



Contents lists available at ScienceDirect

## Soil Biology &amp; Biochemistry

journal homepage: [www.elsevier.com/locate/soilbio](http://www.elsevier.com/locate/soilbio)

# Methane suppression by iron and humic acids in soils of the Arctic Coastal Plain



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## ARTICLE INFO

## Article history:

Received 27 June 2014

Received in revised form

23 January 2015

Accepted 24 January 2015

Available online 13 February 2015

## Keywords:

Arctic peatlands

Methane

Iron

Humic acids

Greenhouse gas emissions

Carbon–climate feedbacks

## ABSTRACT

Methane–climate interactions are reasonably well understood; the biogeochemical controls on net methane fluxes are less so. Within anoxic soils, alternative electron acceptors such as iron and humic substances influence microbial metabolic function, and thus affect the amount of carbon lost as methane (CH<sub>4</sub>). We present three years of data from wet sedge tundra landscapes near Barrow, Alaska that show an inverse relationship between dissolved iron and CH<sub>4</sub> concentrations. We found increasing organic layer thickness related to increases in active layer organic matter content, and decreases in both bulk density and extractable iron. Organic layer depth was also a good proxy for carbon dioxide (CO<sub>2</sub>) and CH<sub>4</sub> dynamics, with increasing organic layer depths relating to lower dissolved iron, higher amounts of dissolved CH<sub>4</sub>, and lower CO<sub>2</sub>:CH<sub>4</sub> ratios in the upper active layer. Net CH<sub>4</sub> fluxes were also significantly suppressed following the experimental addition of iron and humic acids. Iron and humic acid treatment effects were indistinguishable for CH<sub>4</sub> net flux; in contrast, post-treatment CH<sub>4</sub> fluxes were an average of 0.74-fold the control treatment flux rates. These results suggest that *in-situ* CH<sub>4</sub> production is tied to alternative electron acceptor availability, and that organic layer thickness is a good predictor of biogeochemical controls on CH<sub>4</sub> fluxes in wet-sedge Arctic Alaskan tundra.

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## 1. Introduction

Northern permafrost soils contain as much as half of the world's belowground organic carbon (Tarnocai et al., 2009), a large proportion of which is held in cold, anoxic wetlands. Temperatures are increasing disproportionately in northern latitudes (Serreze and Barry, 2011), and permafrost thaw is creating an ever-expanding pool of soil carbon vulnerable to microbial decomposition and loss as carbon dioxide (CO<sub>2</sub>) and methane (CH<sub>4</sub>) (Schuur et al., 2008). As thaw depths increase, underlying mineral layers will

contribute additional carbon, nutrients and minerals to the active soil layer, potentially altering microbial decomposition rates. Arctic tundra ecosystems are currently a net source of CH<sub>4</sub> to the atmosphere (Schuur et al., 2008; McGuire et al., 2012); increases in CH<sub>4</sub> release will positively influence climate feedbacks (Serreze and Barry, 2011). Despite recent work regarding the relationships between carbon-cycling and soil minerals, the biogeochemical controls regulating CH<sub>4</sub> fluxes from permafrost-affected soils remains enigmatic (Heitmann et al., 2007; Keller and Bridgman, 2007; Keller et al., 2009; Lipson et al., 2010; Blodau and Deppe, 2012; Bridgman et al., 2013; Friedman et al., 2013; Keller and Takagi, 2013; Lipson et al., 2013a, 2013b).

Within anoxic subsurface systems, the differential potential free energy yields of terminal electron acceptor (henceforth, we use 'acceptors') reactions create a thermodynamic ranking of microbial respiration redox reactions. In addition to the classic inorganic acceptors, organic acceptors (i.e. humic substances) are gaining recognition as redox-reactive species within soils, especially in northern wetlands where they are in high abundance

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(Heitmann et al., 2007; Blodau and Deppe, 2012; Bridgham et al., 2013). The reduction of humic substances is thought to be thermodynamically favorable to CH<sub>4</sub> production (Cervantes et al., 2000; Blodau and Deppe, 2012; Friedman et al., 2013; Keller and Takagi, 2013), and also functions as an electron shuttle with other redox pathways, such as iron(Fe)-reduction (Lovley and Woodward, 1996). CH<sub>4</sub>-producing microbes are at the bottom of the thermodynamic hierarchy, and will typically be suppressed by alternate microbial respiration processes in the presence of alternative acceptors. Given the importance of understanding CH<sub>4</sub> production for predicting climate feedbacks (Schuur et al., 2013), the interaction between organic and inorganic acceptors and CH<sub>4</sub> deserves more attention within carbon- and acceptor-rich Arctic tundra soils.

A model system for investigating the links between CH<sub>4</sub> and organic and inorganic acceptors is Barrow, Alaska, which boasts carbon- and mineral-rich permafrost-affected soils typical of the Alaskan Arctic Coastal Plain. This landscape is dominated by a time series, or chronosequence, of drained thaw lake basins (hereafter referred to simply as 'basins'), which allows comparison of ecosystem function across stages of soil development and vegetation succession (Hinkel et al., 2003; Bockheim et al., 2004; Sturtevant and Oechel, 2013; Lipson et al., 2013b). The basins are categorized by time since the thermokarst lake drained and began re-vegetating. This is important because the short growing seasons and cold, wet conditions in northern systems cause decomposition to occur more slowly than primary production, favoring the sequestration of soil carbon (Callaghan et al., 2004). Thus, older basins have gone through more cycles of organic matter accretion, resulting in thicker organic soil layers. The thickening of the organic layer effectively creates a heterogeneous balance of organic and mineral soil layers within the active layer across the landscape (Hinkel et al., 2003). Previous work has shown that these soils are naturally Fe-rich, with Fe-reduction contributing significantly to anaerobic soil respiration in all basin ages (Lipson et al., 2010, 2013b). The natural variation in the soil mineral component provides a mechanism to investigate mineral-CH<sub>4</sub> interactions on a landscape scale.

The Barrow basin system is also an ideal model system because fewer alternative electron acceptors are present; observable levels of nitrate, sulfate and manganese are relatively low or absent in porewater surveys of the system (Lipson et al., 2010), and the water table remains near the soil surface throughout most growing seasons (Liljedahl, 2011). Barrow has carbon-, humic substances- and Fe-rich soils that vary along the basin chronosequence, making it an ideal site to study the interactions between methanogenic and the Fe and humic substances reduction respiration pathways. Previous work from Barrow has shown net gas fluxes to have high CO<sub>2</sub>:CH<sub>4</sub> ratios (>500) despite prevailing anoxic conditions, suggesting CH<sub>4</sub> suppression by alternative respiratory activities (Lipson et al., 2012; Friedman et al., 2013). Our two main research questions were: 1) how do Fe and humic substances affect methane production and net release, and 2) are there landscape-scale predictors of Fe-CH<sub>4</sub> dynamics? We hypothesized that increasing concentrations of Fe and/or humic substances in the soil would correspond to decreasing methane production and lower net releases. For the landscape scale, we hypothesized that areas with thicker organic layers would have more separation from the underlying mineral layer, limiting access to a mineral source, and thus have lower Fe contents and correspondingly higher methane levels. We examined landscape Fe-CH<sub>4</sub> dynamics using two years of observational data representing both spatial and temporal variability. Observational data were complemented by an *in-situ* experiment to test the direct relationship between net CH<sub>4</sub> fluxes and the dissimilatory reduction of Fe and humic substances.

## 2. Methods

### 2.1. Site description

Observational data were collected from a total of 14 drained thaw lake basins in the area surrounding Barrow, Alaska over two growing seasons in 2010 and 2011. All basins were located within the Barrow Environmental Observatory research reserve (71.2963 N, 156.5891 W), an area characteristic of the Alaskan North Slope acidic wet sedge tundra (Zona et al., 2010). The landscape is underlain by deep continuous permafrost with seasonal active layer thicknesses of 30–90 cm with a 36 cm mean, with an average summer air temperature of 3.3 °C (June to August, 1960–present). Basins sampled were categorized by years since draining: young (0–50 years), medium (50–300 years), old (300–2000 years), and ancient (2000–5500 years) (Hinkel et al., 2003), and included low-centered, and flat polygons. Hydrologically, these basins experience inundated conditions for about half of the growing season, and the water table remains within 5 cm of the soil surface during all but extreme droughts (Olivas et al., 2010a; Liljedahl, 2011; Lipson et al., 2012).

### 2.2. Soil porewater

Transects of approximately 70 m of tundra matting were established within each basin. Ten soil porewater samplers (Rhizon, type MOM for metal studies, Sunvalley Solutions, Florida) were installed at a depth of 0–10 cm randomly along each transect; porewater from the 0–10 cm depth was measured because dissolved gas concentrations in the upper soils are most reflective of the net atmospheric flux potential through either diffusive or wind-driven fluxes. In 2010, we sampled soil porewater every 1–2 weeks from June to September from 8 basins, with 2 representatives from each of the four basin age classes. In 2011, we increased spatial replication to include 6 additional basins, for a total of 14 basins. Soil porewater samples were collected in additive-free vacutainers (Becton Dickinson) and stored in dark refrigeration.

Dissolved gas samples were prepared by equilibrating 1 mL of porewater with an N<sub>2</sub> headspace within an airtight 25-mL glass serum vial fitted with gas-impermeable butyl rubber stoppers (Geo-Microbial Technologies, Inc.). Equilibration units were gently shaken for 12 h, then left undisturbed in a dark cabinet for 12 h. Headspace samples were analyzed for CO<sub>2</sub> and CH<sub>4</sub> concentrations on a gas chromatograph equipped with TCD and FID detectors. Samples for Fe analysis were collected into air-free vials, acidified to pH < 2.0 with 1 M HCl, wrapped in wax and stored in Bitran airtight zip-top bags in dark refrigeration until analysis within 2 weeks of collection. Total Fe and Fe(II) were quantified from acidified samples using a 1,10-phenanthroline colorimetric method (Analytical Methods Committee, 1978; Lipson et al., 2010); Fe(III) content was calculated by difference.

### 2.3. Soil sampling

Frozen 30 cm-length 3 cm-diameter soil cores were extracted using a handheld power drill fitted with coring drill bits and extenders in early June 2010 and 2011, 5 cm sections of which were immediately immersed in pre-weighed bottles, resulting in approximately 15 g wet soil weight in 50 mL 2 M HCl, extracted overnight. Solids were removed prior to analysis by centrifuging 1 mL aliquots. Fe analysis was completed on all samples using the 1,10-phenanthroline method, as described above, with content calculations adjusted to include soil water. Multiple 7 cm-diameter deep cores (40–80 cm) were collected from all basins in both 2010 and 2011 with a SIPRE core drill bit and small gas-powered engine.

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