



Depth-dependent variations of sedimentary dissolved organic matter composition in a eutrophic lake: Implications for lake restoration



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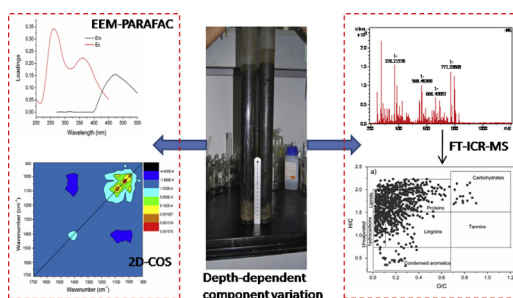
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HIGHLIGHTS

- Vertical change in sediment DOM was quantified by EEM–PARAFAC, 2D–COS, and FT–ICR–MS.
- Humic-like component could be preferentially degraded microbially over fulvic-like component.
- Relative abundance of carbohydrates, lignins and aromatics decreased with depth but not lipids.
- Short-range ordered mineral sorption highly influences the composition and release of DOM in sediment.
- Depth-dependent metal binding heterogeneity highlights the eco-safety of sediment dredging technique.

GRAPHICAL ABSTRACT



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ABSTRACT

Dissolved organic matter (DOM) plays a significant role in regulating nutrients and carbon cycling and the reactivity of trace metals and other contaminants in the environment. However, the environmental/ecological role of sedimentary DOM is highly dependent on organic composition. In this study, fluorescence excitation emission matrix–parallel factor (EEM–PARAFAC) analysis, two dimensional correlation spectroscopy (2D–COS), and ultrahigh resolution electrospray ionization Fourier transform ion cyclotron resonance mass spectrometry (FT–ICR–MS) were applied to investigate the depth-dependent variations of sediment-leached DOM components in a eutrophic lake. Results of EEM–PARAFAC and 2D–COS showed that fluorescent humic-like component was preferentially degraded microbially over fulvic-like component at greater sediment depths, and the relative abundance of non-fluorescent components decreased with increasing depth, leaving the removal rate of carbohydrates > lignins. The predominant sedimentary DOM components derived from FT–ICR–MS were lipids (>50%), followed by lignins (~15%) and proteins (~15%). The relative abundance of carbohydrates, lignins, and condensed aromatics decreased significantly at greater depths, whereas that of lipids increased in general with depth. There existed a significant negative correlation between the short-range ordered (SRO) minerals and the total dissolved organic carbon concentration or the relative contents of lignins and condensed aromatics ($p < 0.05$), suggesting that SRO mineral sorption plays a significant role in controlling the

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composition heterogeneity and releasing of DOM in lake sediments. Higher metal binding potential observed for DOM at deeper sediment depth (e.g., 25–30 cm) supported the ecological safety of sediment dredging technique from the viewpoint of heavy metal de-toxicity.

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1. Introduction

Lake sediments, as a sink for organic matter, nutrients and contaminants, generally play a fundamental role in maintaining the stability of lake ecosystems. Dissolved organic matter (DOM) in sediment is a heterogeneous mixture of complex organic matter, consisting of humic substances (humic acid, fulvic acid, and humin), amino acids, lipids, amino sugars, proteins, and carbohydrates (Zhang et al., 2012). As a small but reactive fraction, DOM directly participates in many physicochemical and biogeochemical processes, such as nutrient and carbon cycling, pollutant sorption, metal complexation, and microbial processes (Guo et al., 2012; Xu et al., 2013a). Therefore, studies on the properties of sedimentary DOM are of great environmental interest.

Reactivity of sediment DOM is highly related to the inherent organic components, which are influenced by many factors including ecological settings, trophic levels, river runoffs, anthropogenic influences, and early diagenesis at different sediment depths (Fu et al., 2006; Chen and Hur, 2015). For example, DOM from macrophyte-dominant sediment generally exhibited a higher pollutant binding potential compared to algae-dominant regions (Xu et al., 2013a). In addition, organic matter enriched with Fe and Al oxides was usually characterized with a slower releasing rate and bioavailability (Kaiser and Zech, 1998). However, for a given lake ecosystem it is expected that depth is the principal factor influencing the ecological role that DOM in sediment pore waters will play. On the other hand, as a widely used technique for lake restoration, sediment dredging removes surface sediment layers, releasing DOM that originally imbedded in deeper sediment layer into sediment-water interface and the overlying water column (Zhong et al., 2010). The potential toxicity of pollutants/contaminants in waters is then affected by dredging depth and their interactions with the DOM released. Thus, investigation on depth-dependent variations of sedimentary DOM is of vital importance both for DOM ecological role analysis and restoration technique evaluation. Previous studies have shown that the organic matter contents change with sediment depth. Using total organic carbon analysis, it was reported that the bulk dissolved organic carbon (DOC) content decreased with increasing depth (Otero et al., 2007). However, detailed information on changes in DOM composition as a function of sediment depth remains poorly understood.

Numerous analytical techniques have been used to characterize the DOM in lake water and sediment, including elemental analysis, attenuated total reflectance Fourier transformed infrared (ATR–FTIR), UV–vis spectroscopy, ¹³C nuclear magnetic resonance, size exclusion chromatography, and fluorescence emission and excitation matrix (EEM) spectroscopy (Fu et al., 2006; Hur et al., 2009; Guo et al., 2012; Chen and Hur, 2015; Zhou and Guo, 2015). In addition, using a multi-way decomposition method, parallel factor (PARAFAC) analysis, some chemically independent but spectrally overlapped fluorophores can be mathematically separated into several components, which can obviously enhance spectral resolution and extracted more information that imbedded in original EEM spectra (Xu et al., 2013b; Chen and Hur, 2015). Moreover, two dimensional correlation spectroscopy (2D–COS) has recently been used to obtain the perturbation-induced dynamics changes of one-dimensional spectra (e.g., infrared and fluorescence) of DOM

samples. The perturbation can be any external environmental variables, such as time (Noda, 2015), temperature (Xu et al., 2013c), UV irradiation (Hur et al., 2011; Xu and Jiang, 2013), metal concentration (Yu et al., 2012; Chen et al., 2015), and salinity (Hussain et al., 2010). Although these elemental and spectral techniques are useful in improving the understanding of the chemical nature of sediment DOM, our knowledge so far is still limited to bulk characteristics because they do not directly identify the presence of specific compounds within the DOM pool.

Ultrahigh resolution electrospray ionization Fourier transform ion cyclotron resonance mass spectrometry (FT–ICR–MS), on the other hand, can be used to elucidate specific compounds within the bulk DOM at the molecular level. The superior capabilities of FT–ICR–MS typically allow the detection of thousands of individual organic compounds and calculation of their elemental formulas based on the exact molecular masses of constituent atoms (Zhang et al., 2012; Ohno et al., 2014). Indeed, FT–ICR–MS has been utilized to characterize DOM components in a range of diverse environments, including freshwater (Hockaday et al., 2009; Zhang et al., 2012, 2014), seawater (Koch et al., 2008), soil (Ohno et al., 2014), and water treatment systems (Cortés-Francisco et al., 2014; Gonsior et al., 2014). However, to the best of our knowledge, no studies have yet applied this technique to explore the depth profiles of organic composition in lake sediment.

Similar to soil DOM, sedimentary DOM is expected to encounter both biotic (e.g., microbial degradation) and abiotic (e.g., reactive mineral sorption) processes (Kramer et al., 2012; Wang et al., 2007). While variations in DOM composition resulted from biotic processes have been widely recognized (Dauwe and Middelburg, 1998), DOM changes induced by physicochemical processes remain less known, although it has been shown to play an important role in component assignment in soil environment (Kramer et al., 2012).

The objectives of this study were therefore (1) to study the depth-dependent heterogeneity of sedimentary DOM composition using fluorescence, infrared, and mass spectra analytical techniques, (2) to explore the contribution, especially the abiotic reaction, that influences depth profiles of organic components in eutrophic lake sediment, and (3) to assess environmental implications of this component heterogeneity for lake restoration technique evaluation. For this purpose, sediment samples were collected in a eutrophic lake for measurements of sediment properties using traditional physicochemical analysis, and sedimentary DOM composition using fluorescence (EEM–PARAFAC), infrared (2D ATR–FTIR COS) and mass spectra (FT–ICR–MS) techniques. In addition, statistics were used to evaluate the correlation between organic components and reactive mineral contents, and spectral titration technique was used to explore the pollutant binding properties of sediment DOM as a function of depth. Our hypothesis is that the ecological role of DOM and environmental behavior of pollutants are heterogeneous in lake sediments and highly regulated by depth-dependent DOM composition.

2. Materials and methods

2.1. Study site and sample analysis

The sampling site is located at an algae-dominant eutrophic

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