P., L. Perez-Estrada, A. Alpatova, J.W. Martin and M. Gamal El-Din, 2012. Impact of peroxydisulfate in the presence of zero valent iron on the oxidation of cyclohexanoic acid and naphthenic acids from oil sands process-affected water. *Environmental Science & Technology* 46: 8984-8991.

Energy Resources Conservation Board, 2009. Directive 074: Tailings performance criteria and requirements for oil sands mining schemes. Energy Resources Conservation Board, Calgary, Alberta. 14 pp. <http://www.aer.ca/documents/directives/Directive074.pdf> [Last accessed December 4, 2014].

Fedorak, P.M., D.L. Coy, M.J. Salloum and M.J. Dudas, 2002. Methanogenic potential of tailings samples from oil sands extraction plants. *Canadian Journal of Microbiology* 48: 21-33.

Gamal El-Din, M., H. Fu, N. Wang, P. Chelme-Ayala, L. Perez-Estrada, P. Drzewicz, J.W. Martin, W. Zubot and D.W. Smith, 2011. Naphthenic acids speciation and removal during petroleum-coke adsorption and ozonation of oil sands process-affected water. *Science of the Total Environment* 409: 5119-5125.

Giesy, J.P., J.C. Anderson and S.B. Wiseman, 2010. Alberta oil sands development. *Proceedings of the National Academy of Sciences of the United States of America* 107: 951-952.

Government of Alberta, 2008. Launching Alberta's Energy Future: Provincial energy strategy. Government of Alberta, Edmonton, Alberta. 52 pp. http://www.energy.alberta.ca/Org/pdfs/AB_ProvincialEnergyStrategy.pdf [Last accessed December 1, 2014].

Holowenko, F.M., M.D. MacKinnon and P.M. Fedorak, 2000. Methanogens and sulfate-reducing bacteria in oil sands fine tailings waste. *Canadian Journal of Microbiology* 46: 927-937.

- Klamerth, N., J. Moreira, C. Li, A. Singh, K.N. McPhedran, P. Chelme-Ayala, M. Belosevic and M. Gamal El-Din, 2015. Effect of ozonation on the naphthenic acids speciation and toxicity of pH-dependent organic fractions of oil sands process-affected water. *Science of the Total Environment* 506-507: 66-75.
- Nian, S., P. Chelme-Ayala, N. Klamerth, K.N. McPhedran, S. Md. Islam, L. Perez-Estrada, P. Drzewicz, B. Blunt, M. Reichart, M. Hagen, K. Tierney, M. Belosevic and M. Gamal El-Din, 2014. Advanced analytical mass spectrometric techniques and bioassays to characterize untreated and ozonated oil sands process-affected water. *Environmental Science & Technology* 48: 11090-11099.
- Penner, T.J. and J.M. Foght, 2010. Mature fine tailings from oil sands processing harbour diverse methanogenic communities. *Canadian Journal of Microbiology* 56: 459-470.
- Pereira A.S., M.S. Islam, M. Gamal El-Din and J.W. Martin, 2013. Ozonation degrades all detectable organic compound classes in oil sands process-affected water; an application of high-performance liquid chromatography/obitrap mass spectrometry. *Rapid Communications in Mass Spectrometry* 27: 2317- 2326.
- Sanford, E.C., 1983. Processability of Athabasca oil sand: Interrelationship between oil sand fine solids, process aids, mechanical energy and oil sand age after mining. *Canadian Journal of Chemical Engineering* 61: 554-567.
- Siddique, T., P.M. Fedorak, M.D. Mackinnon and J.M. Foght, 2007. Metabolism of BTEX and naphtha compounds to methane in oil sands tailings. *Environmental Science & Technology* 41: 2350-2356.
- Siddique, T., P. Kuznetsov, A. Kuznetsova, N. Arkell, R. Young, L. Carmen, S. Guigard, E. Underwood and J.M. Foght, 2014. Microbially-accelerated consolidation of oil sands tailings. Pathway I: Changes in porewater chemistry. *Frontiers of Microbiology* 5(106): 1-11.
- Siddique, T., T. Penner, J. Klassen, C. Nesbo and J.M. Foght, 2012. Microbial communities involved in methane production from hydrocarbons in oil sands tailings. *Environmental Science & Technology* 46: 9802-9810.
- Wang N., P. Chelme-Ayala, L. Perez-Estrada, E. Garcia-Garcia, J. Pun, J.W. Martin, M. Belosevic and M. Gamal El-Din, 2013. Impact of ozonation on naphthenic acids speciation and toxicity of oil sands process-affected water to *Vibrio fischeri* and mammalian immune system. *Environmental Science & Technology* 47: 6518-6526.
- Voordouw, G., 2012. Interaction of oil sands tailings particles with polymers and microbial cells: First steps toward reclamation to soil. *Biopolymers* 99: 257-262.
- Zhao, H., J. Long, J.H. Masliyah and Z. Xu, 2006. Effect of divalent cations and surfactants on silica-bitumen interactions. *Industrial Engineering and Chemistry Research* 45: 7482-7490.

6 GLOSSARY

6.1 Terms

Acid-Extractable Fraction (AEF)

The organic compounds that are extracted by dichloromethane at pH 2.5. These compounds include naphthenic acids that are in protonated form and will favour the organic solvent phase.

Bioprocess

A process that uses biological cells and/or cellular components (e.g., soluble membrane products) to mitigate the production of desired products (e.g., degradation of organics).

Microbial Consortium

A group of bacteria used in a bioprocess that have previously exhibited synergistic behaviour making the group efficacy greater than the sum of the individual species.

Surfactant

A substance that tends to reduce the surface tension of a liquid in which it is dissolved (e.g., naphthenic acids).

6.2 Acronyms

AEF	Acid-Extractable Fraction
CNRL	Canadian Natural Resources Limited
FTIR	Fourier Transform Infrared Spectroscopy
GC-FID	Gas Chromatography – Flame Ionization Detector
HPLC/MS	High Performance Liquid Chromatography Mass Spectrometry
HRMS	High Resolution Mass Spectrometry
IC50	Inhibitory Concentration (half maximal)
MFT	Mature Fine Tailings
NAs	Naphthenic Acids
OSPW	Oil Sands Process-Affected Water
OSRIN	Oil Sands Research and Information Network
PAHs	Polycyclic Aromatic Hydrocarbons
SEE	School of Energy and the Environment
SHLL	Shell Canada
SRNL	Savannah River National Laboratory

SUNC

Suncor Energy Inc.

SYCD

Syncrude Canada Ltd.

6.3 Chemicals

CH₄

Methane

CO₂

Carbon dioxide

DCM

Dihloromethane

H₂SO₄

Sulphuric acid

KBr

Potassium bromide

NaOH

Sodium hydroxide

LIST OF OSRIN REPORTS

OSRIN reports are available on the University of Alberta's Education & Research Archive at <http://hdl.handle.net/10402/era.17209>. The Technical Report (TR) series documents results of OSRIN funded projects. The Staff Reports (SR) series represent work done by OSRIN staff.

OSRIN Technical Reports – <http://hdl.handle.net/10402/era.17507>

BGC Engineering Inc., 2010. Oil Sands Tailings Technology Review. OSRIN Report No. TR-1. 136 pp. <http://hdl.handle.net/10402/era.17555>

BGC Engineering Inc., 2010. Review of Reclamation Options for Oil Sands Tailings Substrates. OSRIN Report No. TR-2. 59 pp. <http://hdl.handle.net/10402/era.17547>

Chapman, K.J. and S.B. Das, 2010. Survey of Albertans' Value Drivers Regarding Oil Sands Development and Reclamation. OSRIN Report TR-3. 13 pp. <http://hdl.handle.net/10402/era.17584>

Jones, R.K. and D. Forrest, 2010. Oil Sands Mining Reclamation Challenge Dialogue – Report and Appendices. OSRIN Report No. TR-4. 258 pp. <http://hdl.handle.net/10402/era.19092>

Jones, R.K. and D. Forrest, 2010. Oil Sands Mining Reclamation Challenge Dialogue – Report. OSRIN Report No. TR-4A. 18 pp. <http://hdl.handle.net/10402/era.19091>

James, D.R. and T. Vold, 2010. Establishing a World Class Public Information and Reporting System for Ecosystems in the Oil Sands Region – Report and Appendices. OSRIN Report No. TR-5. 189 pp. <http://hdl.handle.net/10402/era.19093>

James, D.R. and T. Vold, 2010. Establishing a World Class Public Information and Reporting System for Ecosystems in the Oil Sands Region – Report. OSRIN Report No. TR-5A. 31 pp. <http://hdl.handle.net/10402/era.19094>

Lott, E.O. and R.K. Jones, 2010. Review of Four Major Environmental Effects Monitoring Programs in the Oil Sands Region. OSRIN Report No. TR-6. 114 pp. <http://hdl.handle.net/10402/65.20287>

Godwalt, C., P. Kotecha and C. Aumann, 2010. Oil Sands Tailings Management Project. OSRIN Report No. TR-7. 64 pp. <http://hdl.handle.net/10402/era.22536>

Welham, C., 2010. Oil Sands Terrestrial Habitat and Risk Modeling for Disturbance and Reclamation – Phase I Report. OSRIN Report No. TR-8. 109 pp. <http://hdl.handle.net/10402/era.22567>

Schneider, T., 2011. Accounting for Environmental Liabilities under International Financial Reporting Standards. OSRIN Report TR-9. 16 pp. <http://hdl.handle.net/10402/era.22741>

Davies, J. and B. Eaton, 2011. Community Level Physiological Profiling for Monitoring Oil Sands Impacts. OSRIN Report No. TR-10. 44 pp. <http://hdl.handle.net/10402/era.22781>

Hurndall, B.J., N.R. Morgenstern, A. Kupper and J. Sobkowicz, 2011. Report and Recommendations of the Task Force on Tree and Shrub Planting on Active Oil Sands Tailings Dams. OSRIN Report No. TR-11. 15 pp. <http://hdl.handle.net/10402/era.22782>

Gibson, J.J., S.J. Birks, M. Moncur, Y. Yi, K. Tattrie, S. Jasechko, K. Richardson, and P. Eby, 2011. Isotopic and Geochemical Tracers for Fingerprinting Process-Affected Waters in the Oil Sands Industry: A Pilot Study. OSRIN Report No. TR-12. 109 pp. <http://hdl.handle.net/10402/era.23000>

Oil Sands Research and Information Network, 2011. Equivalent Land Capability Workshop Summary Notes. OSRIN Report TR-13. 83 pp. <http://hdl.handle.net/10402/era.23385>

Kindziarski, W., J. Jin and M. Gamal El-Din, 2011. Plain Language Explanation of Human Health Risk Assessment. OSRIN Report TR-14. 37 pp. <http://hdl.handle.net/10402/era.23487>

Welham, C. and B. Seely, 2011. Oil Sands Terrestrial Habitat and Risk Modelling for Disturbance and Reclamation – Phase II Report. OSRIN Report No. TR-15. 93 pp. <http://hdl.handle.net/10402/era.24547>

Morton Sr., M., A. Mullick, J. Nelson and W. Thornton, 2011. Factors to Consider in Estimating Oil Sands Plant Decommissioning Costs. OSRIN Report No. TR-16. 62 pp. <http://hdl.handle.net/10402/era.24630>

Paskey, J. and G. Steward, 2012. The Alberta Oil Sands, Journalists, and Their Sources. OSRIN Report No. TR-17. 33 pp. <http://hdl.handle.net/10402/era.25266>

Cruz-Martinez, L. and J.E.G. Smits, 2012. Potential to Use Animals as Monitors of Ecosystem Health in the Oil Sands Region – July 2013 Update. OSRIN Report No. TR-18. 59 pp. <http://hdl.handle.net/10402/era.25417>

Hashisho, Z., C.C. Small and G. Morshed, 2012. Review of Technologies for the Characterization and Monitoring of VOCs, Reduced Sulphur Compounds and CH₄. OSRIN Report No. TR-19. 93 pp. <http://hdl.handle.net/10402/era.25522>

Kindziarski, W., J. Jin and M. Gamal El-Din, 2012. Review of Health Effects of Naphthenic Acids: Data Gaps and Implications for Understanding Human Health Risk. OSRIN Report No. TR-20. 43 pp. <http://hdl.handle.net/10402/era.26060>

Zhao, B., R. Currie and H. Mian, 2012. Catalogue of Analytical Methods for Naphthenic Acids Related to Oil Sands Operations. OSRIN Report No. TR-21. 65 pp. <http://hdl.handle.net/10402/era.26792>

Oil Sands Research and Information Network and Canadian Environmental Assessment Agency, 2012. Summary of the Oil Sands Groundwater – Surface Water Interactions Workshop. OSRIN Report No. TR-22. 125 pp. <http://hdl.handle.net/10402/era.26831>

Valera, E. and C.B. Powter, 2012. Implications of Changing Environmental Requirements on Oil Sands Royalties. OSRIN Report No. TR-23. 21 pp. <http://hdl.handle.net/10402/era.27344>

Dixon, R., M. Maier, A. Sandilya and T. Schneider, 2012. Qualifying Environmental Trusts as Financial Security for Oil Sands Reclamation Liabilities. OSRIN Report No. TR-24. 32 pp. <http://hdl.handle.net/10402/era.28305>

Creasey, R., 2012. Professional Judgment in Mineable Oil Sands Reclamation Certification: Workshop Summary. OSRIN Report No. TR-25. 52 pp. <http://hdl.handle.net/10402/era.28331>

Alberta Innovates – Technology Futures, 2012. Investigating a Knowledge Exchange Network for the Reclamation Community. OSRIN Report No. TR-26. 42 pp. <http://hdl.handle.net/10402/era.28407>

Dixon, R.J., J. Kenney and A.C. Sandilya, 2012. Audit Protocol for the Mine Financial Security Program. OSRIN Report No. TR-27. 27 pp. <http://hdl.handle.net/10402/era.28514>

Davies, J., B. Eaton and D. Humphries, 2012. Microcosm Evaluation of Community Level Physiological Profiling in Oil Sands Process Affected Water. OSRIN Report No. TR-28. 33 pp. <http://hdl.handle.net/10402/era.29322>

Thibault, B., 2012. Assessing Corporate Certification as Impetus for Accurate Reporting in Self-Reported Financial Estimates Underlying Alberta’s Mine Financial Security Program. OSRIN Report No. TR-29. 37 pp. <http://hdl.handle.net/10402/era.29361>

Pyper, M.P., C.B. Powter and T. Vinge, 2013. Summary of Resiliency of Reclaimed Boreal Forest Landscapes Seminar. OSRIN Report No. TR-30. 131 pp. <http://hdl.handle.net/10402/era.30360>

Pyper, M. and T. Vinge, 2013. A Visual Guide to Handling Woody Materials for Forested Land Reclamation. OSRIN Report No. TR-31. 10 pp. <http://hdl.handle.net/10402/era.30381>

Mian, H., N. Fassina, A. Mukherjee, A. Fair and C.B. Powter, 2013. Summary of 2013 Tailings Technology Development and Commercialization Workshop. OSRIN Report No. TR-32. 69 pp. <http://hdl.handle.net/10402/era.31012>

Howlett, M. and J. Craft, 2013. Application of Federal Legislation to Alberta’s Mineable Oil Sands. OSRIN Report No. TR-33. 94 pp. <http://hdl.handle.net/10402/era.31627>

Welham, C., 2013. Factors Affecting Ecological Resilience of Reclaimed Oil Sands Uplands. OSRIN Report No. TR-34. 44 pp. <http://hdl.handle.net/10402/era.31714>

Naeth, M.A., S.R. Wilkinson, D.D. Mackenzie, H.A. Archibald and C.B. Powter, 2013. Potential of LFH Mineral Soil Mixes for Land Reclamation in Alberta. OSRIN Report No. TR-35. 64 pp. <http://hdl.handle.net/10402/era.31855>

Welham, C. and B. Seely, 2013. Oil Sands Terrestrial Habitat and Risk Modelling for Disturbance and Reclamation: The Impact of Climate Change on Tree Regeneration and Productivity – Phase III Report. OSRIN Report No. TR-36. 65 pp. <http://hdl.handle.net/10402/era.31900>

- Eaton, B., T. Muhly, J. Fisher and S-L. Chai, 2013. Potential Impacts of Beaver on Oil Sands Reclamation Success – an Analysis of Available Literature. OSRIN Report No. TR-37. 65 pp. <http://hdl.handle.net/10402/era.32764>
- Paskey, J., G. Steward and A. Williams, 2013. The Alberta Oil Sands Then and Now: An Investigation of the Economic, Environmental and Social Discourses Across Four Decades. OSRIN Report No. TR-38. 108 pp. <http://hdl.handle.net/10402/era.32845>
- Watson, B.M. and G. Putz, 2013. Preliminary Watershed Hydrology Model for Reclaimed Oil Sands Sites. OSRIN Report No. TR-39. 193 pp. <http://hdl.handle.net/10402/era.34250>
- Birks, S.J., Y. Yi, S. Cho, J.J. Gibson and R. Hazewinkel, 2013. Characterizing the Organic Composition of Snow and Surface Water in the Athabasca Region. OSRIN Report No. TR-40. 62 pp. <http://hdl.handle.net/10402/era.36643>
- De Corby, R.G., 2013. Development of Silicon-Based Optofluidic Sensors for Oil Sands Environmental Monitoring. OSRIN Report No. TR-41. 19 pp. <http://hdl.handle.net/10402/era.36936>
- Iqbal, M., T.K. Purkait, J.G.C. Veinot and G.G. Goss, 2013. Benign-by-Design: Synthesis of Engineered Silicon Nanoparticles and their Application to Oil Sands Water Contaminant Remediation. OSRIN Report No. TR-42. 30 pp. <http://hdl.handle.net/10402/era.37308>
- Oil Sands Research and Information Network, 2013. Future of Shrubs in Oil Sands Reclamation Workshop. OSRIN Report No. TR-43. 71 pp. <http://hdl.handle.net/10402/era.37440>
- Smreciu, A., K. Gould and S. Wood, 2013. Boreal Plant Species for Reclamation of Athabasca Oil Sands Disturbances – Updated December 2014. OSRIN Report No. TR-44. 23 pp. plus appendices. <http://hdl.handle.net/10402/era.37533>
- Pereira, A.S. and J.W. Martin, 2014. On-Line Solid Phase Extraction – HPLC – Orbitrap Mass Spectrometry for Screening and Quantifying Targeted and Non-Targeted Analytes in Oil Sands Process-Affected Water and Natural Waters in the Athabasca Oil Sands Region. OSRIN Report No. TR-45. 33 pp. <http://hdl.handle.net/10402/era.37793>
- Liang, J., F. Tumpa, L.P. Estrada, M. Gamal El-Din and Y. Liu, 2014. Ozone-Assisted Settling of Diluted Oil Sands Mature Fine Tailings: A Mechanistic Study. OSRIN Report No. TR-46. 43 pp. <http://hdl.handle.net/10402/era.38226>
- Rochdi, N., J. Zhang, K. Staenz, X. Yang, D. Rolfson, J. Banting, C. King and R. Doherty, 2014. Monitoring Procedures for Wellsite, In-Situ Oil Sands and Coal Mine Reclamation in Alberta. OSRIN Report No. TR-47. 156 pp. <http://hdl.handle.net/10402/era.38742>
- Taheriazad, L., C. Portillo-Quintero and G.A. Sanchez-Azofeifa, 2014. Application of Wireless Sensor Networks (WSNs) to Oil Sands Environmental Monitoring. OSRIN Report No. TR-48. 51 pp. <http://hdl.handle.net/10402/era.38858>
- Marey, H.S., Z. Hashisho and L. Fu, 2014. Satellite Remote Sensing of Air Quality in the Oil Sands Region. OSRIN Report No. TR-49. 104 pp. <http://hdl.handle.net/10402/era.38882>

- Li, C., A. Singh, N. Klammerth, K. McPhedran, P. Chelme-Ayala, M. Belosevic and M. Gamal El-Din, 2014. Synthesis of Toxicological Behavior of Oil Sands Process-Affected Water Constituents. OSRIN Report No. TR-50. 101 pp. <http://hdl.handle.net/10402/era.39659>
- Jiang, Y. and Y. Liu, 2014. Application of Forward Osmosis Membrane Technology for Oil Sands Process-Affected Water Desalination. OSRIN Report No. TR-51. 27 pp. <http://hdl.handle.net/10402/era.39855>
- Zhu, L., M. Yu, L. Delgado Chávez, A. Ulrich and T. Yu, 2014. Review of Bioreactor Designs Applicable to Oil Sands Process-Affected Water Treatment. OSRIN Report No. TR-52. 39 pp. <http://hdl.handle.net/10402/era.39903>
- Oil Sands Research and Information Network, 2014. Oil Sands Rules, Tools and Capacity: Are we Ready for Upcoming Challenges? OSRIN Report No. TR-53. 120 pp. <http://hdl.handle.net/10402/era.39985>
- Iqbal, M., T.K. Purkait, M. Aghajamali, L. Hadidi, J.G.C. Veinot, G.G. Goss and M. Gamal El-Din, 2014. Hybrid Aerogel SiNP Membranes for Photocatalytic Remediation of Oil Sands Process Water. OSRIN Report No. TR-54. 29 pp. <http://hdl.handle.net/10402/era.40004>
- Schoonmaker, A., J-M. Sobze, E. Fraser, E. Marenholtz, A. Smreciu, C.B. Powter and M. Mckenzie, 2014. Alternative Native Boreal Seed and Plant Delivery Systems for Oil Sands Reclamation. OSRIN Report No. TR-55. 61 pp. <http://hdl.handle.net/10402/era.40099>
- Aguilar, M., E. Glucksman, D. Bass and J.B. Dacks, 2014. Next Generation Sequencing of Protists as a Measure of Microbial Community in Oil Sands Tailings Ponds: Amplicon Versus Metagenomic Approaches. OSRIN Report No. TR-56. 24 pp. <http://hdl.handle.net/10402/era.40100>
- Alessi, D.S., M.S. Alam and M.C. Kohler, 2014. Designer Biochar-Coke Mixtures to Remove Naphthenic Acids from Oil Sands Process-Affected Water (OSPW). OSRIN Report No. TR-57. 38 pp. <http://hdl.handle.net/10402/era.40122>
- Oil Sands Research and Information Network, 2014. Survey of Oil Sands Environmental Management Research and Information Needs. OSRIN Report No. TR-58. 67 pp. <http://hdl.handle.net/10402/era.40128>
- Huang, Q., H. Wang and M.A. Lewis, 2014. Development of a Toxin-Mediated Predator-Prey Model Applicable to Aquatic Environments in the Athabasca Oil Sands Region. OSRIN Report No. TR-59. 59 pp. <http://hdl.handle.net/10402/era.40140>
- Currie, R., S. Bansal, I. Khan and H. Mian, 2014. An Investigation of the Methylene Blue Titration Method for Clay Activity of Oil Sands Samples. OSRIN Report No. TR-60. 50 pp. <http://hdl.handle.net/10402/era.40164>
- Welham, C., 2014. Risk and Uncertainty in Oil Sands Upland Reclamation: Best Management Practices within the Context of Climate Change. OSRIN Report No. TR-61. 26 pp. <http://hdl.handle.net/10402/era.40171>

Mahdavi, H., H. Mian, S. Hepperle and Z. Burkus, 2014. Standard Operating Procedures for Analysis of Naphthenic Acids from Oil Sands Process-Affected Water. OSRIN Report No. TR-62. 67 pp. <http://hdl.handle.net/10402/era.40181>

OSRIN Videos – <http://hdl.handle.net/10402/era.29304>

Rooney Productions, 2012. [Assessment Methods for Oil Sands Reclamation Marshes](#). OSRIN Video No. V-1. 20 minutes. Also available on the [University of Alberta You Tube Channel](#) (recommended approach).

Rooney Productions, 2012. [Assessment Methods for Oil Sands Reclamation Marshes](#). OSRIN Video No. V-1. Nine-part mobile device version. Also available on the University of Alberta You Tube Channel ([link to Part 1](#) - recommended approach).

OSRIN Staff Reports – <http://hdl.handle.net/10402/era.19095>

OSRIN, 2010. Glossary of Terms and Acronyms used in Oil Sands Mining, Processing and Environmental Management – December 2013 Update. OSRIN Report No. SR-1. 123 pp. <http://hdl.handle.net/10402/era.17544>

OSRIN, 2010. OSRIN Writer’s Style Guide – November 2013 Update. OSRIN Report No. SR-2. 29 pp. <http://hdl.handle.net/10402/era.17545>

OSRIN, 2010. OSRIN Annual Report: 2009/2010. OSRIN Report No. SR-3. 27 pp. <http://hdl.handle.net/10402/era.17546>

OSRIN, 2010. Guide to OSRIN Research Grants and Services Agreements - June 2011 Update. OSRIN Report No. SR-4. 21 pp. <http://hdl.handle.net/10402/era.17558>

OSRIN, 2011. Summary of OSRIN Projects – October 2014 Update. OSRIN Report No. SR-5. 113 pp. <http://hdl.handle.net/10402/era.20529>

OSRIN, 2011. OSRIN Annual Report: 2010/11. OSRIN Report No. SR-6. 34 pp. <http://hdl.handle.net/10402/era.23032>

OSRIN, 2011. OSRIN’s Design and Implementation Strategy. OSRIN Report No. SR-7. 10 pp. <http://hdl.handle.net/10402/era.23574>

OSRIN, 2012. OSRIN Annual Report: 2011/12. OSRIN Report No. SR-8. 25 pp. <http://hdl.handle.net/10402/era.26715>

OSRIN, 2013. OSRIN Annual Report: 2012/13. OSRIN Report No. SR-9. 56 pp. <http://hdl.handle.net/10402/era.31211>

OSRIN, 2014. OSRIN Annual Report: 2013/14. OSRIN Report No. SR-10. 66 pp. <http://hdl.handle.net/10402/era.38508>