



Contents lists available at ScienceDirect

Science of the Total Environment

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## Reconstructing a long-term record of microcystins from the analysis of lake sediments

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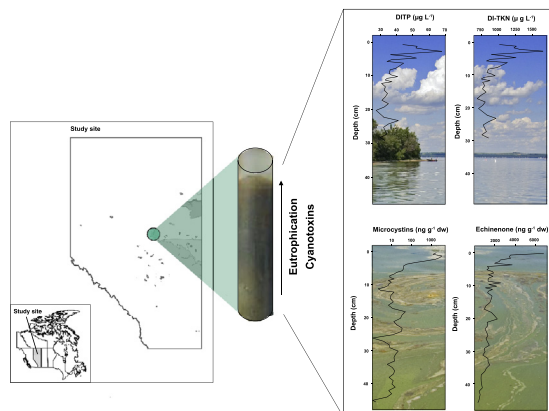
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### HIGHLIGHTS

- Paleolimnology can help track the expansion of harmful cyanotoxins in lakes.
- Microcystin variants were detected in lake sediments prior to land-use changes.
- Sediment microcystins increased rapidly with intensification of modern agriculture.
- The rise in microcystins was greater than that of nutrients and cyanobacteria suggesting a shift towards more toxigenic taxa.

### GRAPHICAL ABSTRACT



### ARTICLE INFO

#### Article history:

Received 26 July 2016

Received in revised form 4 October 2016

Accepted 28 October 2016

Available online xxxx

Editor: D. Barcelo

#### Keywords:

Microcystins

Sediment

Cyanobacteria

Lake history

### ABSTRACT

Based on an analysis of sediment cores from Baptiste Lake (Alberta, Canada), we quantified century-scale trends in cyanobacteria and cyanotoxins, and identified possible drivers of toxigenic cyanobacteria. We measured concentrations of microcystins and pigments preserved in the sediment as proxies of toxigenic cyanobacteria and phytoplankton communities, respectively, while fossil diatom assemblages were used to infer past nutrient concentrations. Microcystins were detected in older sediments (ca. 1800s), pre-dating any significant alteration to the watershed. This demonstrates that toxigenic cyanobacteria may not be a recent phenomenon in eutrophic ecosystems. The dominant variants of microcystin throughout the sediment core were microcystin-LA and microcystin-LR. Other congeners including -LY, -7dmLR, -WR, -LF, -YR, and -LW (-RR was not detected) were mainly found in the upper layers of sediment (post 1980s). Starting in the 1990s, concentrations of microcystins both in the water column and in the sediment record increased in parallel. Total sediment microcystins were strongly correlated with historical nitrogen and phosphorus concentrations inferred from diatom assemblages ( $r = 0.80\text{--}0.81, p < 0.001, n = 22$ ); both nutrients increased over the past two decades coincident with the intensification of agriculture. Microcystins also tracked the rise in cyanobacterial pigments present throughout the core. In contrast, we found no relationship between climate-related variables and sediment microcystin concentrations, although such relationships were detected over the monitoring record with respect to water column concentrations. Overall, the rise in sediment microcystins was much greater than the rise in sediment

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cyanobacteria and diatom inferred nutrient concentrations. Furthermore, we demonstrate that the reconstruction of the microcystin sediment record can provide important insight for the development of realistic lake management goals. Applying this analytical approach to different lakes and regions of the world, where both natural and anthropogenic gradients vary, has the potential to markedly improve our understanding of long-term drivers of cyanotoxin production.

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## 1. Introduction

The rapid proliferation of cyanobacteria in freshwater and marine coastal areas is of particular concern for human and ecosystem health because of the potential for bloom-forming taxa to produce toxins. Monitoring studies and anecdotal evidence suggest that toxigenic blooms are increasing in frequency and intensity due to human-induced eutrophication (Anderson et al., 2002; Heisler et al., 2008). However, cyanotoxins have been periodically detected in oligo- and mesotrophic waters (Zurawell, 2010) and may be triggered by other factors such as climate. Indeed, temperature and water-column stability have also been associated with increased production of certain cyanotoxins, underscoring the interdependence of environmental factors in their proliferation (Paerl and Huisman, 2009; Cerasino and Salmaso, 2012). Although the analysis of historical data sets of cyanotoxins have been insightful, water bodies with a known history of toxic blooms are under-represented and even the best records span only a few decades. As such, paleolimnological studies fill an important gap as they can be used to determine when cyanobacterial proliferations occurred, whether they are increasing over longer time periods, and what might be the underlying causes of such changes (Smol et al., 2001; Riedinger-Whitmore et al., 2005).

Paleolimnological studies increasingly report a change in the phytoplankton community towards cyanobacterial dominance in response to recent eutrophication and climate change in temperate (Cottingham et al., 2000; Eilers et al., 2004) and subalpine to alpine lakes (Jiménez et al., 2015; Milan et al., 2015). Across a broad synthesis of north temperate-subarctic lakes ( $n = 108$  lakes), Taranu et al. (2015) demonstrated that the magnitude of these long-term cyanobacterial trends is significantly related to modern agricultural practices, as well as lake-water nutrient concentrations and average air temperatures recorded throughout the open-water season (i.e. from ice-off in Spring to ice-on in Fall). However, some eutrophic systems have a long history of cyanobacteria that are not directly the result of anthropogenic activities (Hall et al., 1997; Bianchi et al., 2000; Riedinger-Whitmore et al., 2005). Furthermore, whether these proliferations of cyanobacteria are becoming more toxic has only started to be explored in shallow lakes (Pawlik-Skowronska et al., 2010; Efting et al., 2011; Song et al., 2015; Waters, 2016).

Here we analyzed the sediment record for microcystin cyanotoxins and phytoplankton pigments from radiometrically dated lake sediment cores to reconstruct the history of toxic cyanobacteria. Microcystins were chosen because they are the most widely and frequently produced family of cyanotoxins in freshwater as well as the most persistent (Harada and Tsuji, 1998; Zastepa et al., 2014). A few studies have observed the deposition of microcystins in sediments and their persistence in fairly deep sediment layers (Latour et al., 2007; Wormer et al., 2011). Furthermore, a method for the analysis of microcystin congeners has recently been developed for lake sediments (Zastepa et al., 2015). For the present study, we focused on the sediment record from the south basin of Baptiste Lake (Alberta, Canada), which is a deep, eutrophic lake with an anoxic depositional environment that is conducive to the formation of a consistent fossil record (Prepas and Mitchell, 1990). More importantly, Baptiste Lake is one of the few lakes, particularly in North America, to have monitoring data for microcystins extending back to the early 1990s and hence, allows for a comparison of water column

concentrations to the fossil record. The specific objectives of the study were to: (1) determine if sediment microcystins can serve as a proxy for past microcystin concentrations in the lake by assessing their concordance with surface water measurements; (2) test the relationship between sediment microcystins and other paleolimnological (e.g. phosphorus, nitrogen, algal pigments) and climatic (e.g. air temperature) variables to identify drivers of toxigenic cyanobacteria proliferation; and (3) determine if the toxicity of cyanobacteria has changed through time.

## 2. Methods

### 2.1. Study site

Baptiste Lake is located in central Alberta, Canada about 165 km northwest of Edmonton (+54° 45' 23.9754", –113° 33' 38.0016"). It has two distinct basins of similar size connected by a shallow channel (max depth 5.5 m). The deeper and more strongly stratified south basin has an area of 4.7 km<sup>2</sup> and maximum depth of 27.5 m (mean depth 11.9 m). The bottom waters are anoxic during the summer and winter (Prepas and Mitchell, 1990). Baptiste Lake was eutrophic even prior to the first permanent settlements (1880s) in the watershed and has experienced increasing productivity, possibly linked to land-use development and/or a climate-induced change in its mixing regime (meromictic to complete mixing becoming more frequent) (Hickman et al., 1990; Adams et al., 2014). Livestock poisoning cases, suspected to be associated with toxic algae in Baptiste Lake, were reported in the 1950s (O'Donoghue and Wilton, 1951).

### 2.2. Sediment core sampling

Two sediment cores were obtained (+54° 44' 19.7", –113° 33' 7.4") from the south basin in June 2010 using a modified gravity corer (Glew, 1989) at a depth of 26.5 m. Cores were extruded at 0.5 cm intervals to 46 cm, placed in pre-labeled Whirl-Pak® bags, and immediately transported on ice in a dark cooler to be frozen in the laboratory (–20 °C in darkness). Lyophilized sub-samples spanning the length of each core were prepared for dating. The two cores were dated independently in separate laboratories by measuring <sup>210</sup>Pb and <sup>226</sup>Ra activities with a high purity germanium detector in a gamma spectrometer (Core A: DSPec Spectrometer linked to Maestro II Software by Ortec, Tennessee, USA; Core B: Canberra Industries Inc.© Well Detector) according to methods of Appleby (2001). Unsupported <sup>210</sup>Pb was estimated by subtracting supported <sup>210</sup>Pb (measured as <sup>226</sup>Ra activity) from total <sup>210</sup>Pb. <sup>137</sup>Cs resulting from nuclear arms testing circa 1954 (and peaking in 1963 prior to the Nuclear Test Ban Treaty) was used as an independent chronostratigraphic marker. The constant rate of supply, constant initial concentration, and constant flux to constant sedimentation models were evaluated to establish chronology in each core (Appleby and Oldfield, 1978; Blais et al., 1995).

### 2.3. Extraction and analysis of microcystins, phytoplankton pigments, and diatoms

To achieve a detailed chronological resolution, 0.5 cm intervals were extruded as described above and as a result, there was insufficient

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