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Surface Soil Organic Matter Qualities of Three Distinct Canadian Arctic Sites

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Abstract

Cryosolic soils store large amounts of carbon (C) because soil organic matter (SOM) decomposition is slower than plant growth. The response of arctic SOM to climate change is likely to depend not only on temperature, but also upon complex interactions between soil properties and SOM chemistry. We hypothesized that organic surface soils (>17% carbon) have more labile SOM than mineral surface soils (<17% carbon). Furthermore, we hypothesized that high arctic soils have more labile SOM than soils from the Low Arctic and subarctic. This study was conducted in 3 arctic ecosystems: subarctic (Churchill, Manitoba; $n = 138$), Low Arctic (Daring Lake, Northwest Territories; $n = 60$), and High Arctic (Truelove Lowlands, Nunavut; $n = 54$). The 0–10 cm depth of several different Cryosolic soils was sampled. The results from density fractionation and solid-state ^{13}C cross polarization and magic angle spinning (CPMAS) nuclear magnetic resonance (NMR) spectroscopy showed that organic surface soils contained relatively more labile C than mineral surface soils. Organic soils contained about 13% more *O*-Alkyl-C and 30% less Aromatic-C than mineral soils. Furthermore, for Churchill, Daring Lake, and Truelove organic soils, 53, 73, and 20% of the C was included in the light fraction of SOM [LF (LF < 1.55 g mL $^{-1}$)], whereas 24, 19, and 14% of the C was included in the LF of mineral soils, respectively. Organic surface soils of subarctic and low arctic sites contained relatively more labile C than the high arctic site. Results showed that the subarctic and low arctic sites store about 15% more *O*-alkyl-C and 35% less Aromatic-C than high arctic organic soils ($P < 0.001$).

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Introduction

Cryosolic soils, the dominant soil in the Arctic, store large amounts of carbon (C) because soil organic matter (SOM) decomposition has been lower than input by plants over centuries to millennia (Weintraub and Schimel, 2005). These permafrost-affected soils store approximately 25% of the world's organic C, which represents 61% of the C in all the soils of North America (Tarnocai et al., 2008). About 20% of the C is stored above the permafrost table in the organic enriched surface horizon (Ping et al., 2008). Future environmental changes such as temperature increases and precipitation changes (Huntington et al., 2005; Kattsov et al., 2005) will determine whether arctic soils will continue to accumulate SOM (i.e., net sink), or instead become a source of greenhouse gases (GHG) via SOM decomposition (i.e., net source).

The response of arctic soils to climate change is likely to depend not only on temperature increase and water table controls, but also upon complex interactions between soil properties and SOM chemistry (Shaver et al., 2006). Soil organic matter includes a wide range of organic materials from labile to recalcitrant components which slowly accumulate over thousands of years (Trumbore, 1993; Paul et al., 2001). In soil, SOM may be divided in two phases: (1) soluble SOM known as water-extractable organic matter (WEOM), and (2) solid SOM. Both phases may be further analyzed using other characterization techniques (e.g., solubility, molecular size, density, and spectroscopy).

Water-extractable organic matter is defined as the soil fraction (<0.45 μm) included in soil water. Although WEOM concentra-

tions in the soil are generally low, the mobility and lability of this fraction increase its importance in nutrient cycling (Chantigny et al., 2008). For example, WEOM contributes to soil acidity, pollutant toxicity, nutrient mobility and availability, and provides energy for heterotrophic soil microbes (Zsolnay, 1996; Moore, 1998; Chantigny, 2003). Although portions of WEOM may be considered as recalcitrant (Qualls, 2005; Embacher et al., 2007), WEOM generally interacts with soil microbes and is susceptible to rapid incorporation into soil C and N cycling processes (Chantigny, 2003; Zsolnay, 2003), especially when WEOM is extracted from soil surfaces that are rich in fresh plant residues (Kalbitz et al., 2003).

Solid SOM is defined as the fraction of the soil (<2 mm) that includes a wide range of compounds such as plant, animal, and microbial residues (cells, tissues, and metabolites) at various stages of transformation. Physical fractionation techniques separate SOM from mineral soil components on the basis of density and/or size. Separation by density—using heavy liquids—extracts or separates SOM particles that are not bound to mineral soil particles (Six et al., 2002; Gregorich et al., 2006). This fraction is often referred to as physically uncomplexed or unprotected SOM. Alternatively, separation by size—a combination of heavy liquids, dry, and wet sieving techniques—separates SOM that is bound to different soil particles and sizes (Six et al., 2000). This fraction is often referred to as particulate organic matter (POM). Physically unprotected SOM is assumed to play a greater role than POM in rapid N and C cycling processes because physically unprotected SOM is more accessible to soil microorganisms for degradation and decomposi-

tion than the physically protected POM (Gregorich et al., 2006; Lützow et al., 2008; Marschner et al., 2008). Physically unprotected SOM, which includes fresh and partly-decomposed residues of SOM, is separated from soil using the density fractionation technique (Swift, 1996; Six et al., 2002; Gregorich et al., 2006). Finally, solid-state ^{13}C cross polarization and magic angle spinning (CPMAS) nuclear magnetic resonance (NMR) spectroscopy is a non-destructive spectroscopy technique widely used to characterize solid SOM from different ecosystems at various stages of decomposition (Skjemstad et al., 1997; Preston, 2001; Simpson and Preston, 2008). The solid-state ^{13}C CPMAS NMR technique provides a large spectrum of the entire SOM qualities (Schnitzer, 1991; Dai et al., 2001; Dria et al., 2002; Simpson and Preston, 2008), which is suitable when very little is known about SOM. Several functional C groups of SOM are identified by solid-state ^{13}C NMR spectroscopy. Paraffinic Alkyl-C is present in plant polymers and lipids (e.g., cutin and suberin); *O*-alkyl-C (e.g., plant polysaccharides) is labile substrate to a large number of fungi and bacteria; Aromatic-C are molecules mostly derived from lignin decomposition; and carbonyl-C structures also originate from lignin transformation (Kögel-Knabner, 2002). The *O*-alkyl-C group generally is dominated by signals from celluloses and other polysaccharides (e.g., carbohydrates-C), compounds readily decomposed by soil microbes. The Alkyl-C, Aromatic-C, and Carbonyl-C groups tend to decompose slowly (Sollins et al., 1996; Skjemstad et al., 1997; Lützow et al., 2006).

In arctic soils, SOM affects many processes related to soil nutrient cycling. For example, Grogan et al. (2001) showed that soil respiration derived from fresh litter residues was the principal source of CO_2 efflux. More recently, Buckeridge et al. (2010) found that soils with the greatest quantity and highest lability of SOM had both the most rapid N cycling (high rates of gross mineralization and negative net mineralization, indicating rapid production and consumption) and the tallest vegetation (i.e., tall shrubs). Furthermore, Ping et al. (1998) indicated that high carbohydrate concentrations found in Alaskan arctic soils (i.e., 2 to 3 times greater than southern soils) were relatively easily decomposed and were the ideal substrates for CH_4 production. Therefore, the most labile compounds of SOM are dominant controls on soil nutrient processes such as GHG production and N mineralization.

In the Arctic, surface soil (i.e., upper soil horizons) properties and conditions are especially important because this is where biological processes, including in the rhizosphere, are most active and therefore exert a strong interaction with the vegetation patterns across the landscape (Michaelson et al., 2008). Differentiating between mineral and organic soil horizons is crucial for assessing the fate of surface soils because the relative importance of the various chemical, biological, and physical processes varies between mineral and organic surface soils (Uhlir et al., 2007; Nowinski et al., 2010). For example, Kramer et al. (2004) found that organic surface horizons in Alaska contained relatively more labile materials (e.g., high *O*-Alkyl-C content) than mineral horizons. An *in situ* warming experiment in Antarctica showed that warming increases SOM C and N contents in organic soil horizons to a greater extent than in mineral soil horizons (Day et al., 2008). Furthermore, the reduced vegetation cover on mineral surface soils ensures a high

heat flux and a relatively deep active layer, whereas thick organic surface soils insulate the soil, creating a shallower active layer. Nevertheless, relatively little is known about SOM qualities nor how qualities vary across the Arctic.

We hypothesize that organic surface soils have more labile SOM than mineral surface soils. Furthermore, because the rates of SOM decomposition generally decrease at lower temperatures, we hypothesize that high arctic soils have more labile SOM than soils from the Low Arctic and subarctic. This pattern will, however, be modulated by the influence of litter quality (Freschet et al., 2012).

Material and Methods

SAMPLING LOCATIONS

This study was conducted in three distinct arctic sites: subarctic (Churchill, Manitoba), Low Arctic (Daring Lake, Northwest Territories), and High Arctic (Truelove, Nunavut) (Fig. 1). Daring Lake and Truelove were sampled in 2008, and Churchill was sampled in 2009. To capture within-site variations, the sampling locations were distributed throughout the landscape. Furthermore, vegetation found above sampling locations ranged from lichens and bryophytes to shrubs for all ecosystems. All three ecosystems were sampled near the end of their growing season, from 2 to 3 weeks before plant senescence.

Subarctic: Churchill

The Churchill site is located in Manitoba, Canada ($58^\circ45'\text{N}$, $93^\circ51'\text{W}$) in the subzone E arctic bioclimate (Walker et al., 2005). All sites sampled for this study were located within the tundra vegetation zone between 1 and 5 km from the shores of Hudson Bay. The Precambrian Shield, which underlies the entire coastal region, was buried by the younger Ordovician and Silurian limestones and dolomites that were reworked and deposited during the most recent glaciations (8000 B.P.). The area is characterized by raised beaches formed during the regression of the postglacial Tyrrell Sea and by isostatic rebound (Dredge, 1992). On boggy wetlands and where winter temperatures are harsh and snow cover is thin, hummocks have developed. The Churchill climate is classified as arctic continental with a mean temperature of -7.5°C and a mean annual precipitation of 412 mm (Lafleur et al., 2001). Between 1996 and 2006, a permanent weather station installed about 8 km west of our sampling area ($58^\circ44'\text{N}$, $94^\circ03'\text{W}$) measured a mean annual temperature of $-5.8 \pm 1.6^\circ\text{C}$ (mean \pm standard deviation) and a mean annual precipitation of 501.2 ± 89.1 mm (Environment Canada, 2011). The growing season typically occurs from early June to late August with an average daily maximum of 9.7°C (Lafleur et al., 2001). The region is underlain by continuous permafrost of about 80 m thickness (Dredge, 1992). Static and Turbic Cryosols (Soil Classification Working Group, 1998) were sampled across the range of local parent materials, including fluvial and marine. Lichen species, as well as *Dryas integrifolia*, were the more dominant vegetation types. On well-drained and wind-protected areas, some trees such as *Picea glauca* and small shrubs such as *Vaccinium uliginosum* were particularly abundant. In topographic depressions, *Carex aquatilis* as found in wetter parts whereas *Vaccinium uliginosum* was found in driest locations (small

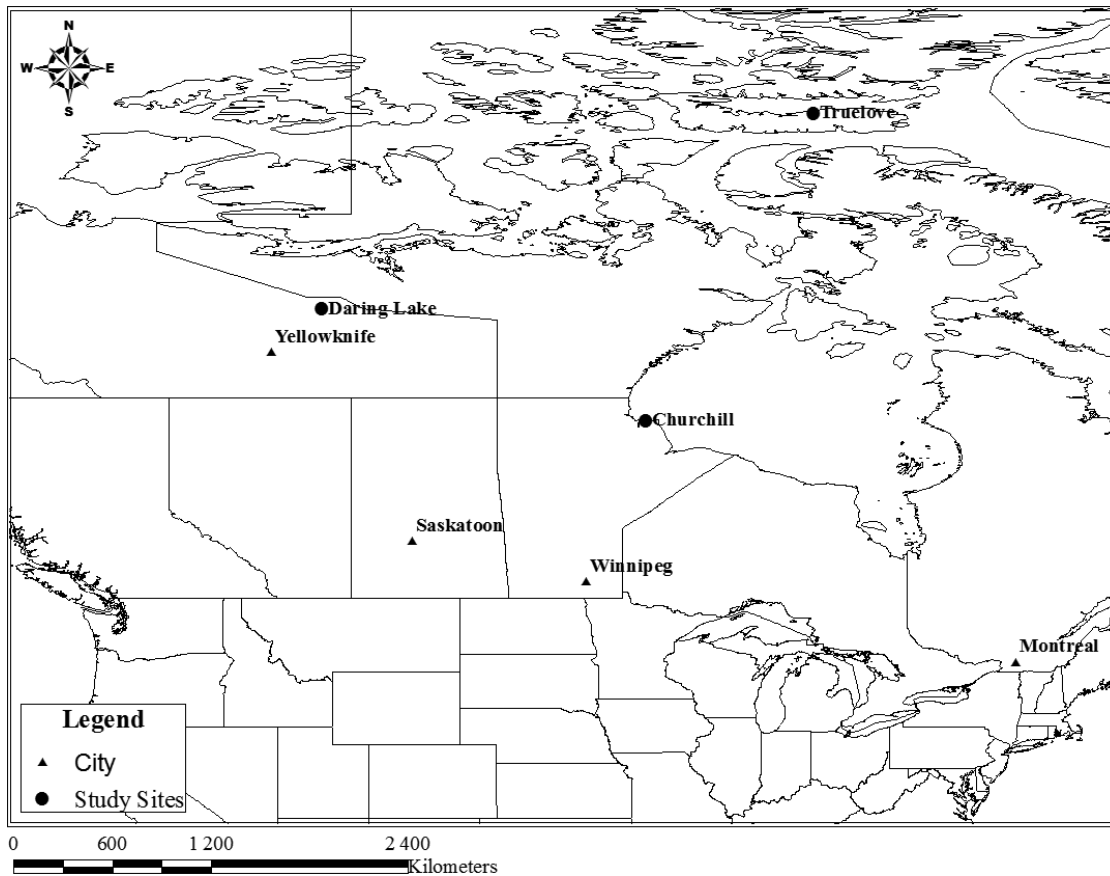


FIGURE 1. Sites investigated for this study: Subarctic (Churchill, Manitoba; 58°45'N, 93°51'W), Low Arctic (Daring Lake, Northwest Territories; 64°52'N, 111°33'W), and High Arctic (Truelove, Nunavut; 75°40'N, 84°53'W). All are in Canada.

hummocks). A detailed account of the vegetation of the Churchill lowland areas has been published by Johnson (1987).

Low Arctic: Daring Lake

The Daring Lake site is located in the Northwest Territories, Canada (64°52'N, 111°33'W) 70 km north of the tree line and approximately 300 km northeast of Yellowknife, Canada. Daring Lake is located in the subzone E arctic bioclimate (Walker et al., 2005). Exposed bedrock and lakes account for a large proportion (~50%) of the landscape. The area is characterized by complex esker systems which are mainly composed of sandy till materials with evidence of soil mass movement (Dredge et al., 1999; Ramp-ton, 2000), and localized deposits of fine-grained materials (e.g., silty sand to clayey silt) and peat (Dredge et al., 1999). The entire region is underlain by a thick permafrost layer (>160 m) with the summer active-layer depths ranging from 15 to 120 cm depending on regional characteristics such as vegetation cover, soil materials, and soil moisture (Dredge et al., 1999). Seven-year climate records from a permanent weather station (64°52'N, 111°34'W) indicated a mean annual air temperature of -9.0 °C (Nobrega and Grogan, 2008). No precipitation data are shown for this site because when and how data were collected cannot be clearly answered and/or verified. Static and Turbic Cryosols were sampled across the range of local parent materials, including lacustrine and fluvial. The rocky soils and windy areas had sparse vegetation cover with lichens and

Empetrum nigrum as the predominant species. Vascular species such as *Arctostaphylos alpina* and *Betula glandulosa* were particularly abundant on well-drained and wind protected areas. Depressions had high vegetation diversity growing on silty and/or organic materials (i.e., poorly sorted sediments) sometimes associated with mud boils. *Carex* spp. and *Eriophorum vaginatum* were particularly abundant.

High Arctic: Truelove

The Truelove (75°40'N, 84°53'W) site is a polar oasis (~43 km²) situated on the northern coast of Devon Island (~54,000 km²), Nunavut, Canada. Truelove is located in the subzone B arctic bioclimate (Walker et al., 2005). The geomorphic characteristics of the Pleistocene deposits of Truelove lowlands make it unique compared to the higher (300 m) and drier surrounding Cambrian age plateau. Similar to Churchill, the raised beach system at Truelove was formed during the Holocene by the retreat of the ice cap (in this case the Devon ice cap), progressive isostatic uplift, and wave as well as ice-push actions (Bliss, 1977; Lev and King, 1999). Therefore, the Precambrian metamorphic bedrocks of the lowlands are mantled with limestones and dolomites with a complex assemblage of fluvial, lacustrine, and periglacial deposits. Raised beaches shield the adjacent meadows from wind, increase meadow snow cover, and increase summer moisture by restricting lateral drainage (Svoboda, 1977). Therefore, this unique ecosystem has developed

greater biological diversity than the surrounding plateau area of Devon Island (King, 1991). The entire coastal lowland region is underlain by a thick permafrost layer (>200 m) (Brown, 1977). The climate data available for Truelove is very limited. Between 1970 and 1974, Truelove received mean annual precipitation of 185 mm, of which only 36 mm was rainfall (Rydén, 1977). Between 1996 and 2006 at Grise Fiord (~80 km north of Truelove; 76°25'N, 82°54'W), a permanent weather station measured a mean annual temperature of -14.2 ± 1.0 °C (mean \pm standard deviation) and a mean annual precipitation of 183.8 ± 34.2 mm (Environment Canada, 2011). Static and Turbic Cryosols were sampled across the range of local parent materials, including fluvial and organic. Lichens were the predominant vegetation on rocky and windy areas. The well-drained and wind protected areas were characterized by a pronounced hummocky surface (15-cm-high hummocks). *Dryas integrifolia* and *Cassiope tetragona* were the dominant species. Depressions were poorly drained zones that retained most of the water that drained from the surrounding area. Graminoids and bryophytes were the most common plant functional types. On boggy wetlands and where winter temperatures are harsh and snow cover is thin, hummocks (i.e., peat polygons) have developed. The peaty and mossy soils associated with hummocks had two distinct vegetation types: lichens were abundant on hummocks, whereas the wind-protected and moist soils between hummocks favored mosses and graminoids. A more detailed vegetation description of this area has been published by Bliss (1977).

METHOD OF SAMPLING

The soils sampled as well as the number of samples for each location differed among ecosystems (Table 1). Sample locations were distributed throughout the ecosystems in order to represent landscape variability. At Churchill, sampling locations included Brunisolic, Regosolic, and Gleysolic soils. At Daring Lake, Regosolic and Brunisolic soils were sampled. Finally, Brunisolic, Gleysolic, and Organic soils were sampled at Truelove. For each soil type and/or topographic location, an equally spaced sampling protocol (5 m) was used to minimize any experimenter bias. A recent study estimated a soil moisture spatial dependency with less than 1.7 m ranges for three arctic sites (Banerjee et al., 2011), making

spatial dependency among sampling locations unlikely. A more detailed description of the sampling protocol can be found in Paré and Bedard-Haughn (2012). For each sampling point, the soil was classified as either mineral or organic, based on its SOC content (i.e., Mineral < 17% SOC < Organic) (Soil Classification Working Group, 1998). At each point, the soil (0–10 cm) and associated vegetation (i.e., above and below ground materials) were gently cut with a soil knife and placed into a 10-cm-diameter plastic pot (Histoplex Histology Containers, 500 mL). The 0–10 cm increment was selected because N and C cycling processes are generally most strongly active in this section of the soil profile in the Arctic (Nadelhoffer et al., 1991). Although sampling was not performed by horizon, for the soils sampled, this increment would correspond to the Ah horizon in the majority of the mineral soils and an Om horizon in most organic soils, with minor inclusions of subsurface horizons mixed in where surface horizons were less than 10 cm. When stone rock content exceeded 10% of the volume (visually determined), the soil was gently sieved to <4.75 mm. A subsample was taken immediately for determination of water-extractable organic matter. All soil samples were stored frozen. In the laboratory, soils were thawed, the roots were removed, and the soils were sieved to <2 mm and then air dried prior to analysis.

SOIL GENERAL ANALYSIS

Soil gravimetric water content (moisture) was calculated using oven weight loss (105 °C for 24 h). Soil pH was measured in 0.01 M CaCl₂ (1:10 soil:solution) using a portable pH meter (model SP80 PC pH/cond, VWR International, Mississauga, Ontario) (Hendershot et al., 2008). Soil organic carbon (SOC) was determined by dry combustion (model C632, Leco Corporation, St. Joseph, Michigan) at 840 °C (Wang and Anderson, 1998). To remove inorganic C, all samples were acid treated with sulfurous acid (6% H₂SO₃) prior to analysis (Skjemstad and Baldock, 2008). Total nitrogen (TN) was also determined by combustion, using a CNS analyzer (Leco CNS-2000, Leco Corporation, St. Joseph, Michigan).

SOIL ORGANIC MATTER CHARACTERISTICS

Soil Organic Matter Density Fractions

The SOM density fractionation technique was used to separate the light fraction (LF) from the heavy fraction (HF) of SOM (Gregorich and Beare, 2008). Approximately 20 mL of air-dried and 2 mm sieved soil (i.e., ~17 g of mineral soil and ~5 g of organic soil) were shaken (200 rpm for 1 h) in 100 mL of NaI solution with a specific density adjusted to 1.55 g mL⁻¹ (Paré and Bedard-Haughn, 2011). After shaking, the samples were covered to prevent density change in the NaI solution and stored at ambient laboratory conditions for 48 h. Thereafter, the floating LF was collected using a vacuum system and filtered through a 0.45 µm membrane (Millipore Corporation, Billerica, Massachusetts). A second density fractionation cycle (as above) was performed to ensure a complete separation of the LF from the HF. Thereafter, both LF and HF fractions were (i) washed in 100 mL of 0.01 M CaCl₂ solution and 100 mL of de-ionized water, (ii) dried (60 °C for 48 h), and (iii) ground (<420 µm) prior to analysis. Organic C and total N of each SOM fraction (C-LF, C-HF, N-LF, and N-HF) were determined

TABLE 1

Sites and number of sample sampled for this study.

Site	Surface soil†		Number of samples (n)
	Mineral (n)	Organic (n)	
Subarctic (Churchill, Manitoba)	65	73	138
Low Arctic (Daring Lake, Northwest Territories)	47	13	60
High Arctic (Truelove, Nunavut)	7	47	54
Total	119	133	252

†Number of sample of mineral < 17% Soil Organic Carbon < organic surface soils.

as above. In order to remove carbonates, all HF samples were acid treated with 6% H₂SO₃ prior to organic C determination (Skjemstad and Baldock, 2008).

Solid-State ¹³C CPMAS NMR

Solid-state ¹³C CPMAS NMR spectroscopy was used to characterize the chemical structures of the SOM (Simpson and Preston, 2008). All analyses were carried out at the Canada Plant Biotechnology Institute in Saskatoon. Solid-state NMR spectra were acquired on a Bruker DRX-400 NMR spectrometer (Bruker BioSpin Ltd, Milton, Ontario) ($B_0 = 8.46$ T; $\nu_L(^1\text{H}) = 360.119$ MHz; $\nu_L(^{13}\text{C}) = 90.563$ MHz). A 7 mm double-resonance magic-angle spinning (MAS) probe was used. The magic angle was set by observing the ⁷⁹Br free-induction decay signal and maximizing the number of rotational echoes for solid KBr while using a spinning rate of 2.3 kHz. Chemical shift referencing and ¹H pulse width calibration were carried out using a solid sample of adamantane with a spinning rate of 2.3 kHz. The chemical shift of the high frequency ¹³C NMR signal for adamantane was set to 38.56 ppm, and a ¹H $\pi/2$ pulse width of 5.0 ms was found.

All soil samples were examined under identical experimental conditions. Samples were packed into 7 mm (o.d.) zirconia rotors and spun at a frequency of 5.0 kHz. A ¹H \rightarrow ¹³C cross-polarization pulse sequence was used to acquire data using a spectral width of 300 ppm, acquisition time of 18.96 ms, contact time of 1.0 μ s, ¹H $\pi/2$ proton pulse width of 5.0 μ s, and pulse delay of 1 s. A total of 6144 acquisitions were summed for each soil sample, and data were processed using an exponential multiplication factor of 30 Hz. Bruker Topspin 1.3 software (Bruker BioSpin Ltd, Milton, Ontario) was used to determine the relative amounts of various functional groups present in the soil samples by chemical shift regions or spectra ranges (Table 2).

TABLE 2

Spectra ranges, region names, and chemical content of soil organic matter determined by solid-state ¹³C NMR spectroscopy.[†]

Spectra range (ppm)	Region name	Chemical content
0–45	Alkyl-C (AC)	Lipids Fatty acids Plant polymers
45–110	O-alkyl-C (OAC)	Methoxyl-C (45–60 ppm) Proteins Carbohydrates (CC) (60–94 ppm) Side chains of lignin and protein
110–160	Aromatic-C (AroC)	Lignin derived molecules Protein derived molecules
160–220	Carbonyl-C (CbyC)	Esters Carboxyl groups Amide Carbonyls

[†] Information adapted from Helfrich et al. (2006), Simpson and Preston (2008), and Blumfield et al. (2004).

Water-Extractable Organic Matter

Approximately 10 g of fresh soil was gently mixed with 100 mL of water to determine the soil water-extractable organic matter (WEOM) (Chantigny et al., 2008). All samples were extracted on site and incubated *in situ* for 24 h, hand shaken, and filtered through a 0.4 μ m polycarbonate membrane filter (Whatman Inc., Piscataway, New Jersey) following the protocol of Chantigny et al. (2008). A vacuum pump (2005G2, Soil Moisture Equipment Corp., Santa Barbara, California) was used to facilitate filtration. Filtered extracts were stored frozen until analysis.

Water-soluble organic C (WSOC, mg kg⁻¹ soil) and total N (WSN, mg kg⁻¹ soil) concentrations in the filtered extract were determined simultaneously by oxidation and chemiluminescence measurement methods (TOC-V and TNM-1 Measurement Unit, Shimadzu Scientific Instruments, Kyoto, Japan). Ammonium concentrations (NH₄⁺, mg kg⁻¹ soil) were determined colorimetrically following the phenolphthorite method (Solorzano, 1969) using a SmartChem 200 Discrete Autoanalyzer (Westco Scientific, Brookfield, Connecticut). Nitrate concentrations (NO₃⁻, mg kg⁻¹ soil) were determined by reducing NO₃⁻ to nitrite (NO₂⁻) by passage through an open tubular copperized cadmium reactor. Nitrite concentrations were then determined colorimetrically by diazotizing with sulphanilamide followed by coupling with N-(naphthyl)-ethylenediamine dihydrochloride using the SmartChem 200 Discrete Autoanalyzer. Water-soluble organic nitrogen (WSON, mg kg⁻¹ soil) was calculated according to Equation 1.

$$\text{WSON} = \text{WSN} - ([\text{NH}_4^+] + [\text{NO}_2^-]) \quad (1)$$

STATISTICAL ANALYSIS

Variance homogeneity was evaluated with the Levene test. Data were transformed (i.e., logarithm or exponential) when they were not normally distributed. Multiple-factors ANOVA (type III sums of squares) was used to determine differences in soil properties and SOM quality characteristics between sites (df = 2) and soil type (df = 1) using general linear model procedure in SPSS version 13 for Windows (SPSS Inc., 2004). Because we had unequal group sizes, the Games-Howell post-hoc test was used to determine differences among sites.

Results and Discussion

SOIL ORGANIC MATTER DENSITY FRACTIONS

The C and N measured in the LF and HF of SOM were higher in organic soils than in mineral soils (Table 3). Churchill and Daring Lake organic soils had more C and N stored in the LF of SOM than Truelove (Table 3). This may be explained by the difference in vegetation between sites where Churchill and Daring Lake (i.e., subarctic and Low Arctic) organic soils had greater above-ground biomass (e.g., tall shrubs) than Truelove (i.e., High Arctic). Low and similar C-LF and N-LF values were measured for all mineral surface soils (Table 3) because the vegetation on mineral soils (e.g., dry heath lichens) was similar among sites. Organic soils from all three sites had different C-HF and N-HF values, suggesting different N cycling processes among these sites (Hassink, 1995a; Curtin and Wen, 1999; Accoe et al., 2004). For both types of surface soils

TABLE 3

Statistical comparisons of organic matter quality parameters among sites (S) and between soil type (T).†

	Churchill	Daring	Truelove	Churchill	Daring	Truelove	ANOVA		
		Mineral			Organic		S (P)	T (P)	S × T (P)
<i>Soil Properties</i>									
Moisture §, g g ⁻¹	0.5(0.6) ^b	0.6(0.3) ^a	0.8(0.5) ^a	2.8(1.4) ^a	3.2(1.1) ^a	1.4(1.1) ^b	0.007	<0.001	<0.001
pH	6.5(0.2) ^a	4.0(0.4) ^b	6.5(0.1) ^a	5.8(1.0) ^b	3.3(0.1) ^c	6.1(0.4) ^a	<0.001	<0.001	0.352
SOC ¶, g 100 g ⁻¹	7.6(4.2) ^a	4.9(3.5) ^b	11.7(4.9) ^a	40.3(7.2) ^a	40.0(6.2) ^a	29.3(5.4) ^b	<0.001	<0.001	<0.001
TN §, g 100 g ⁻¹	0.4(0.2) ^b	0.2(0.2) ^c	0.9(0.4) ^a	2.1(0.7) ^a	1.4(0.2) ^b	1.9(0.2) ^a	<0.001	<0.001	<0.001
Soil C:N §	20.2(6.3) ^b	25.9(8.3) ^a	14.7(2.6) ^c	22.2(10.6) ^b	29.3(3.4) ^a	15.1(2.3) ^c	<0.001	0.122	0.723
<i>Density Fractions</i>									
C-LF §, g 100 g ⁻¹	1.8(1.4) ^a	0.9(1.3) ^b	1.6(2.9) ^{ab}	21.1(10.7) ^b	30.0(7.9) ^a	5.9(6.0) ^c	<0.001	<0.001	<0.001
C-HF ¶, g 100 g ⁻¹	5.4(3.8) ^b	4.4(2.5) ^b	12.0(4.4) ^a	17.0(6.0) ^b	9.4(3.4) ^c	22.1(3.3) ^a	<0.001	<0.001	<0.001
N-LF ¶, g 100 g ⁻¹	0.07(0.06) ^a	0.03(0.04) ^b	0.08(0.15) ^{ab}	0.87(0.54) ^a	0.97(0.24) ^a	0.31(0.28) ^b	<0.001	<0.001	<0.001
N-HF ¶, g 100 g ⁻¹	0.23(0.18) ^b	0.22(0.13) ^b	0.86(0.37) ^a	1.02(0.53) ^b	0.34(0.15) ^c	1.59(0.31) ^a	<0.001	<0.001	<0.001
LF C:N ¶	27.2(5.0) ^b	31.6(6.1) ^a	22.9(4.1) ^b	27.2(10.4) ^b	31.1(3.9) ^a	18.6(2.6) ^c	<0.001	0.075	0.266
HF C:N §	25.6(8.4) ^a	21.2(4.7) ^b	15.2(3.5) ^c	19.7(8.3) ^b	28.5(4.4) ^a	14.2(1.9) ^c	<0.001	0.795	<0.001
<i>Solid-State ¹³C NMR Spectroscopy</i>									
Carbonyl-C (CbyC) §, %	9.3(2.5) ^a	10.3(6.3) ^a	9.5(4.8) ^a	6.9(1.2) ^b	4.5(1.0) ^c	8.2(1.6) ^a	<0.001	<0.001	<0.001
Alkyl-C (AC), %	20.1(2.6) ^b	26.1(4.3) ^a	20.3(4.9) ^b	19.3(1.9) ^b	23.2(3.2) ^a	22.7(4.3) ^a	<0.001	0.494	0.008
Aromatic-C (AroC) §, %	11.7(3.9) ^b	15.3(4.6) ^a	17.7(7.0) ^a	8.6(1.5) ^b	7.0(1.6) ^c	12.7(2.3) ^a	<0.001	<0.001	<0.001
O-Alkyl-C (OAC) ¶, %	58.8(5.0) ^a	48.3(7.5) ^b	52.6(6.7) ^{ab}	65.2(2.9) ^a	65.3(4.0) ^a	56.4(3.4) ^b	<0.001	<0.001	<0.001
Carbohydrates-C (CC), %	42.3(4.8) ^a	33.2(5.6) ^b	33.8(4.5) ^b	47.5(3.0) ^a	48.5(3.8) ^a	37.7(3.5) ^b	<0.001	<0.001	<0.001
CC:MC §	5.4(1.4) ^a	4.6(1.1) ^b	3.6(1.1) ^c	6.6(1.5) ^a	7.6(1.8) ^a	4.0(1.2) ^b	<0.001	<0.001	<0.001
OAC:AroC §	5.8(2.8) ^a	3.5(1.4) ^b	3.4(1.3) ^b	7.8(1.6) ^a	10.0(3.3) ^a	4.6(1.0) ^b	<0.001	<0.001	<0.001
OAC:AC §	3.0(0.5) ^a	1.9(0.4) ^b	2.7(0.4) ^a	3.4(0.5) ^a	2.9(0.5) ^b	2.6(0.7) ^b	<0.001	<0.001	<0.001
<i>Water-Extractable Organic Matter (WEOM)</i>									
WSOC ¶, µg g ⁻¹	38.5(36.0) ^a	48.2(46.7) ^a	60.3(37.8) ^a	175.4(143.2) ^b	622.8(257.7) ^a	198.4(160.4) ^b	<0.001	<0.001	<0.001
WSON ¶, µg g ⁻¹	0.9(1.6) ^b	1.1(1.0) ^b	2.8(1.4) ^a	2.4(7.3) ^b	10.1(4.6) ^a	10.3(8.4) ^a	<0.001	<0.001	0.031
WEOM C:N ¶	17.9(4.7) ^b	57.7(18.3) ^a	20.6(6.4) ^b	20.2(3.4) ^b	70.0(16.0) ^a	17.4(4.6) ^b	<0.001	0.117	0.011
n	65	47	7	73	13	47			

Mean (standard deviation). †Multiple-factors ANOVAs (type III sums of squares) among sites and soil types. Site (df = 2): Subarctic (Churchill), Low Arctic (Daring Lake), and High Arctic (Truelove). Soil Type (df = 1): mineral (<17% carbon) and organic (>17% carbon). Logarithm (§) or exponential (¶) transformations applied to meet normality. Sites not sharing a letter (per soil type) are different at $P < 0.05$ using Games-Howell as a post hoc test. Light fraction (LF < 1.55 g mL⁻¹) and Heavy fraction (HF > 1.55 g mL⁻¹) of soil organic matter. WSOC: Water-soluble organic carbon. WSON: Water-soluble organic nitrogen.

(i.e., mineral and organic), Truelove had the lowest LF C:N and HF C:N values most likely because Churchill and Daring Lake (i.e., subarctic and Low Arctic) had more lignin-rich plants (e.g., shrubs and trees) than Truelove (i.e., High Arctic). Small trees (e.g., *Picea glauca*) and shrubs (e.g., *Vaccinium uliginosum*) characterized vegetation at Churchill and Daring Lake, whereas herbaceous plants (e.g., *Dryas integrifolia*) characterized vegetation at Truelove. Further work is required to test this hypothesis because C:N ratios of plant biomass were not measured.

When compared with the values reported in the comprehensive review of LF literature by Gregorich et al. (2006), we found that the LF C:N values were ~50% higher for Churchill and Daring Lake and ~25% higher for Truelove compared with agricultural, forest, and grassland soils. The results from Churchill and Daring Lake corresponded to those found for surface soils from the southern part of Siberia with similar vegetation types (Gundelwein et al., 2007). However, direct comparison with this study is problematic

because of differences in sampling depths and in density of the heavy liquid used to separate LF from HF. Recently, Paré and Bedard-Haughn (2011) determined an optimum liquid density of 1.55 g mL⁻¹ to separate LF and HF of SOM from three Canadian Arctic sites. A liquid density of 1.55 g mL⁻¹ may serve as a reference starting point to separate LF and HF from arctic SOM.

SOLID-STATE ¹³C CPMAS NMR

The proportion of labile C [e.g., O-Alkyl-C (OAC) and Carbohydrates-C (CC)] was significantly higher in organic than mineral surface soils (Table 3). Furthermore, mineral surface soils had relatively more recalcitrant C [e.g., Alkyl-C (AC); and Aromatic-C (AroC)] than organic surface soils (Table 3). Most literature has consistently demonstrated that the preferred materials for decomposition by soil microbes are in the OAC group because this group is generally dominated by celluloses and other polysaccharides (e.g.,

carbohydrates-C), whereas AC and AroC groups tend to decompose slowly over time (Sollins et al., 1996; Skjemstad et al., 1997). Therefore, C-group ratios such as OAC:AroC and OAC:AC are good indicators of the overall characteristics of SOM since soil microorganisms preferably degrade OAC groups in most unmanaged ecosystems such as oak, pine, and mixed forests, and northern uncultivated organic soils from Québec (Quideau et al., 2000). Organic soils had significantly higher 'lability' ratios (e.g., OAC:AroC, and OAC:AC), suggesting that the organic soils were more labile than mineral soils (Table 3). These results support and reinforce the previous SOM density fraction findings.

Churchill mineral surface soils store relatively more labile C (e.g., higher CC:MC and OAC:AroC ratios) than Daring Lake or Truelove mineral surface soils (Table 3). Churchill and Daring Lake organic soils had more labile C than Truelove organic soils (Table 3). This may be explained by the difference of vegetation between sites where Churchill and Daring Lake (i.e., subarctic and Low Arctic) organic soils had higher above-ground biomass (e.g., tall shrubs) than Truelove (i.e., High Arctic). Therefore, this condition contributed to greater accumulation of fresh and labile SOC in the Churchill and Daring Lake organic surface soils compared to Truelove.

Compared with other unmanaged ecosystems, both organic and mineral soils had ~20% more OAC and ~20% less AC than a northern hardwood forest soil from New Hampshire, U.S.A. (Usiri and Johnson, 2003) and ~20% more OAC and ~5% less AC than two prairie soils (Baldock et al., 1992). Recently, Pedersen et al. (2011) and Grover and Baldock (2012) also found high proportions (>65%) of OAC at the soil surface in northern Alaska and Australian Alps, respectively. Our arctic soils had ~10% more OAC and 10% less AC than a spruce forest in Bavaria, Germany (Helfrich et al., 2006). For our arctic surface soils, CC represented 33 to 48% of the total C pool, which was approximately 70% of the entire OAC group. Similarly, Strebel et al. (2010) recently found that the CC concentrations represented approximately 40% of the C pool of a high arctic site in Svalbard, Norway.

Therefore, high proportions of OAC and CC and small proportions of AC, AroC, and CbyC of arctic surface soils indicated a small degree of humification of SOM from arctic surface soils. Low temperatures, the permafrost table, and its interaction with soil hydrology are soil conditions that reduce SOM decomposition (Hobbie et al., 2000; Tarnocai et al., 2008) and hence explain the smaller degree of humification of SOM from arctic surface soils compared to soils of more temperate ecosystems.

WATER-EXTRACTABLE ORGANIC MATTER

The C and N measured in WSOC and WSON were significantly higher in organic than in mineral surface soils (Table 3) because soils with higher SOC generally have higher WSOC and WSON pools (Zsolnay, 1996; Chantigny, 2003). The WEOM C:N values did not differ between mineral and organic soils (Table 3), which suggest that the WEOM characteristics were similar between both types of soil. However, WEOM C:N values cannot be used as a stand-alone measurement, and a better knowledge of WEOM characteristics (e.g., labile and aromatic structures of WEOM) is still needed (Woods et al., 2011). For mineral surface soils, WSOC values did not significantly differ among sites, whereas WSON

values were slightly higher for Truelove (Table 3). Daring Lake organic soils had significantly higher WSOC than Churchill and Truelove organic soils (Table 3). Higher plant biomass, and hence higher SOC content, found in Churchill and Daring Lake compared to Truelove (Table 3) could not explain this difference. Other factors include temporal variability, pH, and/or solubility of root exudates. First, differences in WEOM among sites could be simply caused by temporal variations, since WEOM can vary considerably among seasons and years (Zsolnay, 2003; Embacher et al., 2007). However, because Daring Lake had approximately threefold higher WSOC than Churchill and Truelove, we believe that this large difference could not be caused only by temporal variations. Second, low soil pH measured in Daring Lake compared to Churchill and Truelove (Table 3) may promote dissolution of SOC since most of SOC solubility is pH-dependent (Swift, 1996; Anderson and Schoenau, 2008). Hypothetically, the significantly lower soil pH found in Daring Lake could result in a different soil WSOC steady state. However, higher soil WEOM values measured at near-neutral pH (pH 7.4) compared to acid soils (pH 4.5–5.3) do not support this hypothesis (Kuiters and Mulder, 1993). Third, it is possible that plant roots from Daring Lake released more water-soluble organic compounds since plants can directly increase soil WSOC by releasing C into soil solution (Séguin et al., 2004). Plants from Daring Lake might gain some advantages by doing so because this site appears to be the most N limited, as reflected by the lowest TN and the highest soil C:N values (Table 3). Further experimentation would be required to confirm which explanation is most plausible.

Churchill and Truelove mineral and organic soils had lower WEOM C:N values than Daring Lake (Table 3). The parent soil material may explain this phenomenon. Churchill and Truelove soils were both formed on carbonate-rich parent materials, whereas Daring Lake soil was formed on acidic parent materials—reflected by soil pH values (Table 3). Soils close to neutrality, such as Churchill and Truelove soils, tend to have a more active and diverse soil microbial community than acidic soils (Persson et al., 1989; Anderson and Joergensen, 1997), such as found in Daring Lake, contributing to higher WEOM C:N ratios in acidic mineral and organic soils. Nevertheless, this mechanism needs to be verified since other explanations may also account for such differences in WEOM C:N values (hydromorphism of SOM, water table depths and movements, soil oxygen levels, etc.).

The WSOC values were approximately tenfold lower compared to most temperate forest surface soils (Zsolnay, 1996; Chantigny, 2003). Furthermore, the WEOM C:N ratios were about fourfold higher than those measured during many years of forest-stand experimentation from northern Alberta (Teklay and Chang, 2008). However, WEOM varied considerably among seasons, as well as among years, because WEOM is strongly affected by climatic conditions such as temperature and precipitation (Zsolnay, 2003). Therefore, comparisons among studies and ecosystems are extremely difficult. Despite this, the huge gap between temperate forests and arctic soils suggested that arctic WEOM pools are small compared to more temperate forest ecosystems. These findings can be confusing since arctic soils stored relatively high amounts of SOM and highly labile C compared to such ecosystems. However, low WSOC and high WEOM C:N highlight the relative limitation of accessible nutrients for arctic soil microbes and plants. Further-

more, these results may suggest tighter soil nutrient cycling in arctic soils compared to temperate forests.

SOIL ORGANIC MATTER CHARACTERISTICS IN A CHANGING CLIMATE

The effect of climate change on SOM decomposition has been widely studied in the Arctic (Shaver et al., 1998; Christensen et al., 1999; Rodionov et al., 2006; Oelbermann et al., 2008; Rinnan et al., 2008). However, only few studies have attempted to elucidate differences among distinct arctic sites (ecosystems). Increasing *in situ* temperature by 2 °C increased SOM decomposition in the subarctic (Abisko, Sweden) but had no significant impact on SOM in the High Arctic (Ny-AaNESund, Svalbard) (Robinson et al., 1997). Therefore, the latter study suggested that SOM qualities, rather than soil temperature, might initially drive SOM decomposition in the Arctic. This conclusion was earlier supported by Nadelhoffer et al. (1991) who found that, under field moisture and temperature ranges, SOM qualities were primary factors explaining differences of SOM decomposition rates among arctic ecosystems and sites. Because the LF of SOM is composed primarily of fresh to partially decomposed plant residues (Spycher et al., 1983; Elliott and Cambardella, 1991), which are highly labile (Janzen, 1987; Hassink, 1995a, 1995b) and can be rapidly modified by environmental changes such as climate change (Janzen et al., 1992; Biederbeck et al., 1994), soils storing a greater proportion of LF are at risk of losing SOM. For Churchill, Daring Lake, and Truelove organic soils, 53, 73, and 20% of the C was included in the LF, respectively, whereas 24, 19, and 14% of the C was included in the LF of mineral soils, respectively.

Most literature has demonstrated that the preferred materials for decomposition by soil microbes are in the OAC group, whereas AC, AroC, and CbyC groups tend to decompose more slowly. Soils from Churchill and Daring Lake had generally higher OAC:AroC and OAC:AC ratios than soils from Truelove, indicating that subarctic (Churchill) and low arctic (Daring Lake) surface soils store relatively more labile SOM than the high arctic soils (Truelove). Similar to Robinson et al. (1997), these results suggest that subarctic and low arctic SOM is likely more sensitive to climate change than SOM from the High Arctic. Furthermore, the results suggest that organic surface soils from the Arctic are likely more sensitive to climate change than mineral surface soils.

In arctic soils, the bioavailability of WEOM plays a large role in determining whether C is lost through leaching or gaseous emissions following its decomposition by heterotrophic soil microbes (Neff and Hooper, 2002; Gundelwein et al., 2007). However, it is extremely difficult to predict the real effects of climate change on soil WEOM because soil WEOM is temporally and spatially highly variable (Zsolnay, 2003). Nevertheless, with both higher temperatures and higher precipitation expected for most of the terrestrial Arctic (Huntington et al., 2005; Kattsov et al., 2005), it is likely that WEOM could play a greater role in soil nutrient cycling as well as soil C sequestration. The results from this study indicate that WEOM was more vulnerable to climate change in organic than mineral surface soils because (1) low WSOC and WSON values were measured in mineral soils compared with organic soils, and (2) different sites (i.e., climates) did not differ in WSOC and WSON in mineral soils.

Conclusions

This study substantially improved our knowledge related to SOM characteristics of three distinct arctic surface soil sites. As expected (first hypothesis), all analyses of solid SOM (density fractions and solid-state ¹³C CP/MAS NMR) showed that organic surface soils (>17% C) contained relatively more labile C than mineral surface soils (<17% C). In opposition to our second hypothesis, this study showed that the subarctic and low arctic sites stored relatively more labile SOM than the high arctic site. Compared to temperate ecosystems from literature, arctic surface soils accumulated more labile C with a high release potential under more suitable conditions (i.e., warmer climates). Therefore, high SOM lability may trigger a substantive release of GHG into the atmosphere—enhancing the climate change effect. However, higher SOM decomposition could also lead to higher soil nutrient availability (e.g., higher soil N mineralization via SOM decomposition) for plants and therefore increase the C sequestration potential of arctic ecosystems (i.e., more C absorbed by plants because nutrients are less limiting). The relative persistence of arctic SOM, within and among sites, will ultimately be controlled primarily by the extent and type of changes in their immediate biophysical environment and the interaction of those changes with SOM chemistry (Schmidt et al., 2011). Cryoturbation and soil moisture redistribution need to be considered as these processes directly and indirectly affect soil C storage as well as soil nutrient availability of arctic soils (Dai et al., 2001; Shaver et al., 2006). A better understanding of these mechanisms involved in soil C storage as well as soil nutrient cycling in the Arctic is needed.

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