

1. DRAFT

Sediment Phosphorus Release and Harmful Cyanobacterial Blooms in South Eastern Georgian Bay: Field and diageneteic modeling study

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1. INTRODUCTION

1.1 Background

High phosphorus (P) level in surface water, deep layer oxygen depletion and outbreaks of potentially harmful cyanobacterial blooms (cHABs) have been reported from the embayments of southeastern Georgian Bay. While it is widely accepted that excessive external P loading enhances primary production and can lead to cHABs (e.g. Schindler et al 2012), this link is not evident in Georgian Bay. In some embayments anoxic conditions in deep water close to the sediment water interface (SWI) have been observed over much of the summer, and in fact may persist over the winter as a result of incomplete mixing and the high organic matter inputs from the catchment. Anoxic conditions in deep waters are often accompanied with P release from sediment, as so-called 'internal loading', but some of embayments may not conform to this established model. Recent and limited field evidence suggests no detectable P internal loading in some embayments, which also have a low incidence of cHABs (Powe et al 2013).

Importantly, however, there are no direct measures of internal loading in GB; estimates to date have been inferred from P concentrations in samples collected a distance above the sediment surface, and may not have sampled deep enough to detect P release at the sediment water interface. Furthermore, the mechanism behind the P release from the sediments is not completely understood. In particular, chemical binding in the sediments strongly impact the P release processes. In the proposed study we will monitor these P binding forms and environmental conditions (oxygen, redox potential and pH) at the sediment water interface and develop a dynamic diagenetic model to predict P flux from sediment into water column and sediment characteristics, e.g. dissolved and solid P, organic C and oxygen.

Our work will thus address an important data gap by providing the first direct measures of P release from sediments in a series of embayments representative of different cHAB risk levels. In addition, we will characterize the sediment chemistry in these different embayments, providing insight into sediment biogeochemical mechanisms that govern P release to the water column. Lastly, we will relate these data to physicochemical and biological data collected concurrently in the embayments to investigate how internal loading may be modified by mixing and flushing, and how this is related to water quality and cHAB development. These data will provide input into the modelling efforts that are currently being carried out by us and other groups. We hypothesize, that differences in sediment chemistry/ binding forms and mixing/flushing result in differences in internal P loading and susceptibility to blooms. We propose to build upon previous data from representative embayments along the Georgian Bay (Surgeon, Twelve Mile and Deep Bays), new field measurements of P-binding forms in sediment, high resolution data of redox conditions, pH and nutrients at the sediment-water interface. The proposed modelling and experimental work would be applied to different basins of Georgian Bay, which differ significantly in their morphometry, material loading and sediment redox conditions. In particular, we will focus on the pore water profiles of O2, pH, alkalinity, soluble reactive phosphate (SRP), nitrate and ammonia (NO3, NH4), and major ions and metals (SO4, Ca, Al, Fe, and Mn) in the upper sediment and O2, pH, and redox potential at the sediment-water interface.

A diagenetic modeling simulating different scenarios will improve our understanding of the internal loading in these systems (McCulloch et al., 2013, Gudimov et al., 2013). The dynamic diagenetic modelling framework developed by M Dittrich's group and applied for lakes of different tropic states (Katsev and Dittrich, 2013, Dittrich et al., 2009, 2011, McGulloch et al., 2013, Gudimov et al, 2013) can serve as a tool to investigate the interplay among the sediment processes, to verify concepts, and to predict potential system behaviours. Diagenetic non steady-state modelling of the biogeochemical processes will lead to quantification of internal P loading and prediction of end-of-summer dissolved oxygen in the hypolimnion, P fluxes from sediment and their link to cHABs.

This proposed project will combine advanced computational tools developed by the PI, existing data on water quality, and sediment porewater and solids data collected from different Basins. The existing information from the system in combination with the additional field data offers a promising starting point in modeling (Gudimov et al., 2013).

Our goals are to quantify the release of nutrients (notably phosphorus, P) at the sediment/water interface and evaluate their link with dissolved bottom-water oxygen and cHABs (Watson et al. 2008). The proposed project will lead to quantitative understanding of the mechanisms for phosphorus mobilization in sediments of Georgian Bay and to identification of processes control under a variety of conditions.

We will address the following research objectives:

- quantify P fluxes from sediment, sediment oxygen demand and link these to hypoxia and CHAB
- quantify both seasonal and annual changes in redox conditions, phosphorus release and sediment oxygen demand and link them to chemical and physical processes
- predict internal P release depending on different loading scenarios using a developed, coupled biogeochemical deep-water-sediment reaction-transport model

Several monitoring and research programs have been conducted in the south-eastern Georgian Bay over previous years. However we lack the knowledge required to prioritize the reduction in the nutrient loading, and to design and improve further monitoring and restoration program to improve the water quality and aquatic habitat in Georgian Bay. This project address a key knowledge gap. It provides the first direct measures of internal nutrient loading and sediment chemistry along the Georgian Bay coastline, and how this may be linked with impaired water quality and the risk of algal blooms. This will allow management to evaluate the relative importance of external and internal nutrient inputs in different embayments and how this may affect/delay responses to management/remedial action, thereby developing more effective management in this region - with direct relevance to Lake Simcoe and other water bodies.

The study assists with the goals of the LSGBCUF in the following ways:

Samples collected and data procured by monitoring efforts will support and contribute to the work of Georgian Bay researchers and would assist in the development of a P management strategy

Data collected from analyses of Georgian Bay sediment will be entered into a database that would add value to and assist in the development of the Georgian Bay scientific database/information system

Modelling will help in developing appropriate scenarios regarding the "if", "how", and to"what extent" internal loading will impact P concentrations in lakes and bays of the Georgian Bay.

1.2 Site Description

The geographic area included this project consists of the coastal region between Port Severn and the French River (west of Highway 400/69). The sites that will be included in this study are :

Twelve Mile Bay (N 45° 23' 41.3982", W 80° 13' 25.7916")

Deep Bay (N 45° 5' 0.927", W 79° 56' 44.088")

Honey Harbor: North Bay and South Bay (N 445236, W794730) Tadinac Bay, north of Port Severn, Ontario (N 79.9761, W 45.06318)

Twelve Mile Bay is a long narrow bay with high density of cottages built closer to the water flouting current regulation. High P levels and eutrophication has been observed due to limited water mixing and circulation in the inner portion resulting in an isolated eastern portion. Schiefer et al., 2007 reported the occurrence of anaerobic activity in the sediments releasing nutrients that recurs annually. Moreover with very low flushing rate in this eastern part of the Twelve mile Bay, metals and nutrients tend to remain and accumulate

Tadinac Bay has low shoreline development and represents a 'reference systems, while **North and South Bay** (Honey Harbour), which develop bottom anoxia, are representative of moderately productive systems which have intermittent and small outbreaks of cyanobacteria (e.g. Water Quality Survey of Honey Harbour, 2012 SSEA 2013)



Figure 1 Sampling Locations in South-Eastern Georgian Bay.

Table 1. Physical characteristics of the study sites. * not sampled in 2014

	Mean depth	Max depth	Surface area	Watershed area
Bay	m	m	km ²	km ²
Deep Bay*	10	21	2.85	26.76
Tadinac Bay				
Twelve Mile Bay	12	24	2.98	27.18
North Bay	19	22	2.2	8.9
South Bay	8	11	1.9	n/a

2. METHODS

2.1 Sediment Sampling

Samples were collected from North Bay (NB): 44°53'3.75"N, 79°48'29.96"W), South Bay (SB) : 44°52'37.80"N, 79°47'13.11"W), Twelve Mile Bay (12 Mile Bay): 45°05'0.93"N, 79°56'44.09"W and Tadenac Bay (TB) : 45°03'33.72"N, 79°58'48.52"W (reference site)

using a gravity corer (UWITEC) and Plexiglass core tubes 5.5 cm in diameter and ca. 70 cm in length. Sediment sampling were carried out thwo times (August, 2014; September, 2014; February, 2015) over the course of the study. For each sampling time, seven to eight cores were taken from each station. The sediment cores were sealed and transported to laboratory in a thermo-isolated custom built box for storage. The storage temperature was maintained at 4°C to prevent any atmospheric exchanges. *In situ* pH and oxygen (O₂) and redox potential were measured by microelectrodes on the undisturbed cores upon arrival in the laboratory. Image analyses were performed on sediment cores to differentiate sedimentary layers prior to laboratory analysis (Figure 2). The layers of sediments that have been chosen in this study are shown in Table 2.

Station: South Bay (SB)	Station: 12 Miles Bay (12M)
0-2cm	0-2 cm
2-4 cm	2-3 cm
4-6 cm	3-4 cm
6-7 cm	4-5 cm
7-9 cm	5-6 cm
9-12 cm	6-7 cm
12-14 cm	7-9 cm
14-16 cm	9-11 cm
16-19 cm	11-13 cm
19-25 cm	13-14 cm
	14-16 cm
	16-20 cm
Total: 10 layers	Total: 12 layers

Table 2. Sediment layers which were chosen for the Phosphorus fractionation.

The collected sediment cores were sliced into seven-eight sections to determine the binding forms of P and various geochemical parameters by sediment depth. Sediment samples for geochemical analyses including porosity, dry weight, total organic matter, metals content and dissolved substances were performed in Dittrich's laboratory at University of Toronto Scarborough.



Figure 2: Sediment core image from Station HB, August 13, 2013

2.2 Depth Profiles of O₂, redox and pH

Oxygen, Redox and pH measurements by microelectrode were carried out on the undisturbed sediment core upon arrival to the laboratory. Vertical profiles of O₂ and pH were measured from overlying water into the sediment by lowering microelectrodes that were mount on a manipulator. Oxygen concentrations were measured using a Clark-type O₂ microelectrode (Unisense OX50, Denmark). The electrode was calibrated with air-saturated deionized water (e.g. vigorous bubbling in the Unisense calibration chamber) and zero reading through O₂- free sodium hydrosulfite solution. pH gradient were measured by pH electrodes (Unisense pH-N, Denmark). The electrode was calibrated with commercial pH buffers (pH 4, 7, and 9). A calibration constant of 99% must be achieved prior to microelectrode measurements. Oxygen and pH profiles were obtained from replicates experiments from the same sediment core.

2.3 Phosphorus (P) in Sediment

Phosphorus in the sediment cores is quantified in two approaches: total sediment phosphorus (TP) by persulfate digestion and phosphorus fractionation. Phosphorus fractionation is used to show the relative concentration of different P binding forms in sediment. The P fractionation technique applied in this study was adapted from sequential chemical extraction proposed by Psenner (Psenner, 1988) and modified followed by Rydin (Rydin, 2000). y

Sequential fraction of P in sediment include loosely adsorbed (labile) P (extracted with NH₄Cl, NH₄Cl-TP), redox-sensitive P (extracted with bicarbonate dithionite, BD-TP), P bound to hydrated oxides of aluminum (extracted with NaOH, NaOH-SRP), organic bound P (extracted with NaOH-NRP), carbonates-bound P (apatite-P) (extracted with HCl, HCl-TP) and refractor P (Refractor-P) (Hupfer, 1995). Freeze dried sediment samples (1.0 g wet weight equivalent) were added to 50 mL polypropylene centrifugation tubes and stepwisely extracted with 25 mL of each extractant (Figure 3). The sediment samples were centrifugated (8000 rpm, 10 min) between fractionation steps to collect supernatant. Samples of unfiltered supernatant were subjected to persulfate digestion to determine the total phosphorus in each fraction. The remaining supernatants were filtered using a syringe fitted with 0.45 µm cellulose acetate filter. Sub-samples of the filtrate were used for Soluble Reactive Phosphorus (SRP) by spectrophotometric assay and metals content determination by ICP-AES. For each fraction, the organic bound-P (NRP) concentration was calculated as a difference between total phosphorus and SRP.

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2.4. Porosity, Dry Weight and Total Organic Carbon

Sediment water content at different depths were determined by drying at 105°C for 60 h. Loss of ignition (LOI) was determined by the loss of weight during ignition at 550°C for 2 h. The total carbon has been estimated as LOI/2.5 (%) (Heiri et al., 2001).

2.5 Measurements of Dissolved Substances: Alkalinity, SRP and Metals

Pore waters were extracted by centrifugation at 8000 rpm for 10 minutes from sub-sampled sediment into 50 ml centrifuge tubes from two cores. The supernatant was filtered through a 0.45 μ m cellulose acetate syringe filter. The filtrates were titrated for alkalinity determination by Gran titration method using an autotitrator (Metrohm 905 Titrando, Switzerland). Each titration was carried out for no more than 15 minutes to minimize fluctuation of inorganic carbon equilibrium.

Cores for porewater extractions were sectioned in a glove bag filled with nitrogen to avoid oxidation by atmospheric oxygen (Fig. 3). Sediments were sectioned into 50 mL centrifuge tubes and porewaters were extracted from sediment sections using Rhizon porous polymer micro samplers with 0.1 um membrane (Fig. 3b and 4c; Dickens et al. 2007). The remaining fractions were frozen at -18 °C for further analyses. Porewater samples were stored in separate vials with storage methods appropriate for each analysis: NO3 - and NH4 + samples were frozen at -18 °C, Fe2+ and SRP samples were acidified with 6 N HCl to 1% and stored at 4 °C.

Dissolved phosphate (PO₄ -³) was analyzed through Ascorbic Acid-Molybate spectrophotometric assay (reference will be added). Samples to be analyzed for metals were preserved by acidification using concentrated nitric acid. Dissolved metals (Fe, Al, Ca, Mn, Si, As, Cd, Pb) were analyzed using Agilent Inductive Coupled Plasma Atomic Emission Spectroscopy (ICP-AES).





Figure 3. Measurements of a water column data and sediment pore-water collecting in the glove bags.



Figure 4 Schematic representation of the sequential P extraction method used in the present study.

2.4. Calculations of Diffusive P Flux

Diffusive P fluxes *J_{sed}* across the sediment interface were calculated using Fick's first law of diffusion (Schulz and Zabel, 2000):

$$J_{sed} = -\varphi D_{sed} \frac{\partial C}{\partial x}$$
$$D_{sed} = \frac{D_{sw}}{\theta^2} = \frac{D_{sw}}{1 - \ln(\varphi^2)}$$
$$J_{sed} = -\varphi \frac{D_{sw}}{1 - \ln(\varphi^2)} \frac{\partial C}{\partial x}$$

where Φ is porosity, C is concentration, Z is the sediment depth and Dsed is a diffusion coefficient in the pore water volume of sediment, which can be calculated on the basis of the porosity and the diffusion coefficient in free solutions of sea-water (Boudreau, 1997)

2.5 Modelling

Diagenetic reactions in the model

Primary redox reduction	
$X_{org} + O_2 + H_2 O \to NH_4^+ + HPO_4^- + HCO_3^- + H^+ + HS^-$	
$X_{org} + NO_3^- + H_2O \rightarrow NH_4^+ + HPO_4^- + HCO_3^- + H^+ + HS^- + N_2$	
$X_{Org} + X_{MnO2} + H^+ \rightarrow NH_4^+ + HPO_4^- + HCO_3^- + HS^- + H_2O + Mn^{2+}$	
$X_{Org} + X_{FeOOH} + H^+ \rightarrow NH_4^+ + HPO_4^- + HCO_3^- + HS^- + H_2O + Fe^{2+}$	
	-

Secondary redox reaction

$$\begin{split} NH_4^+ + 2O_2 &\to NO_3^- + 2H^+ + H_2O \\ H_2S + 2O_2 &\to SO_4^{2-} + 2H^+ \end{split}$$

Mineral precipitation-dissolution reactions

 $\begin{array}{c} Ca^{2+} + HCO_3^- \rightarrow X_{CaCO3} + H^+ \\ Fe^{2+} + HCO_3^- \rightarrow X_{FeCO3} + H^+ \end{array}$

 $Mn^{2+} + HCO_3^- \rightarrow X_{MnCO3} + H^+$

Acid base equilibrium conditions

 $\begin{array}{c} H_{2}O \rightarrow H^{+} + OH^{-} \\ H_{2}CO_{3} \rightarrow HCO_{3}^{-} + H^{+} \\ HCO_{3}^{-} \rightarrow CO_{3}^{2-} + H^{+} \\ NH_{4}^{+} \rightarrow NH_{3} + H^{+} \\ H_{2}PO_{4}^{2-} \rightarrow HPO_{4}^{-} + H^{+} \\ H_{2}S \rightarrow HS^{-} + H^{+} \\ HS^{-} \rightarrow S^{2-} + H^{+} \end{array}$

Phosphorus binding forms reactions

$$\begin{split} HPO_4^- &\rightarrow X_{Adsorbed,P} \\ HPO_4^- &\rightarrow X_{Al,P} \\ 3Ca^{2+} + 2HPO_4^- &\rightarrow X_{Apatite,P} + 4H^+ \\ 4Fe^{2+} + 4HPO_4^- + 8HCO_3^- + O_2 &\rightarrow 4X_{BD,P} + 8CO_2 + 4X_{FeOOH} \\ X_{BD,P} &\rightarrow Fe^{2+} + HPO_4^- \end{split}$$

Process rates of reactions in the model were introduced as following

rimary redox reduction rate

$$\begin{split} R1 &= k_{02} \frac{S_{02}}{K_{02} + S_{02}} X_{orgdeg} \\ R2 &= k_{N03} \frac{K_{02}}{K_{02} + S_{02}} \frac{S_{N03}}{K_{N03} + S_{N03}} X_{orgdeg} \\ R3 &= k_{Mn02} \frac{K_{02}}{K_{02} + S_{02}} \frac{K_{N03}}{K_{N03} + S_{N03}} \frac{X_{Mn02}}{K_{Mn02} + X_{Mn02}} X_{orgdeg} \\ R4 &= k_{Fe00H} \frac{K_{02}}{K_{02} + S_{02}} \frac{K_{N03}}{K_{N03} + S_{N03}} \frac{K_{Mn02}}{K_{Mn02} + X_{Mn02}} \frac{X_{Fe00H}}{K_{Fe00H} + X_{Fe00H}} X_{orgdeg} \end{split}$$

econdary redox reaction rate

$$R1 = k_{nitri} \frac{S_{NH4}}{K_{nitri,NH4} + S_{NH4}} \frac{S_{O2}}{K_{nitri,O2} + S_{O2}}$$

 $R2 = k_{oxi,HS} S_{HS} S_{O2}$

on-redox mineral precipitation-dissolution reactions

$$MR1 = \begin{cases} k_{eq,MnC03,prec} \left(\frac{S_{Mn} S_{C03}}{K_{eq,MnC03}} - 1 \right) & \text{if } \frac{S_{Mn} S_{C03}}{K_{eq,MnC03}} > 1 \\ k_{eq,MnC03,diss} \left(\frac{S_{Mn} S_{C03}}{K_{eq,MnC03}} - 1 \right) X_{MnC03} & \text{if } \frac{S_{Mn} S_{C03}}{K_{eq,MnC03}} < 1 \\ MR2 = \begin{cases} k_{eq,CaC03,prec} \left(\frac{S_{Ca} S_{C03}}{K_{eq,CaC03}} - 1 \right) & \text{if } \frac{S_{Ca} S_{C03}}{K_{eq,CaC03}} > 1 \\ k_{eq,CaC03,diss} \left(\frac{S_{Ca} S_{C03}}{K_{eq,CaC03}} - 1 \right) X_{CaC03} & \text{if } \frac{S_{Ca} S_{C03}}{K_{eq,CaC03}} < 1 \\ k_{eq,FeC03,prec} \left(\frac{S_{Fe} S_{C03}}{K_{eq,FeC03}} - 1 \right) & \text{if } \frac{S_{Fe} S_{C03}}{K_{eq,FeC03}} > 1 \\ MR3 = \begin{cases} k_{eq,FeC03,diss} \left(\frac{S_{Fe} S_{C03}}{K_{eq,FeC03}} - 1 \right) & \text{if } \frac{S_{Fe} S_{C03}}{K_{eq,FeC03}} > 1 \\ k_{eq,FeC03,diss} \left(\frac{S_{Fe} S_{C03}}{K_{eq,FeC03}} - 1 \right) X_{FeC03} & \text{if } \frac{S_{Fe} S_{C03}}{K_{eq,FeC03}} > 1 \end{cases}$$

cid dissociation reaction rate

$$R1 = k_{eq,w} \left(1 - \frac{S_H S_{OH}}{K_{eq,w}} \right)$$

$$R2 = k_{eq,CO2,HCO3} \left(S_{CO2} - \frac{S_H S_{HCO3}}{K_{eq,CO2,HCO3}} \right)$$

$$R3 = k_{eq,CO3,HCO3} \left(S_{HCO3} - \frac{S_H S_{CO3}}{K_{eq,CO3,HCO3}} \right)$$

$$R4 = k_{eq,HPO4,H2PO4} \left(S_{H2PO4} - \frac{S_H S_{HPO4}}{K_{eq,HPO4,H2PO4}} \right)$$

$$R5 = k_{eq,NH3,NH4} \left(S_{NH4} - \frac{S_H S_{NH3}}{K_{eq,NH3,NH4}} \right)$$

hosphorous Binding Form Reactions

$$BR1 = k_{Adsorb} \left(Q_{max} \frac{(K_{Adsorb}S_{HPO4}M_{HPO4})}{(1 + (K_{Adsorb}S_{HPO4}M_{HPO4}))} - X_{Adsorb,P} \right)$$

$$BR2 = \frac{\left(\frac{S_{Ca}}{1000}\right)^3 \left(\frac{S_{HPO4}}{1000}\right)^2}{K_{eq,Apatite}*10^{-4pH}}$$

$$BR3 = k_{P_{BD}}S_{O2} S_{Fe}$$

$$BR4 = k_{deg,BD} \frac{K_{O2}}{K_{O2}+S_{O2}} X_{BD,P}$$

$$BR5 = k_{Adsorb,Al} \left(Q_{max,Al} \frac{(K_{Adsorb,Al}S_{HPO4}M_{HPO4})}{\left(1 + (K_{Adsorb,Al}S_{HPO4}M_{HPO4}) \right)} - X_{Al,P} \right)$$

3. RESULTS

3.1 Sediment -Water Interface (SWI)

3.1.1. Dissolved Oxygen, pH and Alkalinity



Figure 5. Sediment-water interface at 12 Miles Bay



Figure 6. Sediment-water interface at South Bay in August 2014



7. Sediment-water interface at South Bay in September 2014



Figure 8. Sediment-water interface at North Bay in September 2014

3.2 Water column parameters



Figure 9. Water column parameters in Tadenac Bay and 12 Miles.

3.3 Organic Carbon, Total P, P Binding Forms and Metals in the Sediment

Depth profiles of P binding forms, porosity, total organic carbon (TOC) and total phosphorus (TP) are shown in Figure 10-14. For all the stations the TOC (%) was ~ 7.5%. The TOC (%) was comparable to studies reported by Milani and Grapentine, 2006; Milani et al., 2000; Thorburn, 2004 (Table 1).



Figure 10. P Binding forms and porosity for South Bay





Figure 11. P Binding forms, total P, organic matter and porosity 12 Miles Bay





Figure 12. P Binding forms, total P, organic matter and porosity for Honey Harbor





Figure 13. P Binding forms, total P, organic matter and porosity for North Bay





Figure 14. P Binding forms, total P, organic matter and porosity for North Bay

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