

Quantifying and characterising organic  
carbon and microbes in newly developed  
soils following glacier retreat in northern  
latitudes

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Quantifying and characterising organic  
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*This thesis is dedicated to my family,  
late Murzabay Akhmetkaliyev,  
late Bigaysha Kaliyeva,  
Gulzhian Akhmetkaliyeva  
and  
late Gennadiy Gomola*

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*Эта диссертация посвящается моей семье,  
Мурзабаю Ахметкалиеву,  
Бигайше Калиевой,  
Гульжиан Ахметкалиевой  
и  
Геннадью Гомола*

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# Abstract

Glacier retreat in northern latitudes expose nutrient rich glacial landscapes that may develop soils and ecosystems via accumulation of soil organic carbon (OC). Accumulation of soil derived OC was investigated in three contrasting glacial systems (Tarfala in Sweden, Vatnajökull ice cap in Iceland and Zackenberg in Greenland) in order to understand the main source of OC in soils and distribution of soil OC along downstream transects from the glacier front. Soil and sediment samples were analysed for OC concentration, bacteriohopanepolyol biomarkers (BHPs) and glycerol dialkyl glycerol tetraethers (GDGTs), groups of membrane lipids that can be used to trace major microbial groups, DNA sequencing of microbes and major elements.

Soil and sediment samples from Sweden showed low OC concentrations ( $0 - 1.08 \pm 0.09\%$ ), while samples from Iceland ( $0.01 - 5.24 \pm 2.68\%$ ) and Greenland ( $0.01 \pm 0.01 - 4.88 \pm 3.56\%$ ) had higher values. Soils from older moraines showed highest OC concentrations (up to 8.96% in Greenland), while fluvial sediment samples from all study areas had low to no OC. BHPs and GDGTs were rare in fluvial sediments, observed in riverbank soils and most common in moraines. Distribution of soil specific BHPs and the  $R'_{\text{soil}}$  index suggests soil development in recently deglaciated areas along downstream transects from the glacier front, followed by stabilisation in older soils in Iceland and Greenland. Microbial communities stabilised along transects, quickly adapting to the new environment. Acidobacteria, Actinobacteria, Chloroflexi, Proteobacteria, Planctomycetes and Verrucomicrobia were the most abundant phyla identified in post-glaciated terrains, while candidate phylum AD3 had surprisingly high concentration in samples from Sweden. Linking biomarkers with bacterial community showed that soil marker BHPs in samples from Sweden were mainly produced by Rhodospirillaceae or purple non-sulfur bacteria, while Bradyrhizobiaceae, Hyphomicrobiaceae and Nitrosomonadaceae were responsible for production of soil marker BHPs in Iceland and Greenland. BrGDGTs, indicative of terrestrial OC, were produced by Acidobacteria.

Based on these data, it can be concluded that soil and ecosystem development in front of retreating glaciers leads to the build-up of new terrestrial OC stores and future glacier retreat in deglaciating systems in northern latitudes might increase terrestrial OC productivity.

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# 1 Introduction

## 1.1 Climate change in the Arctic and Subarctic

Glaciers cover ~11% of Earth's land surface. Most of the glaciers are located either in high elevation altitude areas (up to over 8000 m above sea level (asl) in the Himalaya and Karakoram) or in high latitudes, such as the Arctic (above 66°33' N) and Subarctic (between 50°N and 70°N) – a region that is experiencing the most rapid and greatest magnitude of warming on Earth. Based on observations during 1979-2021, Arctic mean temperature increased by 0.73°C per decade, as opposed to global 0.19°C per decade (Rantanen et al., 2022). Predicted warming through this century (Meredith et al., 2019) will result in global glacier recession with exposure of nutrient rich deglaciated terrains that have the potential to develop soils and ecosystems. Soil development in post-glaciated areas of Arctic and Subarctic regions takes place despite extreme weather conditions, such as cold winters and wet summers, and lack of organic input (Doğrul Selver et al., 2012). As glaciers retreat due to climate change, soils that develop in nutrient-rich terrains accumulate organic carbon (OC) (Vilmundardóttir et al., 2014).

## 1.2 Soil development

During Quaternary glaciation characterized by periods of glacier advance and recession, glaciers shaped landscapes and deposited glacial debris that has been developed into re-vegetated land. As glaciers retreat due to the current climate change overridden soils and vegetation are exposed, leading to soil development. Therefore post-glaciated areas present spatial representation of temporal changes in terms of OC accumulation and ecosystem development. Generally, younger soils are close to the glacier front, with older soils along the transects being more developed (Schmidt et al., 2008).

Soil development studies have previously focused on plant succession (e.g., Bormann and Sidle, 1990; Matthews, 1992; Chapin et al., 1994 in Schmidt et al., 2008) and recently, have expanded into investigating microbial succession along glacier foreland chronosequences (space for time substitution) (e.g., Schütte et al., 2009; Schütte et al., 2010; Mapelli et al., 2018; Wojcik et al., 2020; Venkatachalam et al., 2021). Studies suggested that soil development in glacier forelands takes place over time as a result of bacterial succession and plant colonization of the terrain after deglaciation. Soil OC accrues due to microbial metabolism and decomposition of plant material in these soils. For example, soil OC in the foreland of the Fláajökull glacier in south-eastern Iceland increased from  $\sim 0.19$  to  $1.7 \text{ kgCm}^{-2}$  with the soil age from youngest (<10 years old) to oldest (122 years old) (Wojcik et al., 2020). Similarly, Skaftafellsjökull glacier foreland had soil OC in the range of 0.04 to  $1.1 \text{ kgCm}^{-2}$  (Vilmundardóttir et al., 2015). However, studies of soil development in post-glaciated terrains did not provide information about the direct source of OC. Some studies from non-glaciated catchments have successfully used microbial soil biomarkers to distinguish between OC sources (e.g., Zhu et al., 2011; Doğrul Selver et al., 2012; Doğrul Selver et al., 2015).

### **1.3 Biomarkers and their role in identifying terrestrial input**

The presence or absence of some organic biomarkers is usually characteristic to a particular environment (Killops and Killops, 2005). Therefore, in order to better understand the processes involved in accumulation of soil OC in deglaciated areas, this study uses organic biomarkers, bacteriohopanepolyols (BHPs) and glycerol dialkyl glycerol tetraethers (GDGTs), which were previously used as soil tracing biomarkers (i.e., Cooke et al., 2008; Cooke et al., 2009; Doğrul Selver et al., 2012; Doğrul Selver et al., 2015).

#### **1.3.1 Bacteriohopanepolyols (BHPs)**

There is a direct relation between the carbon content and bacteriohopanepolyol (BHP) concentration and composition in soils (Höfle et al., 2015). BHPs are a group of membrane lipids, with different BHPs produced by different bacteria, and therefore used as biomarkers of bacterial presence. BHP distribution also provides information about biogeochemical processes in various environments (Rethemeyer et al., 2010). Number,

position and nature of functional groups in the side chain determine structural diversity of BHPs (Rethemeyer et al., 2010; Hofte et al., 2015) (see Figure 1.1 for structures).

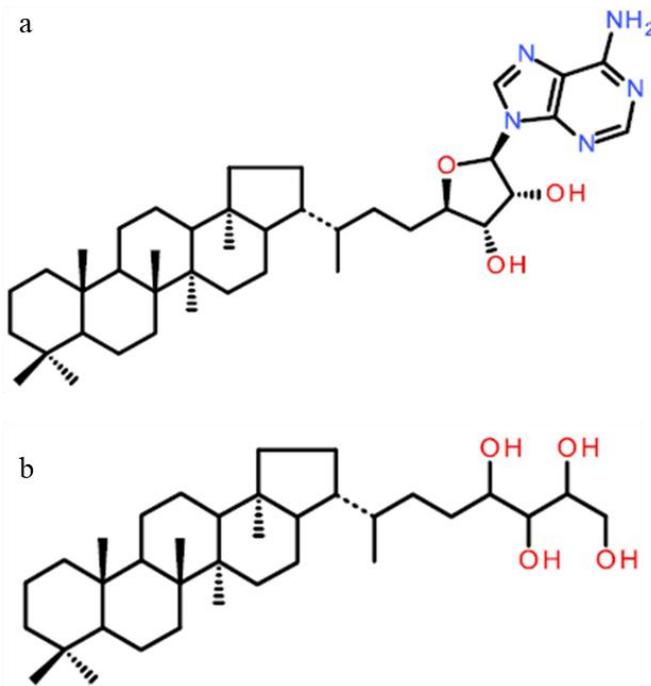


Figure 1.1. Structures of (a) soil BHP compounds and (b) bacteriohopanetriol (BHT). Adapted from Langworthy et al., 1976 and Neunlist et al., 1988.

BHPs can be used for studying modern trends of organic matter transport and paleo studies, including assessment of bacterial activity in post-glaciated till in Northeast England (Cooke, 2011). Even though previously there were few studies on BHP distribution of post-glaciated areas, previous studies of different soil horizons and permafrost layers have shown a difference in the distribution of BHPs (e.g., Zhu et al., 2011; Doğrul Selver et al., 2012; Höfle et al., 2015). BHPs have the potential to be used to locate the source and origin of organic carbon, including the transport of organic carbon from glaciers down the stream (Cooke, 2011) due to the spatial variability in BHP distribution depending on sampling location (Höfle et al., 2015).

The distribution of BHPs is determined by carbon and nitrogen concentrations (Höfle et al., 2015). Soils and sediments rich in organic carbon show high concentrations of soil biomarkers (Rethemeyer et al., 2010; Höfle et al., 2015) that can be used to trace major microbial organisms. Different bacteria produce different BHPs, enabling to use them as biomarkers, especially soil-markers such as adenosylhopane and other related structures (Cooke et al., 2008; Cooke et al., 2009). Using microbial soil biomarkers has the advantage

of being able to distinguish between plant derived versus soil derived organic matter, as certain BHPs are related to terrestrial input (Doğrul Selver et al., 2012). This provides an opportunity to understand accumulation of OC in soil due to the bacteria living in terrestrial soils rather than as a result of degradation of plant material. The presence of soil-specific BHPs in sediment samples indicates terrestrial input (Cooke et al., 2009). Terrestrial biomarkers have been found in ancient sediments, which demonstrates the ability of BHPs to persist degradation (van Dongen et al., 2006). Since soil marker BHPs, adenosylhopane and related structures are better preserved at lower temperatures (Rethemeyer et al., 2010; Bischoff et al., 2016), high-latitude glaciers are excellent systems for BHP analysis.

In addition to using BHPs as a proxy for tracing soil biomarkers, the  $R_{\text{soil}}$  index can be used to trace soil derived organic matter (OM). Zhu et al. (2011) suggested using the  $R_{\text{soil}}$  index as a proxy for tracking soil OM. The  $R_{\text{soil}}$  index shows relative abundance of soil marker BHPs to bacteriohopanetetrols (BHTs), which are present in most environmental systems (Doğrul Selver et al., 2012) (see Figure 1.1 for structures). According to Doğrul Selver et al. (2012), environmental conditions, such as the location (Arctic) of sampling sites, affects the distribution of soil marker BHPs and therefore, the  $R'_{\text{soil}}$  is more effective in cold environments compared to the  $R_{\text{soil}}$  index. The  $R'_{\text{soil}}$  index takes into account only non-methylated soil-marker BHPs.

### 1.3.2 Glycerol dialkyl glycerol tetraethers (GDGTs)

Glycerol dialkyl glycerol tetraethers (GDGTs) are also organic biomarkers that can be used to trace soil derived OM. GDGTs are membrane lipids and can be divided into two main groups: branched GDGTs (brGDGTs) and isoprenoid GDGTs (isoGDGTs) (see Figure 1.2 for structures). BrGDGTs are produced by anaerobic soil bacteria (Weijers et al., 2007) and are mostly found in terrestrial environments (Hopmans et al., 2004; Weijers et al., 2006), while isoGDGTs, mainly crenarchaeol, are found in marine environments (Sinninghe Damsté et al., 2002). Previous studies used GDGT based proxy to trace soil derived OM along land to sea/ocean transects (e.g., Doğrul Selver et al., 2012; Doğrul Selver et al., 2015). In this study, for the first time brGDGTs are used to investigate soil OC in post glaciated terrain along with soil marker BHPs.

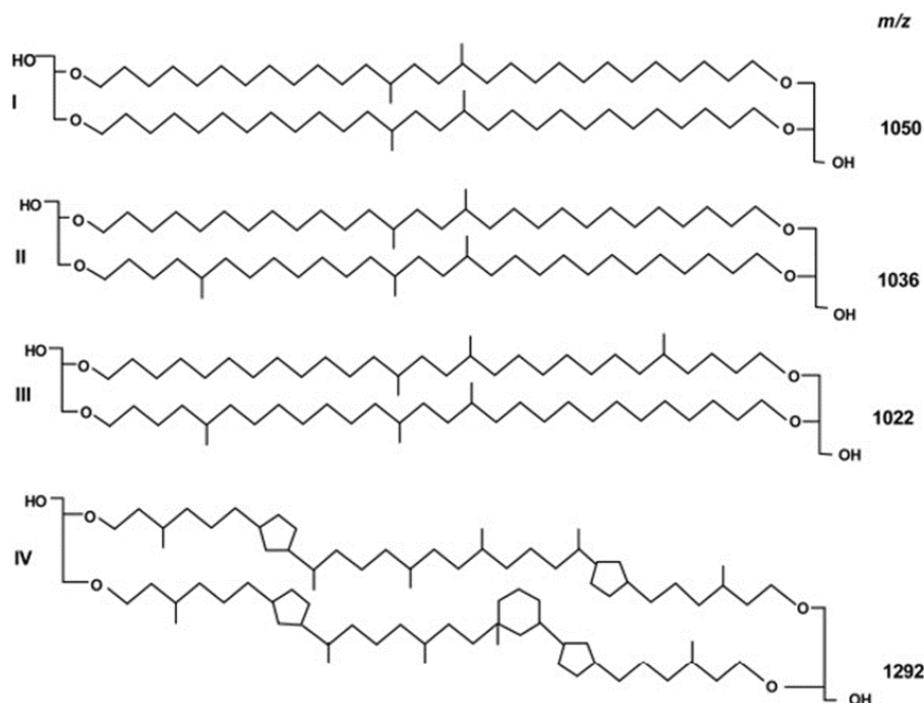


Figure 1.2. Structures of brGDGT I, II, III and crenarchoel (IV) (Doğrul Selver et al., 2012).

According to Weijers et al. (2007), the distribution of brGDGTs is correlated with soil pH and mean annual air temperature (MAT). Therefore, brGDGTs can also be used as a proxy to study paleotemperatures (Weijers et al., 2007) and soil pH (Peterse et al., 2012).

## 1.4 Microbial communities in post-glaciated areas

Despite extreme environmental conditions, post-glaciated areas in the Arctic are productive, allowing microbial colonization and succession along glacier chronosequences (Venkatachalam et al., 2021). Generally, OC in proglacial areas is a product of chemical and mechanical weathering of organic material, bedrock material (Bhatia et al., 2013) and overridden paleosoil erosion (Lawson et al., 2014). Decomposition and release of labile OC in glaciated catchments is a process driven by microbial activities (Rethemeyer et al., 2010).

As glaciers recede, labile OC is exposed, providing an opportunity for different bacteria to colonize the post-glaciated terrain. Labile OC is utilized by heterotrophic microbial communities (Singer et al., 2012). Ancient carbon consumed by heterotrophs show vascular plant or overridden soil origin indicative of terrestrial signature originated during periods of glacier advance (Hood et al., 2009; Singer et al., 2012). Primary succession of deglaciated substrate is first carried out by heterotrophic microbial communities before the enactment

of autotrophic communities, which metabolize modern carbon derived from revegetation of deglaciated terrains (over 50 years) (Bardgett et al., 2007).

Some studies focused on succession of microbial communities along glacier chronosequences (e.g., Schütte et al., 2009; Schütte et al., 2010; Mapelli et al., 2018; Wojcik et al., 2020; Venkatachalam et al., 2021) and found that bacterial diversity increases in the initial stages of succession, with a period of plateau in older soils. For instance, according to Wojcik et al. (2020), bacterial diversity increases from young to old moraines, with a period of plateau in more developed soils in the glacier foreland of Fláajökull in south-eastern Iceland. However, as DNA is prone to quick degradation, there is a risk that the bacterial community might have changed from its original composition. Therefore, since the degradation products of organic biomarkers are preserved, allowing organic biomarkers to be traced through space and time, the coupling of two methods, microbial sequencing and biomarker analysis, is a powerful tool for accessing living and dead bacterial cells, which can aid in tracing the soil OC accumulation in post-glaciated areas (Höfle et al., 2015).

## **1.5 Role of metals and nutrients and water chemistry**

The availability of nutrients and the length of exposure to weathering are crucial for soil development (Burga et al. 2010). The nature of the bedrock has a high impact on the suspended matter concentration. For instance, at Zackenberg, sedimentary rocks have very high values of suspended matter of up to 2500 mg/L due to susceptibility to erosion, while crystalline rocks have a low value of 18 mg/L (Hasholt and Hagedorn, 2000). These values have a direct correlation to mineral weathering, soil development and vegetation. Especially, taking into account that 90% of suspended matter is redeposited before reaching the outflow, contributing largely to soil formation (Hasholt and Hagedorn, 2000). According to Venkatachalam et al. (2021), metals in soils play an important role in the establishment of microbial communities of the post-glaciated ecosystems, while microbial metabolism of exposed OC plays an important role in nutrient cycling and soil development (Schuette et al., 2010). Therefore, in order to understand soil development, it is important to investigate the soil chemistry along glacier foreland transects.

Further, metal and nutrient fluxes in glacial runoff are also controlled by weathering of bedrock material, as well as re-vegetated lands. Re-vegetation of deglaciated lands affects nutrient and metal concentrations in meltwater streams. For instance, dissolved inorganic nitrogen levels increase as a result of colonization of post-glaciated areas by nitrogen-fixing plants (Hood and Scott, 2008). Meanwhile, bedrock lithology has a direct influence on meltwater chemistry (Ahmad and Hasnain, 2000), bedrock erosion largely controlling concentration of nutrients, dissolved and particulate elements in glacial river runoff (Eiriksdottir et al., 2015). Research on nutrients and some elements in Eastern Iceland revealed that in the past 40 years, fluxes of phosphate and nitrate, major elements such as calcium, magnesium and potassium, and trace elements like aluminium and zinc have substantially increased (Eiriksdottir et al., 2015). As soluble elements are less affected by increased runoff compared to insoluble ones, transport of particulates is higher by a magnitude of 0.5 to 4 than transportation of dissolved elements (Eiriksdottir et al., 2015), implying the increase in insoluble inorganics caused by climate change. Water chemistry of meltwater streams and proglacial lakes was investigated in this study in order to observe differences in study sites.

## **1.6 Choosing study sites**

This study investigates the accumulation of soil derived OC across three catchments: Tarfala valley in Sweden, Vatnajökull ice cap in Iceland and Zackenberg valley in Greenland (Figure 1.3). These catchments have been deglaciated for a different period of time, allowing to gain better understanding of soil OC accumulation in these post-glaciated terrains.



Figure 1.3. Overview map of the Arctic and Subarctic regions (Modified from Ahlenius, 2016). Sampling sites are indicated by blue stars: Tarfala valley, Sweden, Vatnajökull icecap, Iceland and Zackenberg valley, Greenland.

**Tarfala valley, Sweden.** The Tarfala valley has a bare landscape and is the youngest out of the three. Study sites within the valley have different ages of deglaciation. The literature suggests that there was an initial moraine formation at the Storglaciären glacier in 2500 BP (Karlen 1973; Ackert 1984; Etienne et al. 2003 in Tonkin et al., 2016) that was overridden by glacial advance in 1910, which was its most recent maximum (Holmlund et al., 2005). This suggests that despite the presence of older moraines, this landscape has been developing anew only in the past 100 years. Meanwhile, the surroundings of the Tarfalajaure seem to have been ice free for a longer time. Records show that the Kebnepakteglaciären used to calve into the Tarfalajaure lake until 1940s (Svenonius, 1910; Schytt, 1959 in Kirchner et al., 2019), while the banks of the lake were already ice free.

Northern Lapland where the Tarfala valley is situated was deglaciated before 9000-8500 14C years BP (Karlen, 1979), however, higher altitude areas must have deglaciated later than that (Dobiński and Glazer, 2018). Overall, the distal parts of the lake except the lake bathymetry have not been studied in detail (Larje and Nyberger, 1960 in Kirchner et al., 2019), limiting the understanding of how long the surroundings of the lake have been ice free. Permafrost is present in some areas of the Tarfala valley with a continuous permafrost layer at an altitude above 1531 meters, discontinues – 1218 meters and sporadic – 875 meters (Fuchs et al., 2015). Mean soil organic carbon storage in the catchment area of the Tarfala valley is  $0.7\pm 0.2$  kg/m<sup>2</sup> for the top 30 cm and  $0.9\pm 0.2$  kg/m<sup>2</sup> for the top 100 cm (Fuchs et al., 2015). Soil organic matter has developed within the last 100 years and is present only in top layers. Moreover, the study (Fuchs et al., 2015) showed that over 80% of soil organic carbon (SOC) is stored in the top 30 cm of soil and 33% is the topsoil layer. Low SOC values are related to the presence of glaciated areas and bare terrain, steep topography and coarse sediments, shallow soil layer and the lack of peat formation, lack of OM burial by cryoturbation or solifluction (Fuchs et al., 2015). Due to these low values, the area is not considered a future OC source but rather a carbon sink.

**Vatnajökull ice cap, Iceland.** The Vatnajökull ice cap is an older catchment. Soils from the glacier forelands in Iceland have different ages. For instance, the oldest soils from the Svínafellsjökull glacier foreland date to 2500 years (Guðmundsson et al., 2002), while soils from Virkisjökull foreland are 5000-6000 years old (Guðmundsson, 1998), Kvíárjökull – 3200 years (Guðmundsson, 1997; Bennet et al., 2010), Fjallsjökull – 4500s years (Rose et al., 1997), Skaftafellsjökull – less than 100 years (Marren, 2008) and Skeiðarárjökull – about 120 years (Bogacki, 1968). The area has no permafrost, while SOC in the top 10 cm of soil varies from 0.04 (2003 moraine) to 1.10 kg/m<sup>2</sup> (1890 moraine) at Skaftafellsjökull (Vilmundardóttir et al., 2007), and from 0.19 (2016 moraine) to 1.7 kg/m<sup>2</sup> (1895 moraine) at Fláajökull (Wojcik et al., 2020). This value in one of the younger glacier forelands studied in Iceland is higher compared to that in the top 100 cm of soil in Sweden, indicating that OC accumulates faster at the Vatnajökull ice cap catchment compared to the Tarfala valley.

**Zackenbergl valley, Greenland.** The Zackenberg valley is an older catchment compared to study sites in Sweden and Iceland. The Zackenberg valley has been ice free since the Early Holocene (ca. 10.5 ka) (Garcia-Oteyza et al., 2022), which is the longest period of post-

glacial development out of the three catchments. The valley is situated within the area of continuous permafrost, posing a risk of release of carbon stored in permafrost along with soil derived OC. Palmtag et al. (2018) estimated mean SOC in the area to be 4.8 kg/m<sup>2</sup> for the 0 - 100 cm of soil depth, with 13% being stored in the organic layer (77% of which is stored in the active layer).

Since the three catchments have been deglaciated for different periods of time, have different permafrost presence and therefore varying SOC content, these sites are well suited for studying accumulation of soil derived OC on a temporal scale.

## 1.7 Knowledge gaps and research question

Glacier forelands serve as spatial representation of temporal changes in terms of soil OC accumulation. Climate change is accelerating glacier retreat in northern latitudes. For instance, in the period of 2006-2015, Arctic glaciers lost mass at a rate of -213±20 Gt per year, faster than any other period in the past 4000 years (Meredith et al., 2019 and references therein). Glacier retreat consequently leads to accumulation of soil OC. Despite the importance of SOC accumulation in glacier forelands, understanding of the source and nature of OC in three catchments investigated in this study are limited. Microbial succession was not studied along these glacier transects. Moreover, despite the previous research, accumulation of organic biomarkers, BHPs and GDGTs, in post-glaciated areas have not been previously investigated.

Currently, there is a significant knowledge gap because:

1. there are a limited amount of studies of soil development and SOC accumulation in deglaciated areas from a range of regions;
2. although there are estimated SOC stock values, there is no knowledge about the dominant source of soil derived OC;
3. there is no knowledge about microbial colonization and succession along glacier downstream transects investigated in this study;
4. there are no studies linking organic biomarkers and microbial communities in post-glaciated areas.

Therefore, this PhD project focuses on investigating soil derived OC in deglaciated forelands in the Tarfala valley in Sweden, the Vatnajökull ice cap in Iceland and the Zackenberg valley in Greenland.

In order to understand the amount of OC, this study quantifies OC concentrations from meltwater and proglacial lake sediments and soils from glacier forelands in three sites in the Arctic and Subarctic regions. Microbial communities are analysed in order to increase the understanding of OC production and accumulation. Because the dominant source of soil OC in post-glaciated areas is unknown, BHP and GDGT analyses are conducted as part of this project in order to trace sources of SOC in glacier forelands. Coupling the two methods, study of microbial communities and biomarker analysis, is used as a proxy to access living (DNA) and dead (BHPs and GDGTs) microbes. Biomarkers are used to identify terrestrial input in post-glaciated areas. Moreover, because chemical composition of sediments and soils affect which microbial communities are established, analysis to identify major and trace metals is conducted as part of this project.

## 1.8 Aims and objectives

The overall aim of the project is to understand how microbial metabolism and bioavailability of OC affects accumulation of soil OC in three nutrient rich deglaciated catchments (Tarfala in Sweden, Vatnajökull ice cap in Iceland, and Zackenberg in Greenland). To achieve this aim the following sub-aims were set:

1. understand accumulation of soil OC at the Tarfala valley in Sweden;
2. understand accumulation of soil OC in the catchment surrounding the Vatnajökull ice cap in Iceland;
3. understand accumulation of soil OC at the Zackenberg valley in Greenland;
4. compare the three catchments in order to understand the differences and similarities in accumulation of soil derived OC.

The aforementioned aims will be achieved via the following objectives:

1. quantify mass discharge nutrients (ions, dissolved and particulate metals) released from receding Arctic and Subarctic glaciers in this study, via meltwater streams, to understand the difference between and within each catchment;

2. measure metals and nutrients (major and trace elements) in sediments and soils;
3. measure total organic carbon (TOC), total nitrogen (TN) and organic biomarkers (BHPs and GDGTs) in soils from glacier forelands, proglacial lake sediments (where present) and meltwater stream sediments to identify sources of organic matter;
4. characterize the microbial community composition and function within soils and sediments.
5. Based on these aims and objectives this study will test the following hypothesis: Organic biomarkers and microbial analysis can be used to understand accumulation of soil derived OC in newly developed soils following glacier retreat in northern latitudes.

## 1.9 Thesis outline and chapter content

**Chapter 2** describes the methodology followed in this study. Field methods, laboratory, as well as data analysis are explained in detail. The study focuses on organic biomarker, DNA sequencing and metal analyses.

**Chapter 3** presents study sites and water chemistry data. Climate, geological setting, and age of deglaciation are described for each catchment, while sampling locations and sample types are shown in detail. Water chemistry data, including major ions, dissolved and particulate metals, is used to show the differences and similarities between and within the catchments. The following result chapters are aimed at understanding the accumulation of soil derived OC in three catchments by looking at catchment chemistry, organic biomarkers and microbial community along downstream transects away from the glacier front.

**Chapter 4** presents findings at the Tarfala valley in Sweden, **Chapter 5** – the Vatnajökull ice cap in Iceland, and **Chapter 6** – the Zackenberg valley in Greenland. The following objectives were carried out for these results chapters:

1. to determine TOC, C/N and major metals in soils and sediments collected from glacier forelands;
2. to determine concentration and distribution of BHPs and GDGTs and their proxies ( $R'_{\text{soil}}$ , MAT and soil pH) in selected samples along downstream transects away from the glacier front;

3. to determine microbial community structure in selected samples selected from glacier forelands;
4. to establish a relationship between organic biomarker distribution and microbial community composition.

The following outcomes are hypothesized based on the objectives above:

1. TOC and organic biomarkers BHPs and GDGTs, as well as soil marker BHPs, accumulate along the downstream transects;
2. the  $R'_{\text{soil}}$  increases along the transects, indicating accumulation of soil marker BHPs;
3. bacterial diversity increases with the time of exposure of samples and distance from the current glacier front;
4. within each glacier foreland, soils are more abundant in OC, organic biomarkers, and bacteria compared to sediments.

**Chapter 7** aims to bring together and synthesize findings from results chapters 4, 5 and 6.

Comparing the three catchments investigated in this project, the following is expected:

1. Sweden is described by the lowest OC and organic biomarkers concentrations, as well as less diverse microbial community;
2. Iceland has higher OC and organic biomarkers concentrations, and more diverse bacterial community;
3. Greenland has the highest OC and organic biomarkers content, as well as the highest microbial community diversity.

**Chapter 8** summarizes the findings of accumulation of soil derived OC in three catchments studied in this PhD project in terms of organic biomarkers and microbial communities.

Recommendations for future research based on the results of this project are outlined.

## 2 Materials and Methods

The aim of this chapter is to describe field sampling, laboratory and data analyses procedures aimed at collecting and analysing soil, sediment and water samples.

### 2.1 Field Sampling

#### 2.1.1 Preparation for fieldwork

Field methods were tested in the lab prior to fieldwork. Since fieldwork includes a component of filtering meltwater samples, filtering methods were tested. Buchner flask setup (flask with a side arm, rubber tubing to the pump, rubber bung, Buchner funnel) was tested using turbulent water samples from local (Manchester) rivers. Water filtering was tested using different pumps. Using a vacuum hand pump (Mytivac) and double action hand pump (915/2825, Argos, UK) resulted in long hours of filtering, while using electric portable pump (340/1778, Argos, UK) didn't have enough suction power to filter turbulent water samples. Therefore, it was decided to order more powerful electric oil free vacuum pumps. The industrial double piston vacuum pump HC400 (RS Components, UK) was used as the main pump and the VPBW single stage oil free vacuum pump (Hvacstore, UK) as a backup.

Next, consumables such as filter papers, glass vials and foil packets were prepared. Glass microfiber filters (GF/F) (Whatman, Sigma-Aldrich, UK) with a pore size of 0.7  $\mu\text{m}$  and diameter of 47 mm were pre-weighed using an electronic balance at the precision of 0.0001 g. Foil packets for soil samples due to organic compounds analysis were folded and pre-combusted. Consumables were muffle furnace at 450 °C for four hours to remove organics. Mason Jars used to collect ice samples were autoclaved.

Finally, re-usable glassware (500 ml or in 1 l duran™ glass bottles, 500 ml measuring cylinder, flasks with a side arm, Buchner funnel) and metal tools (spoon, trowel, ice axe, tweezers) were thoroughly cleaned using methanol and deionised (DI) water.

### **2.1.2 Fieldwork**

While in the field, GPS coordinates were obtained using Garmin GPSMAP® 64 (Garmin, UK). At each study site, glaciers were visited in order to collect soil samples from sites close to the glacier, as well as older moraines, sediment and water samples from streams and lakes, and surface and basal ice. Throughout this thesis, samples collected from moraines, vegetated areas near and away from glaciers, and water bodies are referred to as soils (both mineral and organic matter present), while particles collected from meltwater streams and proglacial lakes, as well as from unvegetated river and lake banks close to these water bodies are referred to as sediment samples (mineral matter only). A spatial series of samples were collected along the transects from the glacier front to older moraines to track changes in organic carbon, organic biomarkers and microbial communities along the downstream transects. Samples were collected in triplicates in order to ensure precision. Water pH, temperature and conductivity were measured using HI-98129 Pocket EC/TDS and pH Tester (Hanna Instruments), water flow rate used for discharge measurements was measured using a MFP126-S flowmeter (Geopacks) (see Appendix 1 for measurements). In the field, the glassware and metal tools were exposed to the sampling site prior to the sampling.

#### **2.1.2.1 Soil and sediment sampling**

Soil samples were collected along the transect of past glaciation margin from river and lake banks. To understand soil derived OC accumulation at different past glacial positions, soil samples were collected from moraines. Study sites, especially Sweden, have underdeveloped thin soil layers, therefore, some of the soils collected mostly represent an A-horizon (the top layer of a soil profile). More developed soils were collected where possible. The vegetated top layer was removed before taking the samples. Sediment samples were collected from either the bottom of the streams and lakes or, where the bottom was buried under gravel and rocks, from the bank of the water bodies (within 0.5

meter where possible). 200 mg of soil and sediment for organic compounds were collected in pre-combusted foil packets and stored cold to avoid degradation of organic material. About 200-300 mg of soil and sediment samples due to the microbial community analysis were collected in zip-lock bags and stored frozen.

### **2.1.2.2 River and lake sampling**

Water samples were collected either in 500 ml or in 1 L duran™ glass bottles. The bottles were rinsed three times with sample water prior to meltwater sampling. Water samples were stored in the fridge and filtered the same day after the return to the field accommodation/lab in order to prevent microbial transformations. The flowchart in Figure 2.1 illustrates how filtering meltwater samples in the field yielded samples for further analyses. Pre-weighed glass microfiber filter (GF/F) (Whatman, Sigma-Aldrich, UK) was used to capture particulate metals, while 50 ml of filtrate was collected in 50 ml metal free centrifuge tubes (Labcon, USA). 0.22 µm Isopore Millipore membrane filter (Merck) was used to filter meltwater (adapted from Liu et al., 2006) and 2 ml of the filtrate was collected for further ion chromatography analysis. Some water samples in Greenland were partially filtered in the field using syringe filters: 0.7 µm GF/F syringe filter (Whatman, Sigma-Aldrich, UK) was used to collect samples for dissolved metals and 0.2 µm syringe filters (Fisherbrand™, Fisher Scientific, UK) to collect samples for ion chromatography. Filtered water samples and GF/F filters were stored in the fridge.

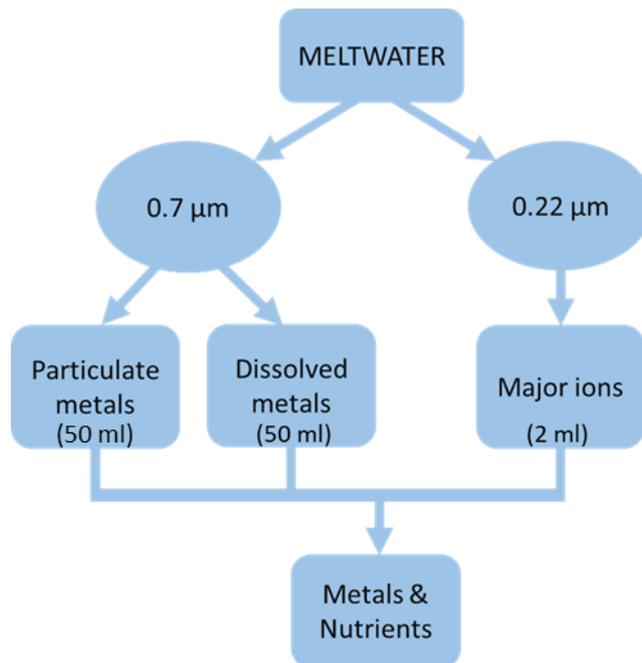


Figure 2.1. Flowchart of filtering meltwater samples prior transportation from the field sites.

### 2.1.2.3 Ice Sampling

During Iceland 2018 and Sweden field campaigns, ice samples were collected in autoclaved Mason Jars using an ice axe. Further, ice was melted at 4 °C, ice sediment settled at the bottom of the jar was collected in foil packets and stored frozen.

### 2.1.2.4 Sample resolution

Sampling locations at each site were chosen to allow understanding of accumulation of soil derived OC along transects away from the glacier front within each catchment. Soil samples were collected to represent accumulation of SOC following glacier retreat from youngest to oldest moraines. Soil samples were collected along downstream transects, such as in Greenland (see section 3.3) and /or from moraines to account for changes in glacier advance and retreat, such as in Iceland (see section 3.2).

Sediment and water samples were collected along downstream transects from a glacier front: meltwater stream draining the glacier if present, proglacial lake if present and a stream/river draining the lake. Water and sediment samples were collected every time a change occurred, such as joining of a new tributary, or meltwater stream/river inflowing/outflowing to/from a lake. One location per such development was sampled in

triplicates to inform of any changes in water chemistry. Due to the length of the transect in Greenland, soil samples were collected in a similar manner next to the water and sediment collection locations where possible (see section 3.3). The exact sampling locations at each catchment are discussed in detail in chapter 3.

## **2.2 Laboratory Analysis**

Laboratory analyses were carried out at Manchester Metropolitan University (MMU), following transportation of the samples back from the field sites. Samples were transported using cool bags packed with ice (gel) packs. Soil, sediment and water samples were analysed to determine organic carbon concentrations, molecular organic biomarkers, elemental and ionic concentrations and DNA sequencing in order to facilitate assessment of how soil develops in deglaciated systems.

### **2.2.1 Soil and sediment samples**

Soil and sediment samples were analysed for organic compounds, microbial community and major metal composition to trace the source of OC (Figure 2.2). Soil and sediment samples for organic compounds and metals were oven dried overnight at 80 °C. Dried samples were sieved through 2 mm sieve and homogenized using a pestle and mortar for further analyses.

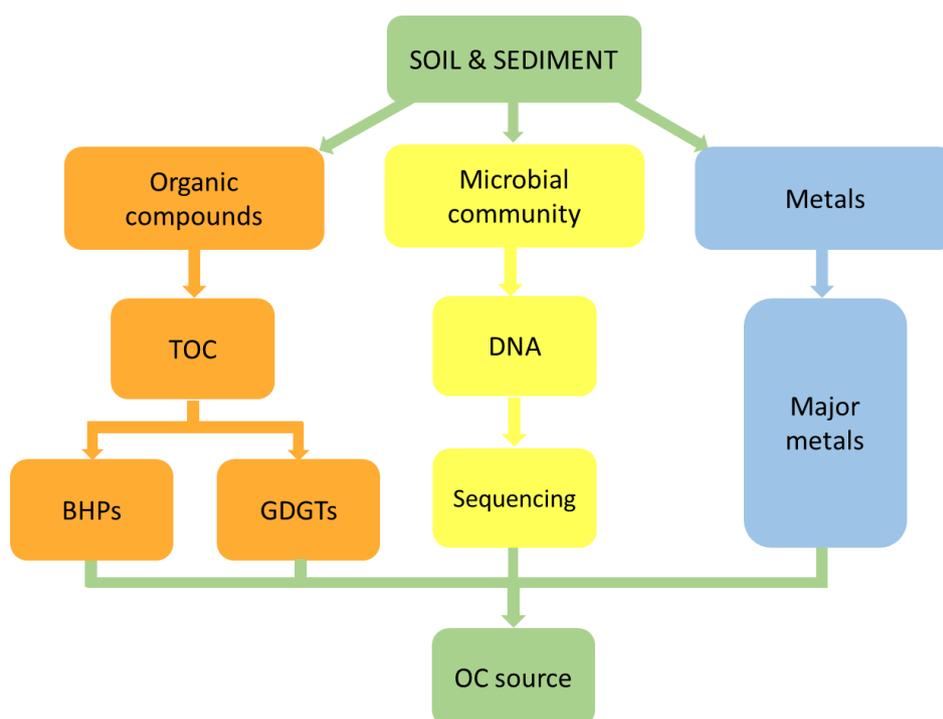


Figure 2.2. Flowchart showing soil and sediment sample analyses to trace OC sources. Orange corresponds to analyses of organic compounds, yellow – microbial communities, blue – metals in soil and sediment samples.

### 2.2.1.1 Organic carbon concentrations in soil and sediment samples

In 2018-2019, total carbon and total nitrogen concentrations were measured using the ‘Leco TruSpec®’ CN analyser. In 2019-2020, a vario EL cube (Elementar) was used to analyse samples for carbon and nitrogen concentrations. ~0.2 g (0.1950 g to 0.2050 g) of the sample was weighed in tin foil in a random order to avoid sample bias. ~0.15 g (0.1490 g to 0.1510 g) EDTA (Ethylenediaminetetra-acetic Acid) 502-092 was used as a calibration standard, where value percentages of carbon is 41.08% and nitrogen – 9.60%. Soil standard (Clay) OAS (Elemental Microanalysis) (C – 2.31%, N – 0.23%) and soil standard (Sandy) OAS (Elemental Microanalysis) (C – 0.83%, N – 0.07%) were used as certified reference materials (CRM).

In order to measure organic carbon concentrations, samples were decarbonated. ~0.5 g of sample was treated with 1 ml of concentrated hydrochloric acid (HCl) (37%; Certified AR for Analysis; Fisher Scientific) diluted with 10.6 ml of DI water and heated for 180 min at 80°C on a hot plate. Further, the sample was cleaned three times with 40 ml of DI water. The dried and re-homogenized sample was analysed using the ‘Leco TruSpec®’ CN analyser

in 2018-2019 and the vario EL cube (Elementar) in 2019-2020 for organic carbon concentrations. ~0.15 g EDTA 502-092 was used as a calibration standard again. Samples with the highest OC concentration were chosen for further biomarker extraction.

#### **2.2.1.1.1 Biomarker extraction**

Biomarkers were extracted from soil and sediment samples with high OC concentration following the protocol described in Tierney et al. (2012), Doğrul Selver et al. (2015), De Jonge et al. (2015) and Pytlak et al. (2021). In detail, 5 grams of soil/sediment sample was combined with 15 ml of Methanol (MeOH): Dichloromethane (DCM) (2:1) solvent and 4ml of Phosphate Buffer. Phosphate buffer was prepared by adding Potassium Dihydrogen Phosphate (KH<sub>2</sub>PO<sub>4</sub>) to 300 ml bi-distilled water to create a 0.05 M solution. The pH of the solution was adjusted to 7.2 using Potassium Hydroxide (KOH) pellets. The sample, treated with MeOH: DCM (2:1) solvent and Phosphate Buffer, was sonicated for 10 min at 40°C and after centrifugation for 5 minutes at 2500 rpm, 5 ml of DCM and 5 ml of Phosphate Buffer were added to the supernatant. The supernatant separated into two layers and the DCM fractions from the bottom of the flask were collected. After re-extracting the sample two more times, all DCM fractions were combined and dried using N<sub>2</sub>. The total lipid extract (TLE) was recovered with DCM: MeOH (2:1) mixture and split into three aliquots: 1 for biomarkers, 1 for glycerol dialkyl glycerol tetraether (GDGTs), 1 for BHPs. The aliquots were dried using N<sub>2</sub>.

#### **2.2.1.1.2 Bacteriohopanepolyol (BHP) separation**

Further, for BHP separation, methods described in Cooke et al. (2009), De Jonge et al. (2016) and Pytlak et al. (2021) were followed. In detail, the aliquot for BHPs was redissolved in 200 µl DCM and passed through a pre-conditioned NH<sub>2</sub> solid phase extraction (SPE) cartridge (Supelclean™ LC-NH<sub>2</sub> SPE Tube, Merck Life Science UK Limited). The cartridge was conditioned using 6 ml of Hexane. The sample was added to the cartridge and 6 ml of Diethyl Ether: Acetic Acid (98:2) was passed through the column yielding a fraction that contains other compounds. Next, 10 ml of MeOH was passed through the column yielding a fraction that contains the BHPs, including soil marker BHPs. Next, the internal standard was prepared by mixing 10.7 mg of 5α-pregnanediol (Tokyo Chemical Industry UK Ltd.) with DCM in a 50 ml volumetric flask. To acetylate the sample, 200 µl of 5α-pregnanediol

standard and 250 µl of Pyridine:Acetic Anhydride (1:1) were added to the dried fraction containing the BHPs. After being re-dissolved overnight, the fraction containing the BHPs was dried using nitrogen again. The sample was redissolved in 500 µl of Propanol:MeOH (60:40) twice and filtered through a 0.2 µm PTFE filter. The dried sample was redissolved in 500 µl Propanol:MeOH ready for injection to the liquid chromatography–mass spectrometry (LCMS).

#### **2.2.1.1.3 Glycerol dialkyl glycerol tetraether (GDGT) separation**

For GDGT separation, methods described in Tierney et al. (2012) and Doğrul Selver et al. (2015) were followed. The aliquot for GDGTs was redissolved in 2ml DCM and passed through a silica column. The silica column was prepared using mineral wool and activated silica. The column was conditioned using 4ml of Hexane:Ethyl Acetate (1:1). The sample was added to the column and 4ml Hexane:Ethyl Acetate (1:1) was passed through the column for core lipid fraction, including GDGTs. Next, 1 µg of a C46 GDGT standard was added to the dried fraction. The fraction was redissolved in 2ml Hexane:Isopropanol 99:1 (v/v) and filtered through a 0.45 µm PTFE filter.

#### **2.2.1.1.4 Liquid chromatography–mass spectrometry (LCMS)**

BHPs and GDGTs were identified and measured using liquid chromatography-mass spectrometry (LC-MS) (Agilent Technologies) equipped with an atmospheric pressure chemical ionization (APCI) source operated in a positive ion mode. LC-MS analysis is a widely used technique due to its high sensitivity and accuracy allowing measuring different compounds during a single analytical run. MassHunter Acquisition Software (Agilent, US) was used to determine BHPs and GDGTs in soil and sediment samples. BHP and GDGT structures were identified using MassHunter Qualitative Analysis Software (Agilent, US). The reproducibility of repeat (duplicate or triplicate) injections on the LCMS was 6% for BHPs and 9% for GDGTs.

The total area of soil marker BHPs, adenosylhopane (m/z 746+788+830), 2-Me-adenosylhopane (m/z 760+802+844), group 3 (m/z 802+844), 2-Me-group 3 (m/z 816+858), group 2 (m/z 761) and 2-Me-group 2 (m/z 775) was calculated to estimate soil marker concentrations showing evidence of terrestrial input. Comparison between the

area of the internal standard 5 $\alpha$ -pregnanediol with a base peak at m/z 345 and the area of individual BHPs was used to access the concentration of each compound.

Total area of brGDGTs: brGDGT Ia (m/z 1022), IIa (m/z 1036), IIIa (m/z 1050), Ib (m/z 1020), IIb (m/z 1034), IIIb (m/z 1048), Ic (m/z 1018), IIc (m/z 1032), and IIIc (m/z 1046) and crenarcheol (m/z 1292) was calculated. Concentration of individual GDGTs was accessed using the area of the C46 GDGT internal standard (m/z 744).

#### 2.2.1.1.5 Biomarker data analysis

BHP based  $R'_{soil}$  index values were calculated as described in Doğrul Selver et al. (2012), using equation (1).

$$R'_{soil} = \frac{G1+G2+G3}{G1+G2+G3+BHT} \quad (1)$$

G1 corresponds to adenosylhopane, G2 – adenosylhopane type 2 and G3 – adenosylhopane type 3.

GDGT proxies, pH and mean annual air temperature (MAT), were calculated as described in De Jonge et al. (2014), using equations (2) and (3), respectively.

$$pH = 7.15 + 1.59 \times \left( \frac{Ic+IIa'+IIb'+IIc'+IIIa'+IIIb'+IIIc'}{Ia+IIa+IIIa} \right) \quad (2)$$

$$MAT = 7.17 + 17.1 \times Ia + 25.9 \times Ib + 34.4 \times Ic - 28.6 \times IIa \quad (3)$$

### 2.2.1.2 Microbial Communities in soil and sediment samples

#### 2.2.1.2.1 DNA extraction

Soil and sediment samples were collected in plastic bags and stored frozen. Genomic DNA was isolated from samples using the DNeasy PowerSoil Kit (Qiagen, Hilden, Germany), following standard procedures outlined in the kit. The Qiagen's DNeasy PowerSoil Kit was suggested as a standardised set of DNA extraction procedures for soil and sediment samples (Lear et al., 2018). In brief, homogenized samples went through steps of cell lysis and inhibitor removal before DNA was captured on a silica membrane, from which it was washed and eluted for downstream applications. In order to ensure sufficient DNA yield, instead of 0.25 g recommended in the procedure, 0.4 g of wet sample was added to a bead

beating tube. Similarly, in step 19 of the kit procedure, instead of 100  $\mu\text{l}$ , 50  $\mu\text{l}$  of Solution C6 was passed through the centre of the white filter membrane to wash and elute captured DNA. Step 19 was repeated twice.

#### 2.2.1.2.2 DNA quantification

The Qubit 2.0 Fluorometer (Invitrogen, Carlsbad, CA 92008 USA) was used to determine the concentration of genomic DNA. Qubit master mix was prepared according to the instructions and readings were performed as described by the provider (Table 2.1). 96 samples were chosen for further downstream analysis based on DNA concentrations.

Table 2.1. Qubit parameters for dsDNA HS

	Component	Concentration, $\mu\text{l}$
<b>Qubit mix</b>	Buffer	199
	Fluorescent dye	1
<b>Standards 1 and 2</b>	Qubit mix	190
	Standards	10
<b>Samples</b>	Qubit mix	198
	Samples	2

#### 2.2.1.2.3 Polymerase chain reaction (PCR)

The polymerase chain reaction (PCR) allowed the amplification of specific DNA fragments and production of large amounts of DNA. DNA was amplified using the Q5 Hot Start High-Fidelity DNA Polymerase (New England Biolabs, Ipswich, MA, USA) following the protocol provided by the manufacturer to target V4 region (Illumina, 2016). Six PCR series were run modifying the initial conditions to test and standardise the amplification parameters following concentrations provided in Table 2.2. All components were defrosted and mixed prior to use, while Q5 Hot Start High-Fidelity DNA Polymerase was stored in ice to avoid inhibition.

Table 2.2. PCR conditions: Master mix and DNA concentrations.

Component	Master mix 1	Master mix 2	Master mix 3	Master mix 4	Master mix 5	Master mix 6
	25 $\mu\text{l}$ Reaction	20 $\mu\text{l}$ Reaction				
<b>5X Q5 Reaction Buffer</b>	5 $\mu\text{l}$	4 $\mu\text{l}$				
<b>10 mM dNTPs</b>	0.5 $\mu\text{l}$	0.4 $\mu\text{l}$				

<b>10 <math>\mu</math>M Forward Primer</b>	1.25 $\mu$ l	1 $\mu$ l				
<b>10 <math>\mu</math>M Reverse Primer</b>	1.25 $\mu$ l	1 $\mu$ l				
<b>Sample DNA</b>	2 $\mu$ l	5 $\mu$ l				
	pure sample	pure sample	pure sample	1:2 dilution	1:10 dilution	pure sample
<b>Q5 Hot Start High-Fidelity DNA Polymerase</b>	0.25 $\mu$ l	0.2 $\mu$ l				
<b>5X Q5 High GC Enhancer (optional)</b>	---	---	5 $\mu$ l	5 $\mu$ l	5 $\mu$ l	---
<b>Nuclease-Free Water</b>	to 25 $\mu$ l	to 20 $\mu$ l				
<b>Number of cycles</b>	35	25	25	25	25	25

The initial run was set to 35 cycles to ensure the full amplification of DNA fragments. The PCR was then tested and based on successful results, approved to be set to 25 cycles. To the first five runs, 23  $\mu$ l of master mix and 2  $\mu$ l of sample (including dilutions) were added for a final volume of 25  $\mu$ l. To the sixth run, 15  $\mu$ l of master mix and 5  $\mu$ l of sample were added for a final volume of 20  $\mu$ l (Table 2.2). Next, the PCR tubes were transferred to a SimpliAmp Thermal Cycler (ThermoFisher Scientific, Waltham, MA, USA) for the amplification run following thermocycling conditions provided in Table 2.3.

Table 2.3. Thermocycling Conditions for PCR.

<b>Step</b>	<b>Temperature, <math>^{\circ}</math>C</b>	<b>Time, minutes</b>
Initial Denaturation	98	2
Amplification, 25 / 35 Cycles	95	20
	65	15
	70	20
Final Extension	72	5
Hold	4	

#### 2.2.1.2.4 Gel electrophoresis

In order to ensure successful amplification, PCR products were analysed using agarose gel (2%) electrophoresis. Gel concentration to prepare 2% agarose gel, loading conditions and run conditions are described in detail in Table 2.4.

Table 2.4. Gel electrophoresis conditions.

<b>Gel conditions</b>	
Tris/Borate/EDTA (TBE) buffer	150 ml
Agarose amount	3 g
<b>Loading conditions</b>	
Orange G loading gel 2X	5 $\mu$ l

Sample volume	10 $\mu$ l
<b>Running conditions</b>	
Voltage	80 V
Time	2 h

### **2.2.1.2.5 Sequencing and data analysis**

After passing quality check at MMU, 97 samples selected for downstream applications and three procedural blanks (kit negative, kit positive and PCR negative) were sent to the NERC Biomolecular Analysis Facility at the University of Liverpool for sequencing of the V4 region of the 16S rRNA gene using the Illumina<sup>®</sup> sequencing platform (Illumina, San Diego, CA, United States), thus characterizing the microbial communities.

Sequencing data were processed using the QIIME2 software package version 2020.8 (Bolyen et al., 2019). Raw amplicon sequencing data was processed using the DADA2 pipeline to create amplicon sequence variants (ASVs) (Callahan et al., 2016). Sequences below 220 bp in length, and those with an average quality score below 30 on a window of 20 bases were discarded. ASVs were assigned to taxa using the GreenGenes database (DeSantis et al., 2006).

Alpha diversity (Shannon) and richness (Chao1) were calculated and plotted using the ‘phyloseq’ package (v1.36.0) running on R 4.10R. Family- and phylum-level bacteria were plotted using Microsoft Excel, while non-metric multidimensional scaling (NMDS) ordination plots of family-level bacterial communities were plotted using the ‘vegan’ package (v2.5-7) running on R 4.10R.

### **2.2.1.3 Metals in soils and sediments**

#### **2.2.1.3.1 Acid digestion of soils and sediments for total metal analysis**

Soil and sediment samples were analysed for Aluminium (Al), Arsenic (As), Cadmium (Cd), Calcium (Ca), Chromium (Cr), Cobalt (Co), Copper (Cu), Iron (Fe), Lead (Pb), Magnesium (Mg), Manganese (Mn), Molybdenum (Mo), Nickel (Ni), Phosphorous (P), Potassium (K), Sodium (Na), Sulfur (S) and Zinc (Zn). Microwave-assisted nitric acid digestion steps described in Niepsch (2019) were followed for acid digestion of soil and sediment samples. First, the PTFE microwave digestion vessels were pre-cleaned. The vessels were filled with 5 ml nitric acid (68%; Primar Plus-Trace Metal Analysis grade; Fisher Scientific) and 5 ml

ultrapure water (18.2 M $\Omega$ ) and processed using CEM Mars Xpress 5 microwave on a cleaning cycle. The vessels were rinsed twice with ultrapure water and dried prior further use. Next, 0.5 g of dried (at 80 °C overnight), sieved (2 mm) and homogenized sample was weighed in pre-cleaned PTFE microwave digestion vessels, acidified by adding 8 ml aqua regia (HCl (37%; Certified AR for Analysis; Fisher Scientific):HNO<sub>3</sub> (68%; Primar Plus-Trace Metal Analysis grade; Fisher Scientific), 3:1) and 2 ml ultrapure water (18.2 M $\Omega$ ), and digested using the microwave-digestion programme detailed in Table 2.5. In order to account for any contamination during the acid digestion steps, procedural blanks were prepared using the abovementioned methodology. Acid digested samples were gravity filtered through Grade 540 Whatman hardened ashless filter paper (110 mm in diameter) (Sigma-Aldrich) into pre-cleaned 50 ml volumetric flasks. Filtrates were made up to volume with ultrapure water (18.2 M $\Omega$ ) and transferred to 50 ml metal free centrifuge tubes (Labcon, USA).

Table 2.5. Microwave (CEM Mars Xpress 5) digestion programme.

Steps	Temperature, °C	Time, minutes	Power, W
Temperature ramp up	90	10	
Microwave digestion	90	5	600
Temperature ramp up	170	10	
Microwave digestion	170	10	1200
Cool down phase		30	

### **2.2.1.3.2 Major metal concentrations using Induced Coupled Plasma – Optical Emission Spectroscopy (ICP-OES)**

Digests were analysed for major metal concentrations using an Induced Coupled Plasma – Optical Emission Spectroscopy (ICP-OES) (Thermo Scientific iCap 6000 series).

An ESSLAB-910B (Inorganic Ventures) was used as a calibration standard. For calibration, different concentrations of calibration standards were prepared (Appendix 2). 1 in 1250 dilution of calibration standard was used as a check standard (CS) ran every 10 samples. 5 procedural blanks were analysed at the beginning of the analytical run. The mean of procedural blanks were subtracted from the main measurements in order to ensure that contamination from the equipment as well as sampling and sample preparation methods are accounted for.

## 2.2.2 Water analysis

### 2.2.2.1 Major ions using ion chromatography (IC)

Water samples were analysed by ion chromatography (IC) using the Thermo Scientific - ICS5000. IC is a technique used to analyse the concentration of negatively (anions) and positively (cations) charged ions based on their retention time when passing through the column. The method is quick and allows analysing a large number of samples in a short time (Dodds et al., 2020) and more environmentally friendly compared to some other standard methods such as Mohr's method and flow methods (Michalski and Pecyna-Utylska, 2021).

In order to assess accuracy of the data the SUPER-05 CRM from Environment and Climate Change Canada was used. The Dionex™ Seven Anion Standard (Thermo Scientific) and Dionex™ Six Cation-II Standard (Thermo Scientific) were used as anion and cation calibration standards accordingly. Details of specifications of the CRM and the calibration standards can be seen in Appendix 3. Different dilutions of calibration standards were used to calibrate the equipment at the beginning of each analytical run (Appendix 4). 1 in 10 dilution of calibration 5 was used as a check standards (CS) throughout the analytical batches. Moreover, in order to account for field methodology errors filtered DI water was run as a sample blank.

### 2.2.2.2 Dissolved metals analysis using ICP-OES

Water samples were filtered through 0.7 µm GF/F (Whatman, Sigma-Aldrich, UK) immediately after sampling. The filtrate was collected in 50 ml metal free centrifuge tubes (Labcon, USA) and stored at 4 °C. Prior to analysis samples were acidified with 1% (v/v) nitric acid (HNO<sub>3</sub>). Dissolved metals were measured using the ICP-OES (Thermo Scientific iCap 6000 series).

In order to assess accuracy of the data SUPER-05 CRM (Environment and Climate Change Canada) was used. Acidified CRM samples were measured every 20 samples. ESSLAB-910B (Inorganic Ventures) was used as a calibration standard. For calibration, different concentrations of calibration standards were prepared (Appendix 5). 1 in 1250 dilution of

calibration standard was used as a check standard (CS) every 10 samples. 5 procedural blanks were analysed at the beginning of the analytical run.

### **2.2.2.3 Particulate metals**

Water samples were filtered through pre-weighed 0.7  $\mu\text{m}$  GF/Fs (Whatman, Sigma-Aldrich, UK) immediately after sampling. The filters were dried at 105 °C overnight and reweighed. Next, PTFE microwave digestion vessels were pre-cleaned according to the steps described in section 2.2.1.3.1. Further, following the acid digestion steps in section 2.2.1.3.1, the filters were digested in the CEM Mars Xpress 5 microwave using aqua regia (HCl (37%; Certified AR for Analysis; Fisher Scientific):HNO<sub>3</sub> (68%; Primar Plus-Trace Metal Analysis grade; Fisher Scientific), 3:1) and gravity filtered through Grade 540 Whatman hardened ashless filter paper (110 mm in diameter) (Sigma-Aldrich). The filtrate made up to 50 ml volume using ultrapure water (18.2 M $\Omega$ ) was analysed on the ICP-OES according to the steps described in section 2.2.2.2.

### **2.2.2.4 Water sampling data analysis**

The mean of procedural blanks were subtracted from the main measurements in order to ensure that contamination from the equipment, sampling and sample preparation methods are accounted for.

## **2.3 Overall data analysis and processing**

Throughout the thesis, data is presented as a mean  $\pm$  one standard deviation (SD) of samples by country, transect or triplicate samples collected in the radius of 0.5 to 1 m. Large SD are indicative of environmental variations within catchments, transects or sample triplicates. While this descriptive statistic is not reliable when showing data for few samples (where  $n=3$ ), it was chosen to provide a better understanding of the range of data. Presenting findings on few sampling points (as little as  $n=2$ ) as mean  $\pm$  one SD has been observed in other studies (i.e., Agnelli et al. (2021); Yu et al. (2010)). Mean and standard deviation were calculated using Microsoft Excel.

OC concentrations were plotted against a Google Earth Pro 7.3.4.8248 map with sampling locations to visualize how OC develops away from the glacier margin. Metal concentrations,

OC concentrations, individual and total BHP and GDGT concentrations were plotted along downstream transects using Microsoft Excel.

Principal component analysis (PCA) of metals and carbon in soil and sediment samples and water data including anions and cations and dissolved and particulate metals in water samples was performed using R 4.10R. Since datasets (OC, organic biomarkers, metals in soils and sediments and ions, dissolved and particulate metals in water samples) are non-normally distributed and there is difference between group variations, a non-parametric multivariate statistical test, permutational multivariate analysis of variance (PERMANOVA), was used to analyse the data. PERMANOVA was performed using the 'vegan' package (v2.5-7) running on R 4.10R.

### 3 Study area

This project covers three sites: Tarfala valley in Sweden (67°55'N, 18°35'E), Vatnajökull icecap in Iceland (64°00'N, 16°38'W) and Zackenberg valley in Greenland (74°30'N, 20°30'W) (Figure 1.3).

Soil, river and lake sediment, water and ice samples were collected during field trips to Sweden, Iceland and Greenland (Table 3.1). Samples were collected in triplicates in order to ensure precision. At study sites in Sweden and Iceland, several glaciers were visited in order to observe differences and similarities between glacier foreland transects in close proximity.

Table 3.1. Summary of the number of samples collected during each field season.

Sample type	Sweden, August 2018	Iceland, May-June 2018	Iceland, September 2019	Greenland, August 2019
Soil	8	5	20	13
Sediment	25	28	9	12
Meltwater from streams, rivers and proglacial lakes	25	28	11	15
Basal ice (ice at the glacier base)	2	2	-	-
Supraglacial ice	1	2	-	-

#### 3.1 Field site – Sweden

The first study site of this project was the Tarfala valley in Sweden. The main transects<sup>1</sup> at the Tarfala valley were Tarjalajaure, including inflows from Kebnepakteglaciären and

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<sup>1</sup> Throughout this study, a path along a glacier foreland along which samples are collected: from the youngest samples at the foot of a glacier to the oldest samples away from the glacier front, are referred to as a transect.

Sydöstra Kaskatjåkkoglaciären, Storglaciären and Isfallsglaciären (Figure 3.1). Glaciers are located in the Tarfala valley on the east of the Kebnekaise massif, northern Sweden.



Figure 3.1. Tarfala valley glaciers, south to north: Storglaciären, Isfallsglaciären, Kebnepakteglaciären and Sydöstra Kaskatjåkkoglaciären (Modified from Google Earth, 2012).

The Tarfala valley is located on the eastern side of the Kebnekaise Massif, Swedish Lapland and has a catchment of 21.7 km<sup>2</sup>, 30% of which is covered by glaciers (Dahlke and Lyon, 2013). The catchment is located in the discontinuous permafrost zone with elevation varying from 980 to 2097 m a.s.l. (Dahlke and Lyon, 2013). The bedrock geology of the Tarfala valley consists of hard, crystalline mafic rocks belonging to the Svea Nappe Complex of the Scandinavian Caledonides (Pomeroy, 2013) and is mostly represented by quartz- and feldspar-rich gneiss, and both amphibolites and granitoids can be found down the valley, the main ones being the Tarfala amphibolite, the Storglaciären gneiss and the Kebne dyke complex (Baird, 2005) (Figure 3.2). Bedrock is exposed at the margins of Isfallsglaciären and Kaskatjåkkaglaciären and some eroded sections at Storglaciären and Kaskatjåkkaglaciären, while most of the valley has sediment cover (Pomeroy, 2013). The valley has a sparse vegetation with shallow soil covering the bedrock and glacial till (Dahlke and Lyon, 2013). Regional climate is mostly continental with prevailing westerly winds (Holmlund et al., 2005). Similar to other Arctic glacier catchments, Tarfala valley experiences snow and ice melt from June to September (Dahlke and Lyon, 2013). The mean annual temperature

at the Tarfala valley is around  $-3.5\text{ }^{\circ}\text{C}$  (1965-2011), while mean annual precipitation was approximately 450 mm/year in 1997 (Ingvander et al., 2013).

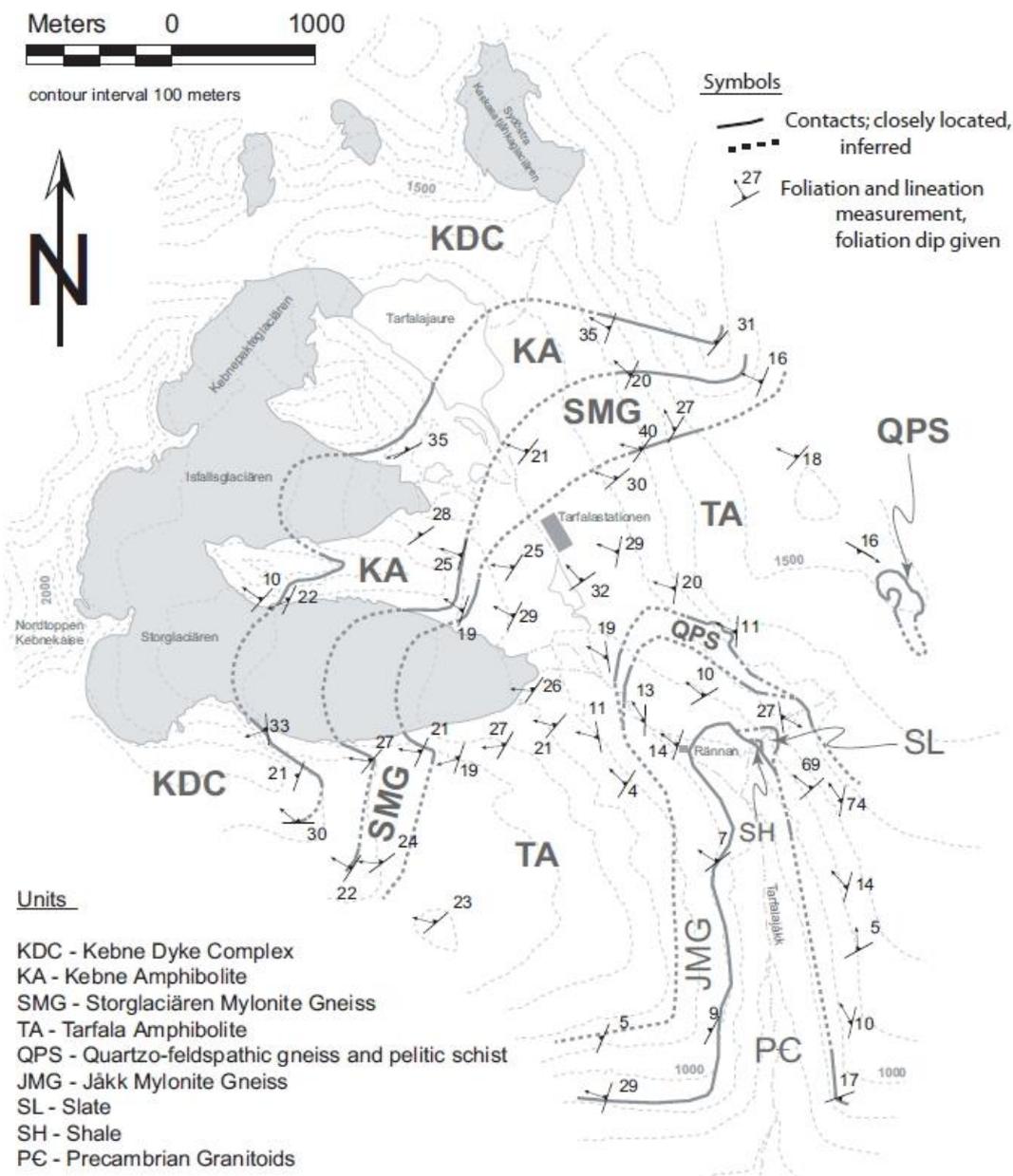


Figure 3.2. Bedrock geology of the Tarfala valley (Adapted from Baird, 2004).

Overall, eight soil samples, 25 water and sediment samples and three ice samples were collected from Tarfala valley for further analysis (Table 3.1, Appendix 6). Soil, river and lake sediment and water samples were collected in triplicate.

### 3.1.1 Tarfalajaure sampling sites

Sampling locations at each site were chosen to allow understanding of soil OC accumulation within each catchment. The Tarfalajaure transect (Figure 3.3) was the main study site investigated at Tarfala valley. The transect included samples along Tarfalajaure lake, Tarfalajåkk river and meltwater streams from the Kebnepakteglaciären and Sydöstra Kaskatjåkkogglaciären glaciers that inflow to Tarfalajaure lake. The Tarfalajaure lake is one of the deepest lakes in the Swedish Arctic with the maximum depth of almost 50 m (Kirchner et al., 2019). 25% of the lake catchment is covered by Kebnepakteglaciären and Sydöstra Kaskatjåkkogglaciären, as well as Sydvästra Kaskatjåkkogglaciären and Sydöstra Kaskasapakteglaciären glaciers (Kirchner et al., 2019). Overall, glaciers at the Kebnekaise massif, including the ones at the Tarfala valley reached their last maximum in 1910 (Karlén, 1973). Kebnepakteglaciären reached its maximum in 1910 and terminated in the lake till mid 1940s (Kirchner et al., 2019). Tarfalajåkk river drains Tarfalajaure lake, meltwater streams from Storglaciären and Isfallsglaciären join the river down the valley.

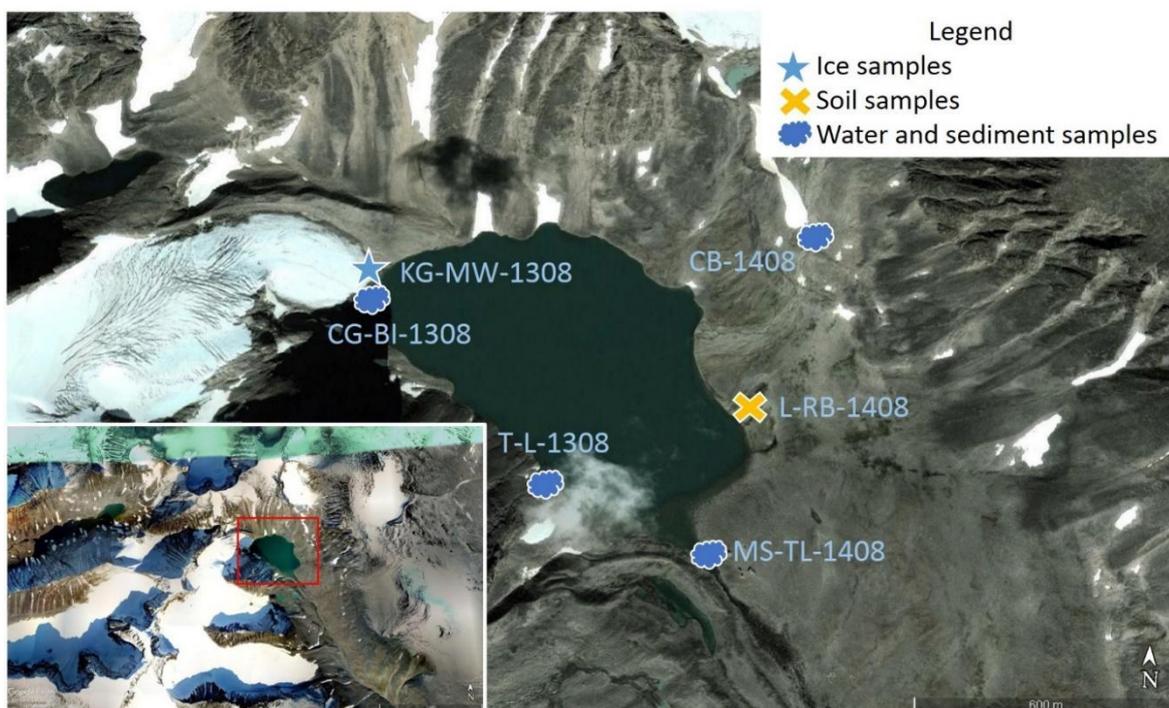


Figure 3.3. Tarfalajaure transect soil, river and lake sediment, water and ice sampling sites at the (Modified from Google Earth, 2012).

Soil, river and lake sediment and water samples were collected along the Tarfalajaure transect, including surroundings of Tarfalajaure lake, a proglacial lake in front of

Kebnepakteglaciären, Tarfalajåkk river and glacial systems up the valley, Kebnepakteglaciären and Sydöstra Kaskatjåkkogglaciären (Figure 3.3 and Figure 3.4). Basal ice samples were also collected from Kebnepakteglaciären.

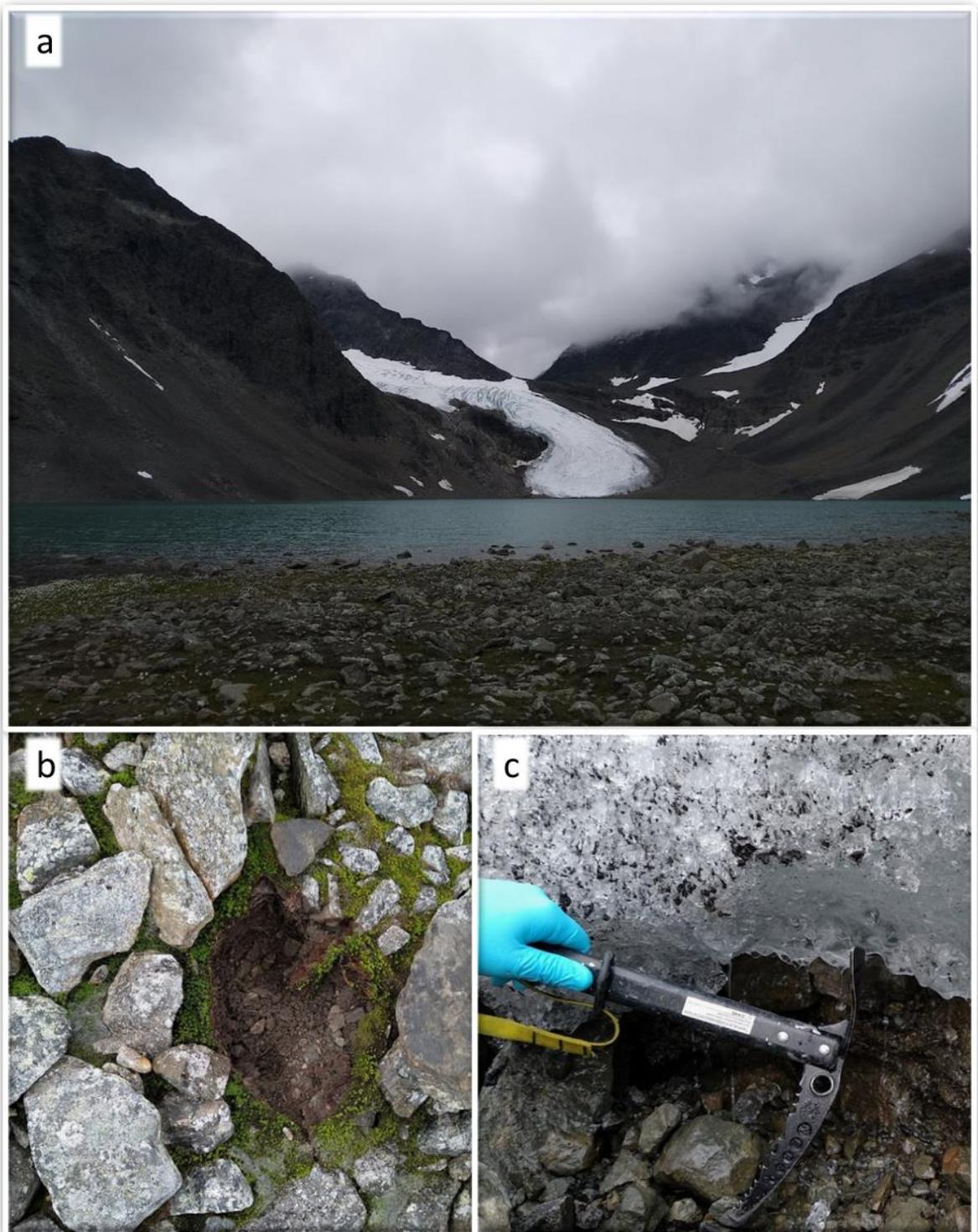


Figure 3.4. Photos from the Tarfalajaure transect: (a) overview of the Tarfalajaure lake and Kebnepakteglaciären glacier; (b) collecting a soil sample after removing rocks and plant material; (c) collecting a basal ice sample.

### 3.1.2 Storglaciären sampling sites

Storglaciären is a predominantly warm polythermal valley glacier, with a cold ice at the near-surface area of the ablation zone (Benn and Evans, 2010). Storglaciären is the most studied glacier in the world (Holmlund, 2016). First mass balance studies of the glacier were conducted in 1945-1946, while the glacier front was first surveyed in 1897 (Holmlund, 2016). Despite being the most studied glacier in the world, literature indicates that soil development in terms of organic biomarkers and microbial diversity have not been previously studied at Storglaciären.

In the past, the glacier used to be drained by two meltwater streams Nordjåkk (north) and Sydjåkk (south). However, the northern stream has disappeared and instead there is a main stream draining the glacier in its central part. The glacier is now drained both by the main stream in the middle of the glacier, and by a smaller Sydjåkk stream at the south of the glacier margin (Figure 3.5). The topography in front of the glacier is steep affecting water turbidity. The glacier has been retreating since its maximum in 1910 (Holmlund et al., 2005) with the exception of a short period of advance from late 1980s to 1996 (Holmlund, 2016) (Figure 3.6).

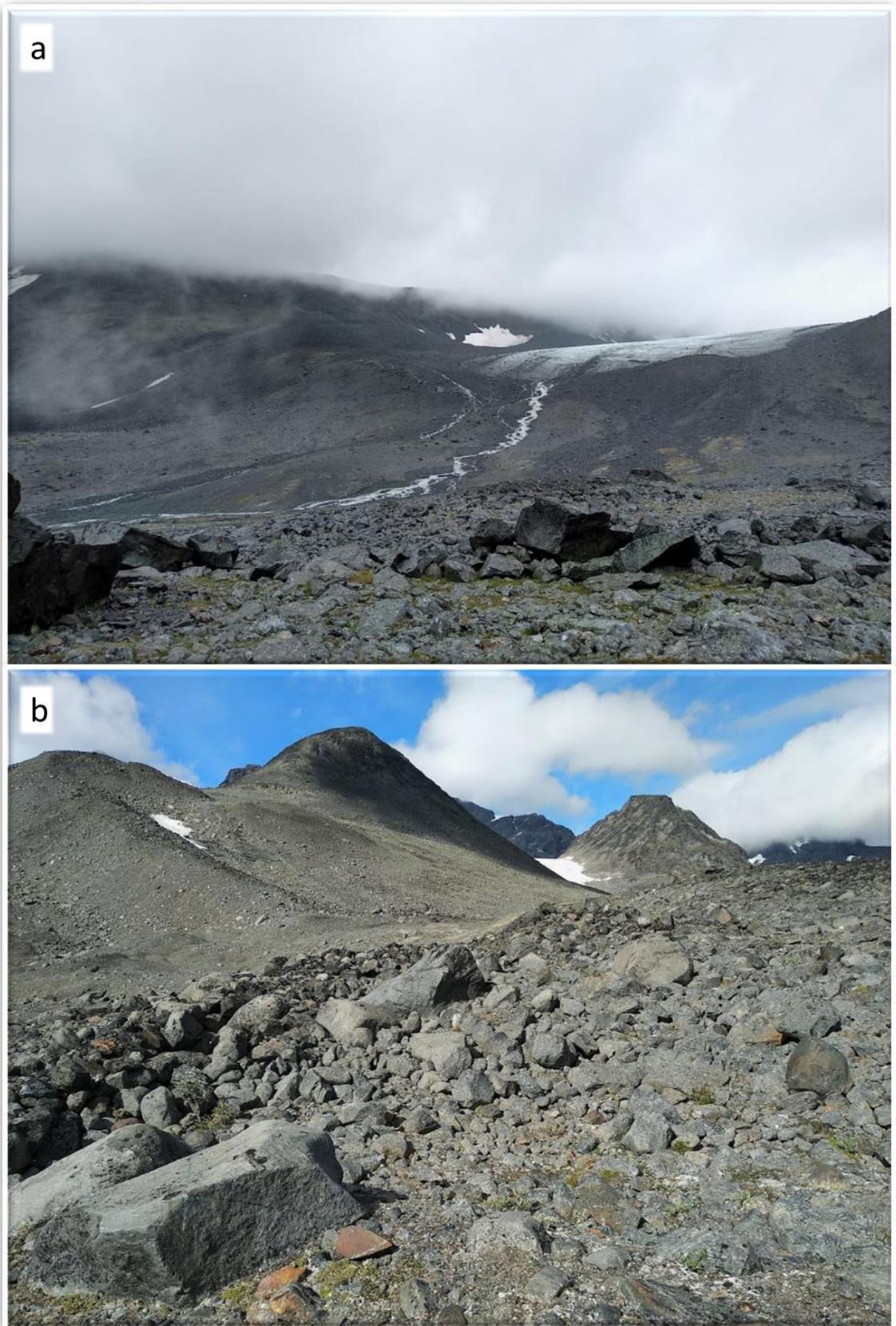


Figure 3.5. (a) Overview of the Storglaciären transect; (b) moraines at the Storglaciären foreland.

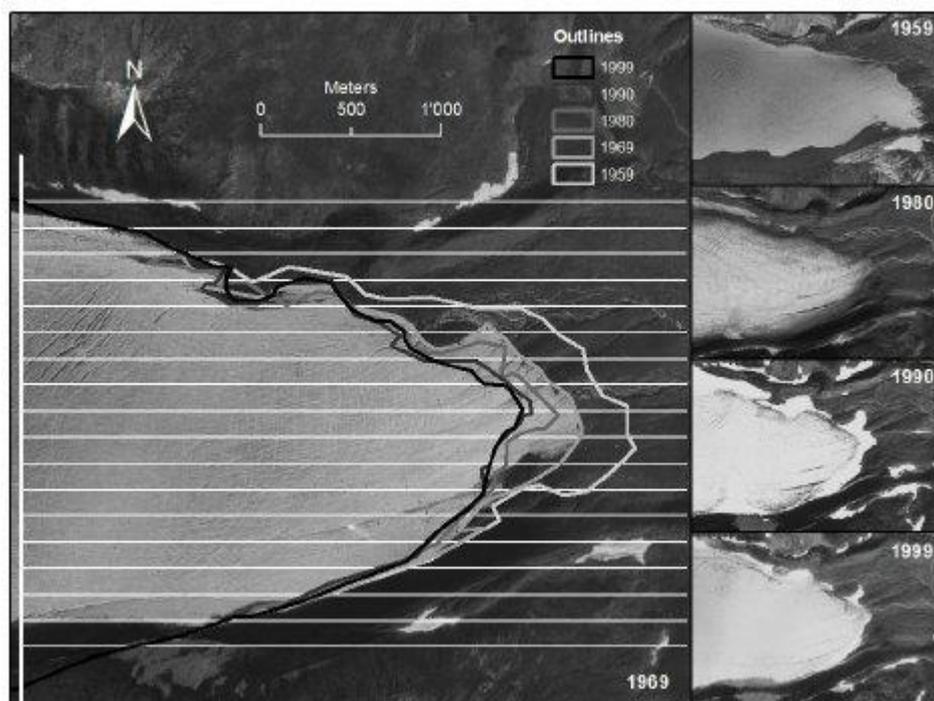


Figure 3.6. Recession of the glacier front of Storglaciären, 1959-1999 (Koblet et al., 2010).

Figure 3.7 shows the Storglaciären sampling sites. Soil samples were collected from moraines along a transect perpendicular to the meltwater streams. Samples were taken from the moraines to the north of the meltwater streams. Water and river sediment samples were collected along a transect starting from the glacier front and moving downstream. Samples were taken from each meltwater stream, as well as downstream from the confluence of two streams and downstream from the Tarfalajåkk river confluence, (the main river running through the Tarfala valley).

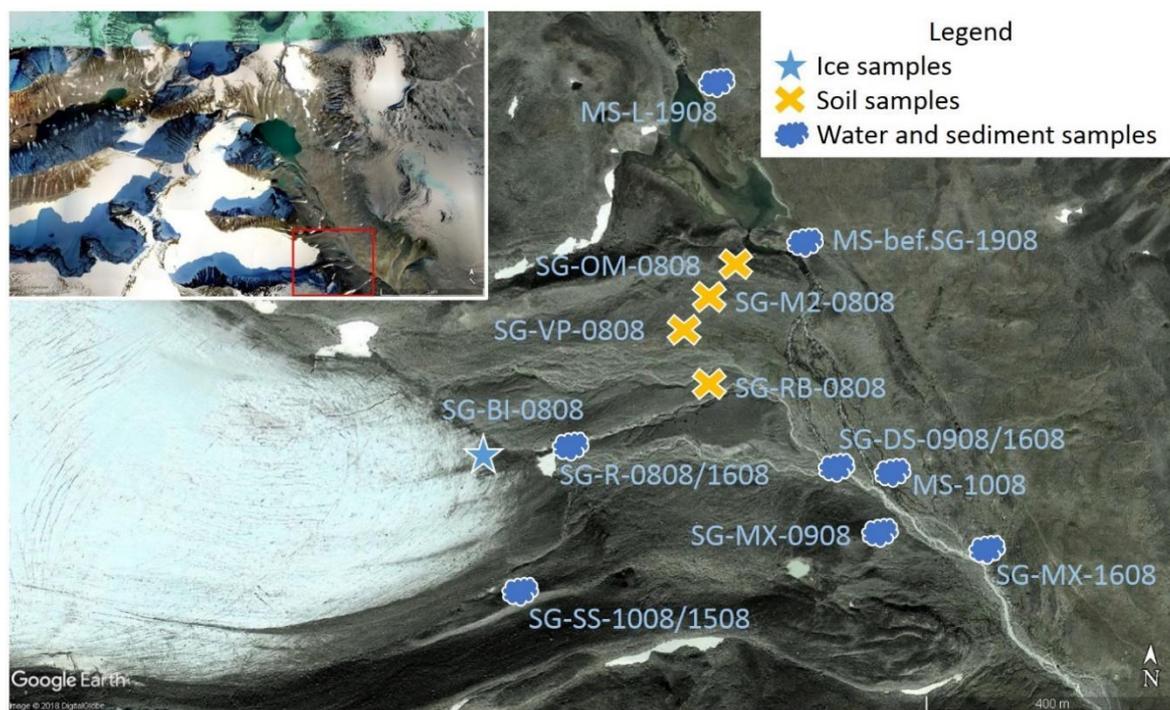


Figure 3.7. Storglaciären sampling sites (Modified from Google Earth, 2012).

### 3.1.3 Isfallsglaciären sampling sites

Isfallsglaciären is a small polythermal glacier (Ely et al., 2017). Similar to Storglaciären, Isfallsglaciären has receded at least 500 m since the 1920s (Eklund and Hart, 1996). Glacial advance in the 18<sup>th</sup> century formed an end moraine in front of the glacier (Eklund and Hart, 1996). Overall, the response time of glaciers to climate change in Sweden is 50 to 100 years, making them good climate indicators of present and past changes in temperature (Holmlund, 2016).

Figure 3.8 shows the sampling sites at Isfallsglaciären. Soil samples were collected from moraines in front of the glacier (IG-LM-1208). Two meltwater streams drain Isfallsglaciären from south and north before inflowing into a lake drained by a main meltwater stream. Another lake is formed in front of the glacier, also fed by the meltwater streams. Water and river and lake sediment samples were collected from these water bodies.

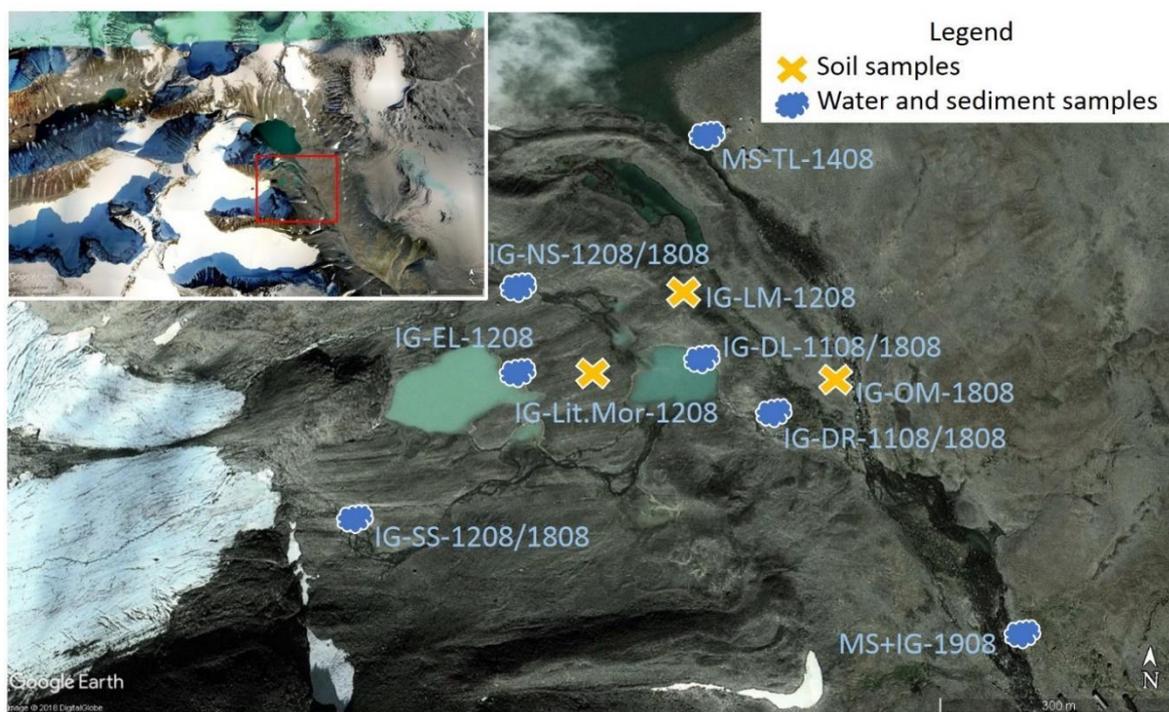


Figure 3.8. Isfallsglaciären sampling sites (Modified from Google Earth, 2012).

## 3.2 Field site – Iceland

Iceland is a Nordic island country, much of which is in the Subarctic, with northernmost parts being in the Arctic Circle. 11% of Iceland comprises glaciers, with Vatnajökull ice cap situated in south-east Iceland being the largest one (Björnsson and Pálsson, 2008). The ice cap partly lies in the active volcano zone, western part overlaying porous lava beds, while eastern part overlays consolidated till (Schmidt et al., 2019). Due to the volcanic nature of the area most of the bedrock underlying the glaciers are comprised of basaltic lava and hyaloclastite, while tephra fallout can be found in proglacial areas (Roberts and Guðmundsson, 2015) (Figure 3.9). Southern side of the Öræfajökull ice cap is younger compared to the northern part of the massif. North of Svínafellsjökull and Fjallsjökull consists of rocks from the Matuyama magnetic chron (2.58–0.78 Ma) or older, while to the south the rocks are normally magnetized rocks from the Brunhes chron (<0.78 Ma) (Roberts and Guðmundsson, 2015). Lower slopes of the ice cap consist of hyaloclastites and lava flow (Roberts and Guðmundsson, 2015). Rhyolite formations are present at margins of the ice cap (see Figure 3.9), including north of the Kvíárjökull. Holocene sediments and tephra lie in glacier forefields investigated in this study. Tephra layers, volcanic ash and dust storms darken the surface of the glacier ice and increases melting.

As for the climate, south Iceland has mild winters and cool summers with small seasonal variations in temperature: an annual average of 5 °C at Svínafellsjökull (Vilmundardóttir et al., 2014) and around 5.5 °C at Virkisjökull (MacDonald et al., 2016). Due to the steep mass balance gradients Icelandic glaciers get a lot of mass input but have high mass loss at their termini driving rapid mass turnover (Benn and Evans, 2010). Moreover, most of the glaciers in Iceland are predominantly temperate in nature (Benn and Evans, 2010). Since the end of the 19<sup>th</sup> century, Vatnajökull icecap has lost over 10% of its total volume (Björnsson and Pálsson, 2008). Mass balance models suggest that by 2060 the volume of Vatnajökull will have decreased by 25% (Björnsson and Pálsson, 2008).

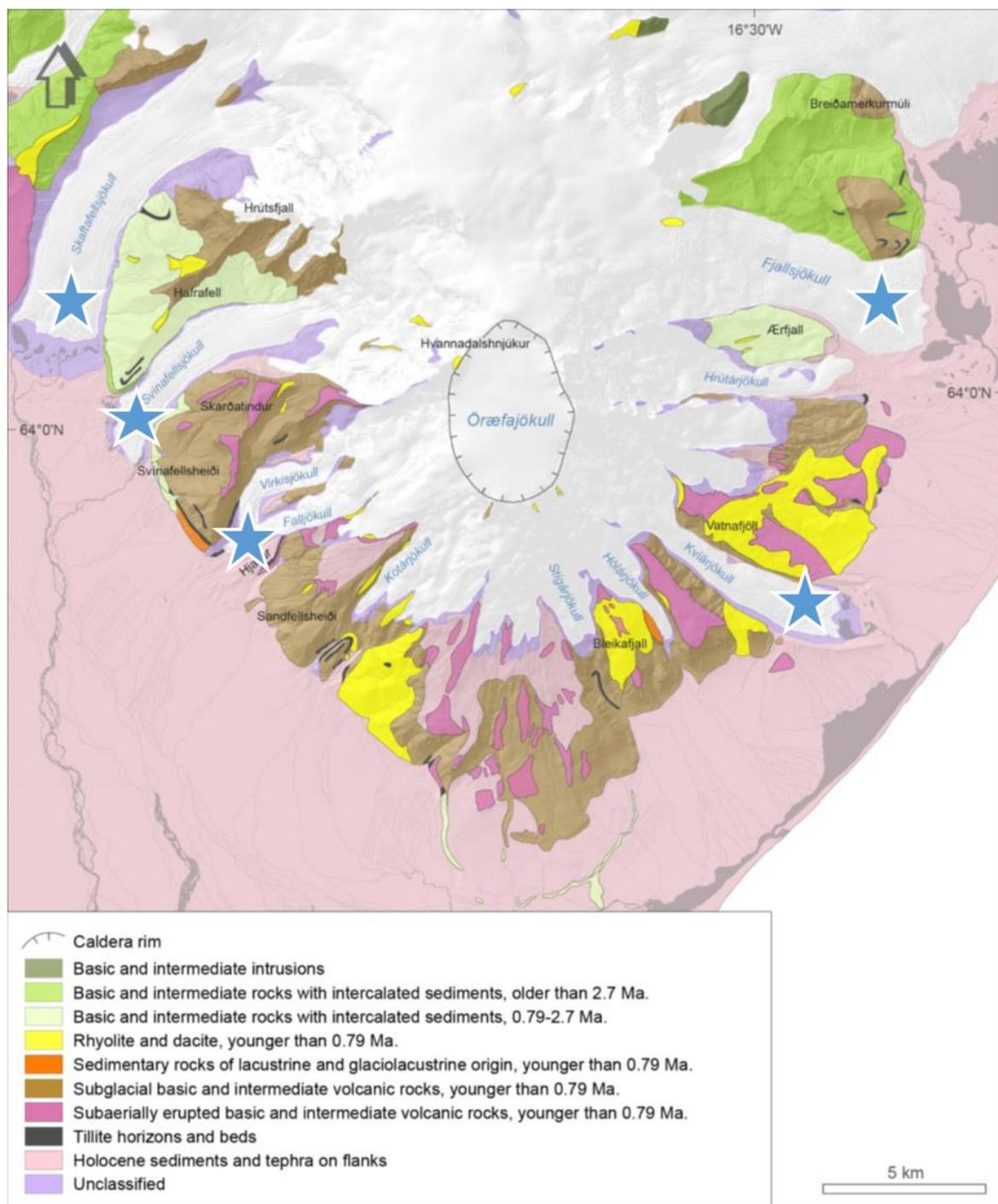


Figure 3.9. Geological map of Virkisjökull icecap (Adapted from Torfason, 1985). Glaciers investigated in this study are indicated by blue stars. Note: Skeiðarárjökull is to the left of Skaftafellsjökull and not shown in the map.

Six glaciers (Skeiðarárjökull, Skaftafellsjökull, Svínafellsjökull, Virkisjökull, Kviarjökull and Fjallsjökull) from the Vatnajökull ice cap were investigated in this study (Figure 3.10). The glaciers are located in the south margin of the ice cap and within the boundaries of the Vatnajökull National Park, established in 1998. Svínafellsjökull and Virkisjökull were studied

in detail with sample collection campaigns in 2018 and 2019. Some water samples were also collected in 2018. In 2019, soil and sediment samples were collected from Kviarjökull and Fjallsjökull. Fewer samples were collected from the Skaftafellsjökull and Skeiðarárjökull due to the size of glaciers and time restrictions.



Figure 3.10. Study sites in Iceland: Skeiðarárjökull, Skaftafellsjökull, Svínafellsjökull, Virkisjökull, Kviarjökull and Fjallsjökull glaciers, Vatnajökull ice cap (Modified from Google Earth, 2012).

### 3.2.1 Sampling sites, 2018 and 2019

#### 3.2.1.1 Svínafellsjökull sampling sites

Svínafellsjökull (63°59' N, 16°52' W) is a temperate outlet glacier. Svínafellsjökull has retreated by 800 metres since the LIA maximum (Lee, 2016). Major glacier advances were recorded approximately 2500 years ago, and during the LIA (Björnsson and Pálsson, 2008; Lee, 2016). For example, the outermost moraine of Svínafellsjökull, Stóralda, was dated to be 2500 years old (Guðmundsson et al., 2002). During the LIA, some glaciers advanced by 10 to 15 km (Björnsson and Pálsson, 2008). Since the LIA, the glaciers in the area have shown a trend of retreat with acceleration in the period of 1930-1960 and since 1995 (Björnsson and Pálsson, 2008).

Figure 3.11 shows the location of the sampling sites at Svínafellsjökull and Figure 3.12 shows site and sample photos. Samples from Svínafellsjökull were collected during field campaigns in 2018 and 2019. Soil samples were collected from moraines in the glacier forefield to account for past glacial depositions: a lake moraine, which is the youngest moraine where the disintegrating glacier terminus was observed in 2002 (Cook, Pers. Comm.), a LIA moraine to the north of the river, and a 2500-year-old Stóralda moraine further north. Sediment and water samples were collected from the proglacial lake (formed to the southeast front of Svínafellsjökull) and the Sviná river outflowing from the lake. Difficulties were encountered while collecting sediment samples – riverbeds mostly consist of gravel and boulders, making it difficult to collect sediment samples, which resulted in samples being collected from riverbanks. Glacier basal and surface ice samples were also collected.



Figure 3.11. Sampling sites at Svínafellsjökull (Modified from Google Earth, 2012).



Figure 3.12. (a) Svínafellsjökull and (b) Virkisjökull glaciers; (c) removing topsoil to collect a soil sample; (d) water sample; (e) collecting a basal ice sample.

### 3.2.1.2 Virkisjökull sampling sites

Virkisjökull (63°58' N, 16°48' W) is also a temperate outlet glacier. Virkisjökull, as it is referred to here, consists of two distinct tributaries that join at a compound glacier terminus– Virkisjökull and Falljökull. Virkisjökull has been undergoing a phase of retreat since the 1990s, which has increased even further since 2007 (Dochartaigh et al., 2007), and overall has retreated by circa 1 km (MacDonald et al., 2016).

Figure 3.13 shows the sampling locations at the Virkisjökull. Samples were collected during field campaigns in 2018 and 2019. Soil samples were collected from four moraines: a recent lake moraine caused by the glacial retreat since 1990s (Dochartaigh et al., 2007), an intermediate moraine, a LIA moraine (Everest et al., 2017) and a 5000-6000-year-old moraine (Guðmundsson, 1998) down the transect. Sediment and water samples were collected along the downstream transect starting from a snout fed directly by glacial melt, a proglacial lake and the Virkisá river. Glacier basal and surface ice samples were also collected.

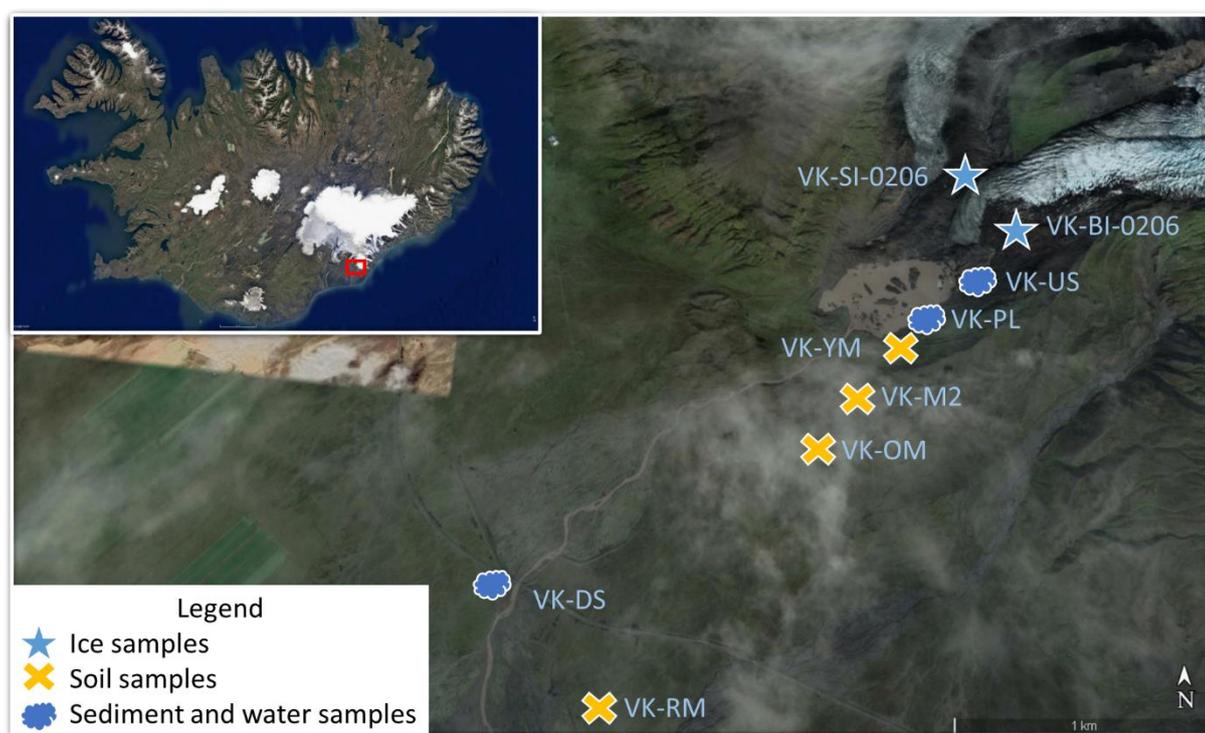


Figure 3.13. Sampling sites at Virkisjökull (Modified from Google Earth, 2012).

### 3.2.1.3 Icelandic water sampling, 2018

In 2018, apart from the main study sites, a meltwater stream from Skaftafellsjökull glacier to the northwest of the main study sites and other meltwater streams from Fjallsjökull and Kvíárjökull glaciers, as well as at Háalda river to the east were also sampled (Figure 3.14).



Figure 3.14. Other sampling sites: Kvíárjökull, Fjallsjökull and Skaftafellsjökull glaciers, and Háalda river (Modified from Google Earth, 2012).

In general, 16 water and sediment (from Svínafellsjökull and Virkisjökull), five water only (from other sites), five soil and five ice samples were collected as a result of the May-June 2018 field campaign (Table 3.1, Appendix 7). Water, river and lake sediment and soil samples were collected in triplicates to ensure data precision.

### 3.2.2 Sampling sites, 2019

A return trip to the Virkisjökull icecap, Iceland in September 2019 gave an opportunity to broaden the study area to include more detailed studies of previously visited Kvíárjökull and Fjallsjökull, and a new site, Skaftafellsjökull. Soil samples were also collected from moraines at the larger Skeiðarárjökull to the west of Skaftafellsjökull (Figure 3.15).

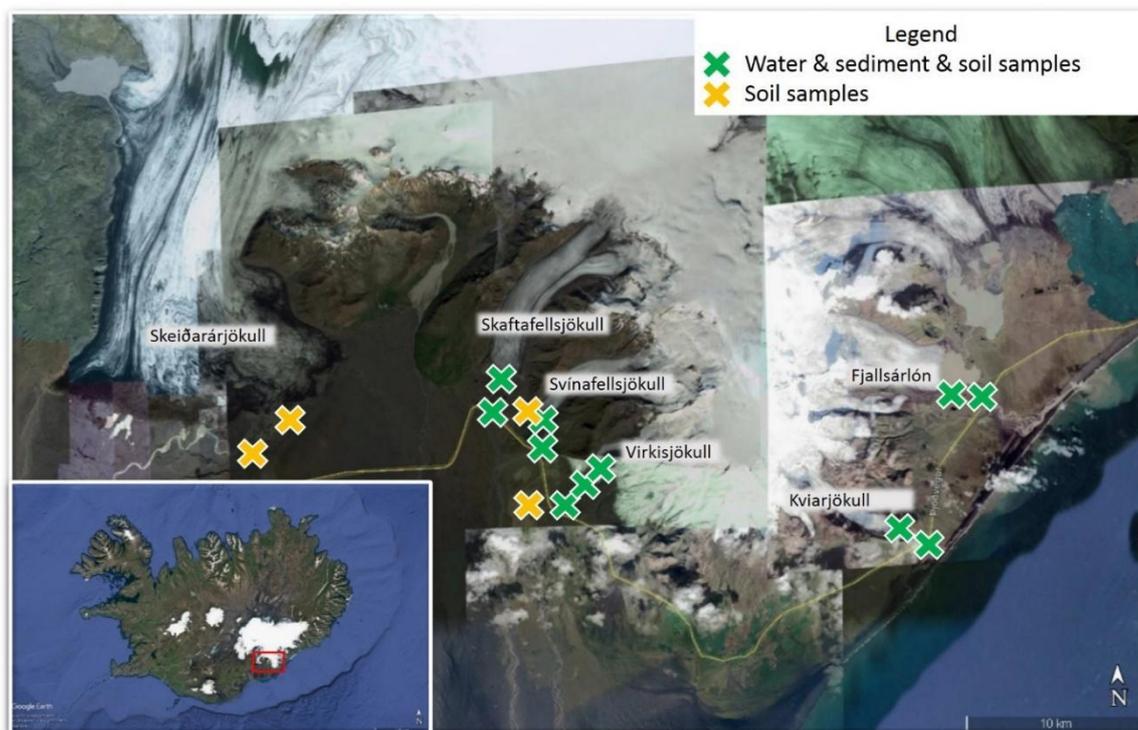


Figure 3.15. Sampling sites at the Virkisjökull icecap in 2019 (Modified from Google Earth, 2012).

### 3.2.2.1 Kvíárjökull sampling sites

Kvíárjökull (63°55' N, 16°30' W) is a temperate outlet glacier located in the south of the Virkisjökull icecap (Figure 3.16). A proglacial lake is located in front of the glacier, which feeds the Kvía river. Lateral moraines on the sides of the lake are some of the largest in Iceland, reaching 150 meters in height, which is indicative of high debris turnover as the glacier advanced in the past (Bennett et al., 2010). The moraines are called Kvíármýrarkambur (south lateral) and Kumbsmýrarkambur (north lateral) depending on the location on either side of the lake and are thought to have formed in 3200 BP; while smaller moraine remnants extending from distal slopes of Kvíármýrarkambur and Kumbsmýrarkambur have a layer of the Öraefi 1362 tephra (Guðmundsson, 1997; Bennett et al., 2010). Moreover, according to Bennett et al. (2010), glacier recession after the LIA started in 1905 leaving behind ice-cored moraines, which were replaced by hummocky moraines as the glacier went through a phase of active retreat between 1964 and 1980. The glacier has been retreating since 1905 except for a period of brief readvance in the mid-1990s.

Figure 3.16 shows the sampling sites at Kvíárjökull. Soil samples were collected from the south lateral moraine and two hummocky moraines, while sediment and water samples were collected from the proglacial lake and the river.

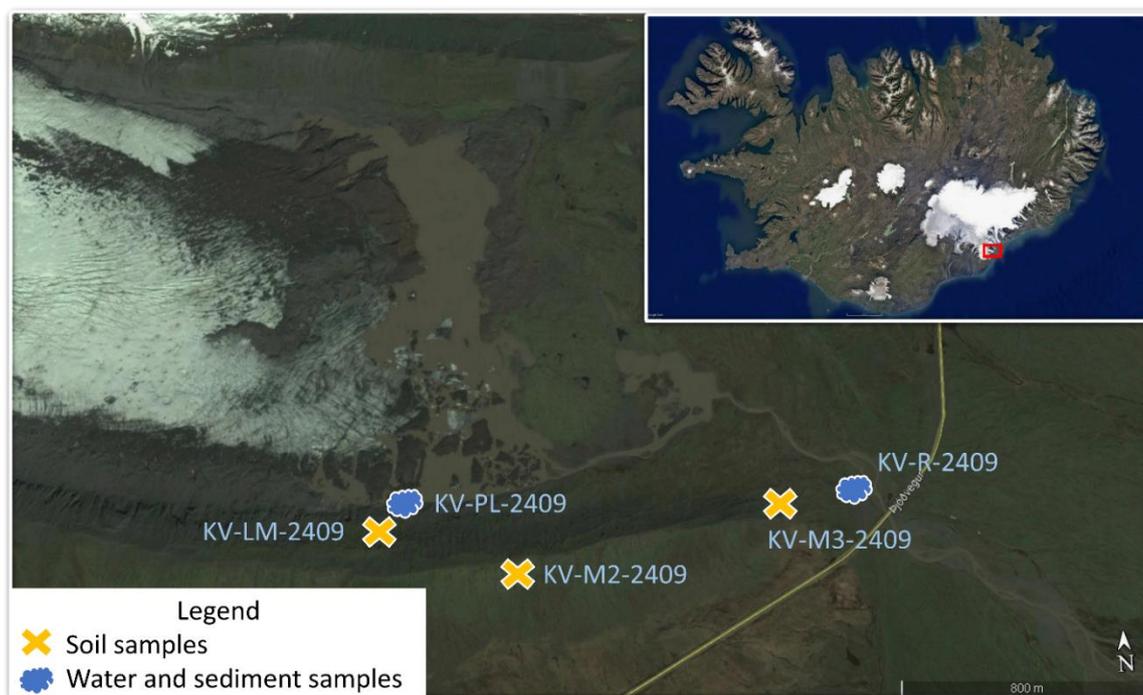


Figure 3.16. Sampling sites at Kvíárjökull (Modified from Google Earth, 2012).

### 3.2.2.2 Fjallsjökull sampling sites

Fjallsjökull (64°01' N, 16°25' W) is another temperate outlet glacier in the southeast of the Virkisjökull icecap (Figure 3.17). The glacier terminates in Fjallsárlón glacier lagoon. The original name of the glacier Hrútárjökull is now a name of a glacier at the western margin of Fjallsjökull. Major glacier advances took place during the mid-Holocene and the LIA. According to Rose et al. (1997), the initial glaciation occurred in the 4500s BP. Moreover, in the mid-Holocene, the area was home to a birch woodland. Fjallsjökull started advancing in the 1830s (LIA) and reached its maximum in 1894 (Thorarinsson, 1943), overrunning farmed areas at the terminus of the glacier; it began retreating again in 1965 (Rose et al., 1997).

Figure 3.17 shows the sampling sites at Fjallsjökull. Soil samples were collected from the moraines, while sediment and water samples were collected from the glacier lagoon and the river.

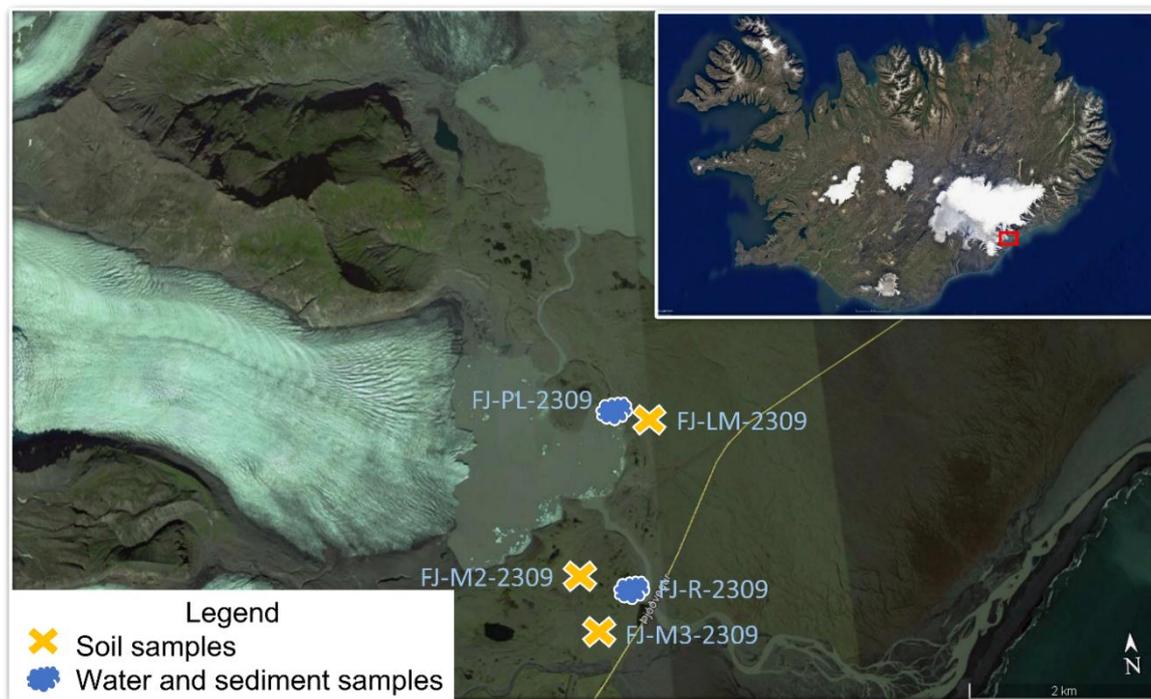


Figure 3.17. Sampling sites at Fjallsjökull (Modified from Google Earth, 2012).

### 3.2.2.3 Skaftafellsjökull sampling sites

Skaftafellsjökull (64°01' N, 16°58' W) is an outlet glacier of the Vatnajökull ice cap (Figure 3.18). During the LIA the glacier was conjoined with Svínafellsjökull (Bradwell and Everest, 2005). Glacier margin fluctuations have been monitored since 1932 (Sigurðsson, 1998; Marren, 2008). Since the start of the monitoring program, the glacier has been retreating with several brief advances in the second half of the 20<sup>th</sup> century: four advances in 1942-1970 (Sigurðsson, 1998) and an advance in 1971-1988 (Marren, 2008). The glacier had a brief period of advance in 1996-1998 and has been retreating since (Marren, 2008). A proglacial lake has developed in front of the glacier margin (Marren, 2008), where the glacier was drained by two meltwater streams in the eastern and western ends of the glacier front.

Figure 3.18 shows the sampling sites at Skaftafellsjökull. Soil samples were collected from the push moraines, the further moraine ridge dating to 1939 (Marren, 2008), while sediment and water samples were collected from the proglacial lake and the Skaftá river.

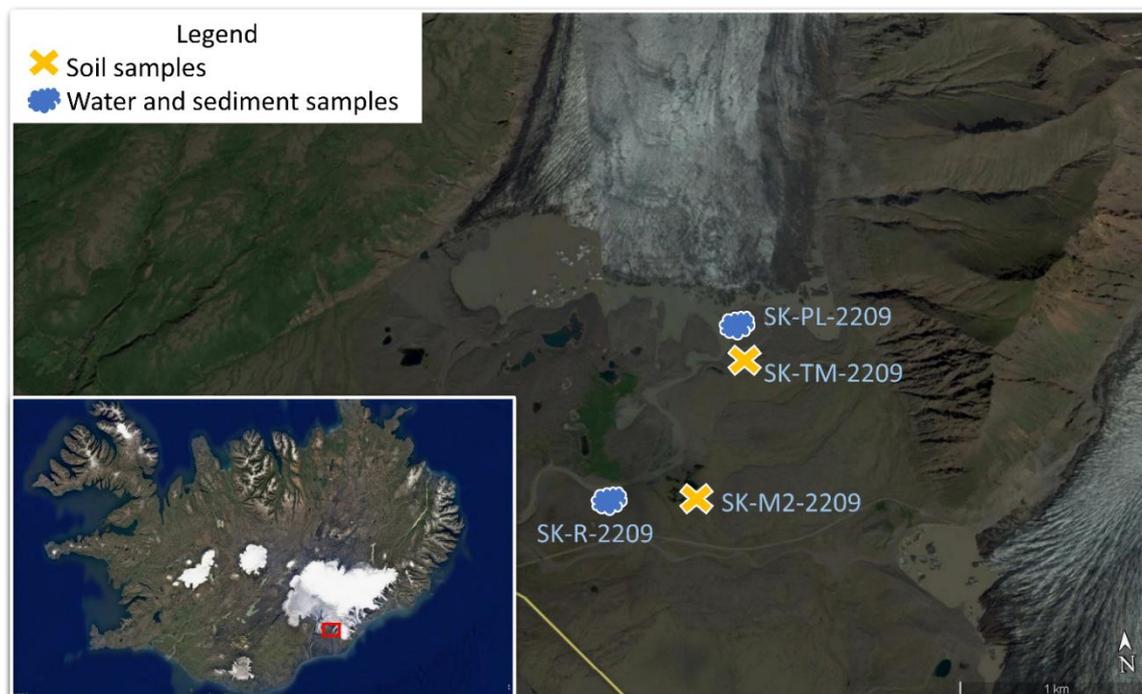


Figure 3.18. Sampling sites at Skaftafellsjökull (Modified from Google Earth, 2012).

#### 3.2.2.4 Skeiðarárjökull sampling sites

Apart from the aforementioned study sites, soil/sediment samples were collected from two moraines at Skeiðarárjökull. Skeiðarárjökull ( $63^{\circ}47' \text{ N}$ ,  $16^{\circ}56' \text{ W}$ ) is a large outlet glacier at the southern margin of the Vatnajökull ice cap. The glacier has a total area of  $160 \text{ km}^2$  and a glacier margin of 25 km making it the largest of the Icelandic glaciers (Bradwell and Everest, 2005). Skeiðarársandur is an active glacial outwash plain at the margin of Skeiðarárjökull. Sediment deposit from frequent jökulhlaups (glacial outburst flood) makes up the stratigraphy of Skeiðarársandur (Maizels, 1991, Maizels, 1993b, Guðmundsson et al., 2002 in Robinson et al., 2008), 1996 jökulhlaup being one of the high-magnitude ones (Robinson et al., 2008). Data from this site give an additional understanding of processes taking place at different catchments of the Vatnajökull icecap.

Overall, 11 water, 9 river and lake sediment and 20 soil samples were collected in the September 2019 field campaign (Table 3.1, Appendix 8). Recurrently, water, sediment and soil samples were collected in triplicates to ensure data precision.

### 3.3 Field site – Greenland

Lastly, Zackenberg valley in Greenland was visited in August 2019. A 50 km transect along the valley was sampled for soil, sediment and water samples.

The Greenland Ice Sheet (GRIS) covers 82% of the surface area of Greenland (Meltofte and Rasch, 2008). The largely ice-free area around the periphery of the ice sheet is also partially covered by glaciers. The Zackenberg River is located in Northeast Greenland (74°30'N, 20°30'W; Figure 3.19); it has a catchment area of 514 km<sup>2</sup> (Meltofte and Rasch, 2008) of which 101 km<sup>2</sup>, or 20%, of the total area is covered by glacier ice (Hasholt et al., 2008). The area has a mountainous terrain. Glaciers, glacial moraines, snow cover, permafrost, lakes, rivers, mountains and alluvial fans are representative of the valley.

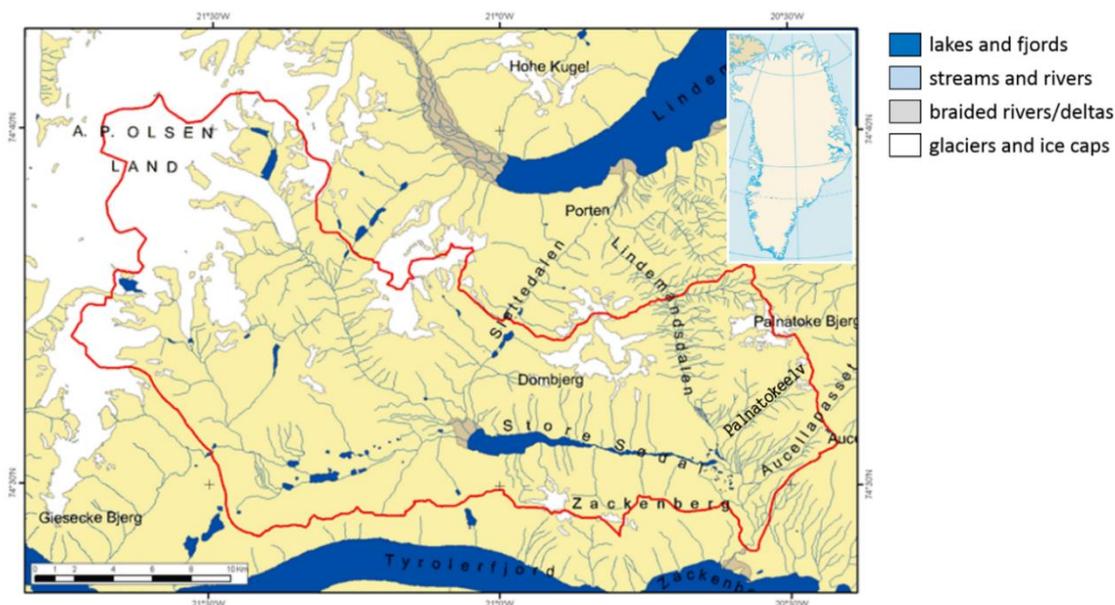


Figure 3.19. Zackenberg River drainage basin (Modified from ZERO (n.d.) and wikimedia.org, 2008).

The A.P. Olsen Land ice cap (74° 39' N, 21° 42' W) covers an area of 295 km<sup>2</sup> (Larsen et al., 2012), while the elevation of the ice cap reaches 1425 m (Hansen et al., 2017). Its outlet glacier in the Zackenberg River basin drains into a meltwater stream to the east feeding the river at 525 m (Hansen et al., 2017). The A.P. Olsen Land ice cap joins four glacial valleys before joining the Store Sødal valley to the east. The meltwater stream creates a wide delta along with other glacial meltwater streams as well as other minor non-glacial streams as they flow into the Store Sødal Lake located in the Store Sødal Valley before flowing south to the Zackenberg Valley. The Store Sødal lake covers an area of 4.9 km<sup>2</sup> (Hasholt et al., 2008)

and has an average depth of 27 m (Meltofte and Rasch, 2008). The lake traps sediment brought in with glacial meltwater, which may have come directly from the glacier, or may have been entrained from the bed and banks of meltwater streams. The Lindemanselven River, from the Lindemansdalen valley to the north of the Zackenberg valley, flows into the stream ~1km from its exit point at the eastern end of the Store SØ Valley along with other tributaries: the Palnatokeelv, draining Palnatokebjerg, and the Aucellaelv, draining the Aucellabjerg (Figure 3.19). The Palnatokeelv and Aucellaelv rivers are mostly fed by snowmelt, as well as a small input from glacial meltwater. Other minor streams also drain into the Zackenberg River. The river outflows to the Young Sund and Tyrolerfjord fjords.

Tertiary igneous rocks, especially plateau basalts, comprise the coastal and outer fjord regions of East Greenland (Mowatt and Naidu, 1994). A large north-south fault runs across the Zackenberg valley (Figure 3.20) with Zackenberg Mountain composed of Precambrian orthogneiss to the west, where majority of the study area is located, and Aucellabjerg Mountain comprised of Cretaceous to Jurassic breccia, sandstone, and mudstone to the east (Pedersen et al., 2013 in Cable et al., 2018; Escher and Watt, 1976 in Christiansen et al., 2008), with a layer of Tertiary basalt covering the sandstone layer as the elevation increases above 600 m (Koch and Haller, 1971 in Meltofte and Rasch, 2008). The western side of the valley has steeper slopes and overlaid by coarse deposits, while the eastern side has a gentler slope covered by sediments (Garcia-Oteyza et al., 2022). Moraines, meltwater plains and delta terraces, as well as permafrost landforms such as ice-wedge polygons and palsas can be found in the lowlands, which were shaped by glacial, fluvial, marine, and periglacial processes (Christiansen et al., 2008).

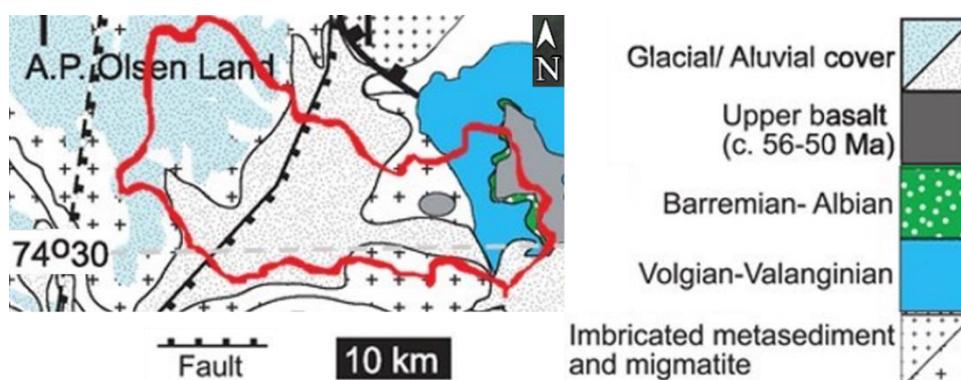


Figure 3.20. Geological map of NE Greenland. Zackenberg River drainage basin is highlighted with red line. (Modified from Hartz et al., 2002).

The study area is situated within a zone of continuous permafrost (thermally sensitive layer which experiences sub-zero temperatures for at least two years in a row (Harris et al., 1988)) with an estimated depth of 200-400 m, the top 45-80 cm of which is the active layer (top layer of ground susceptible to annual thawing and freezing cycles (Harris et al., 1988)) (Christiansen et al., 2008).

As a High Arctic location, Zackenberg valley has polar days and nights that last for 106 and 89 days respectively (Hansen et al., 2017). In 2015, annual air temperature was  $-8.8\text{ }^{\circ}\text{C}$ , with coldest monthly mean air temperature of  $-24.5\text{ }^{\circ}\text{C}$ , while the warmest monthly mean air temperature was  $6.6\text{ }^{\circ}\text{C}$  (Hansen et al., 2017). Snow cover in the area starts to accumulate in September and can remain until May-June and sometimes even July. According to data from the Berkeley Earth (n.d.), at the neighbouring Daneborg station, the annual mean temperature increased by  $3.89\text{ }^{\circ}\text{C}$  in the last century (1944-2013) (Figure 3.21). Average annual precipitation at Zackenberg was 261 mm per annum in the period of 1996-2005 (Hansen et al., 2008). Average annual wind speed at Zackenberg is 2.8 m/s, however, due to foehn events it can rapidly change escalating from  $<2\text{ m/s}$  to  $>20\text{ m/s}$  within few hours (Hansen et al., 2008).

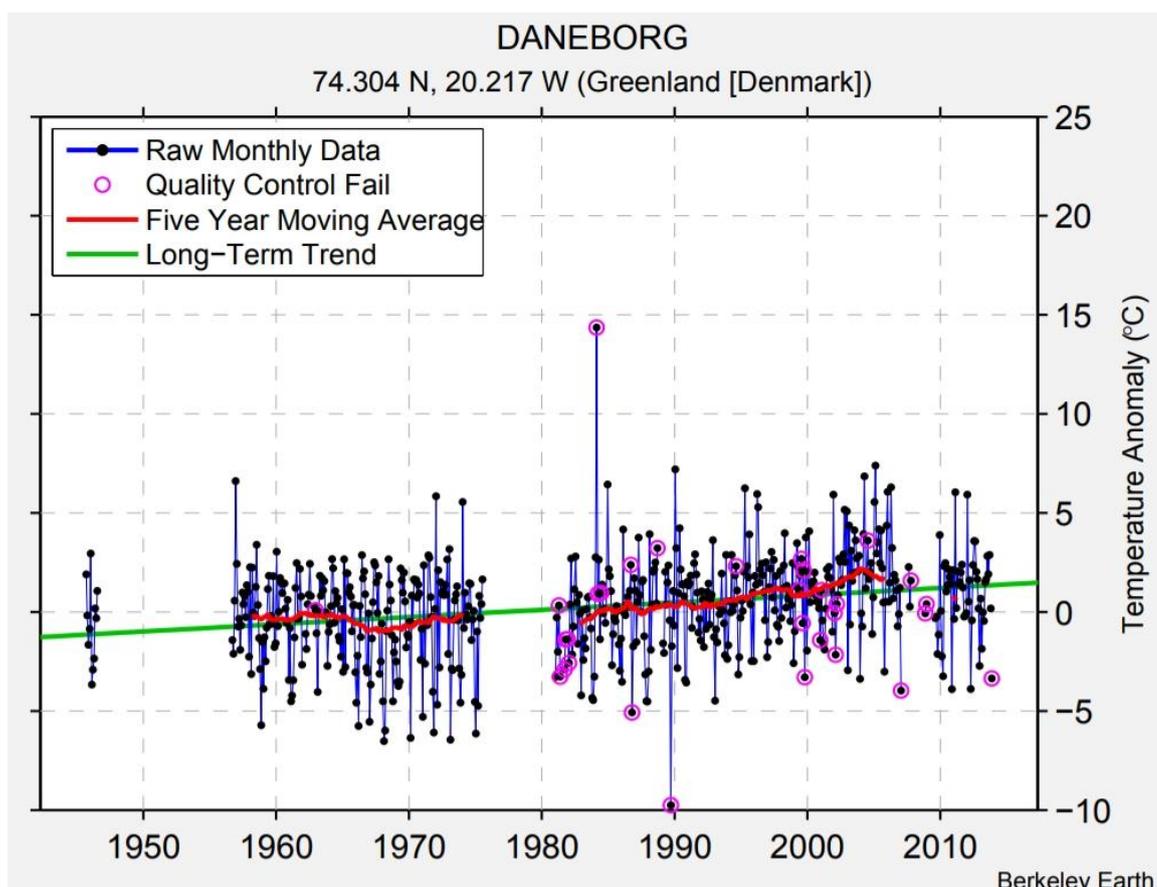


Figure 3.21. Mean annual temperature trend (1944-2013). (Adapted from Berkeley Earth (n.d.)).

The area is mostly unpopulated with the exception of Zackenberg research station located at the Zackenberg River drainage basin and operated during the summer season.

### 3.3.1 Greenland sampling sites

Figure 3.22 shows sampling sites in Greenland. Soil, river and lake sediment and water samples were collected along the 50 km transect downstream of the outlet glacier at the A.P. Olsen Land Ice Cap. Samples were collected along/from (1) the glacial meltwater streams upstream of the area where they join together to form a delta at the head of Store Sø lake (Figure 3.23a); (2) the delta; (3) several locations at the Store Sø lake (Figure 3.23b); (4) in the river downstream from the lake outlet; along the Zackenberg River upstream and downstream from the confluences with the Lindemanselven and Palnatokeelv rivers (Figure 3.23c); (5) downstream from the Aucellaelv river confluence; and (6) several locations upstream from the Young Sund and Tyrolerfjord fjords. The samples were collected in August 2019 over the period of three weeks. Overall, 13 sites were sampled in triplicates (Table 3.1; Appendix 9).

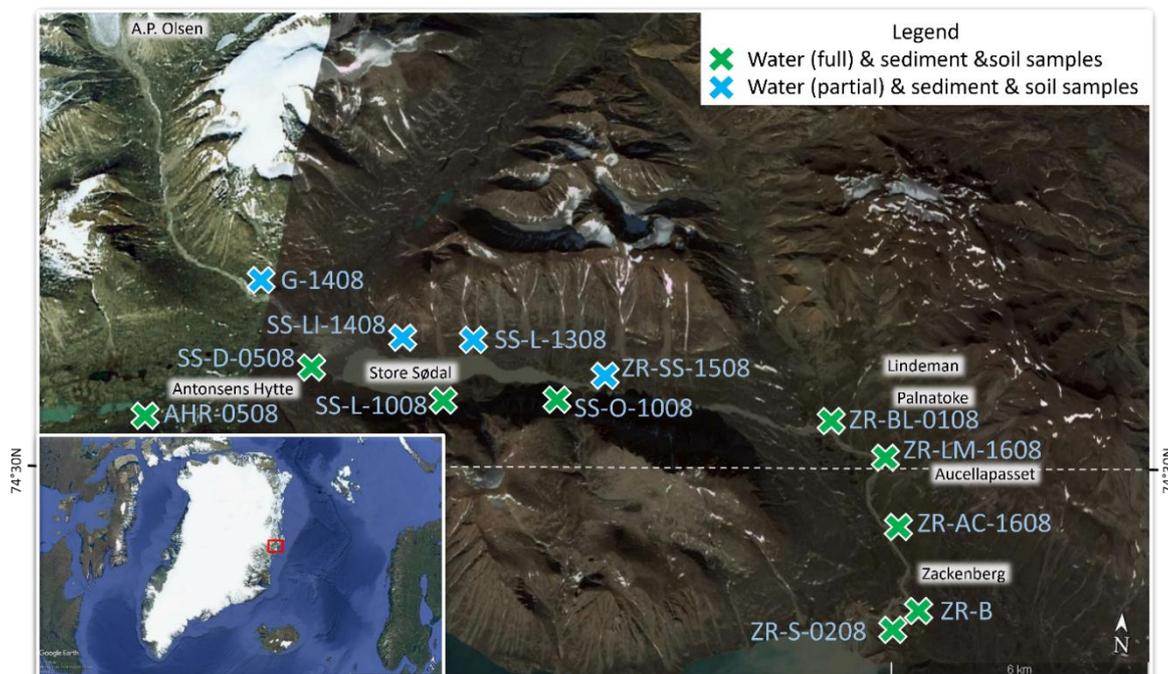


Figure 3.22. Sampling sites in Zackenberg River Drainage Basin, Greenland (Modified from Google Earth, 2012). Partial water filtration was performed in the field using syringe filters only, no filters were obtained for particulate metals analysis. See section 2.1.2.2 for more details.

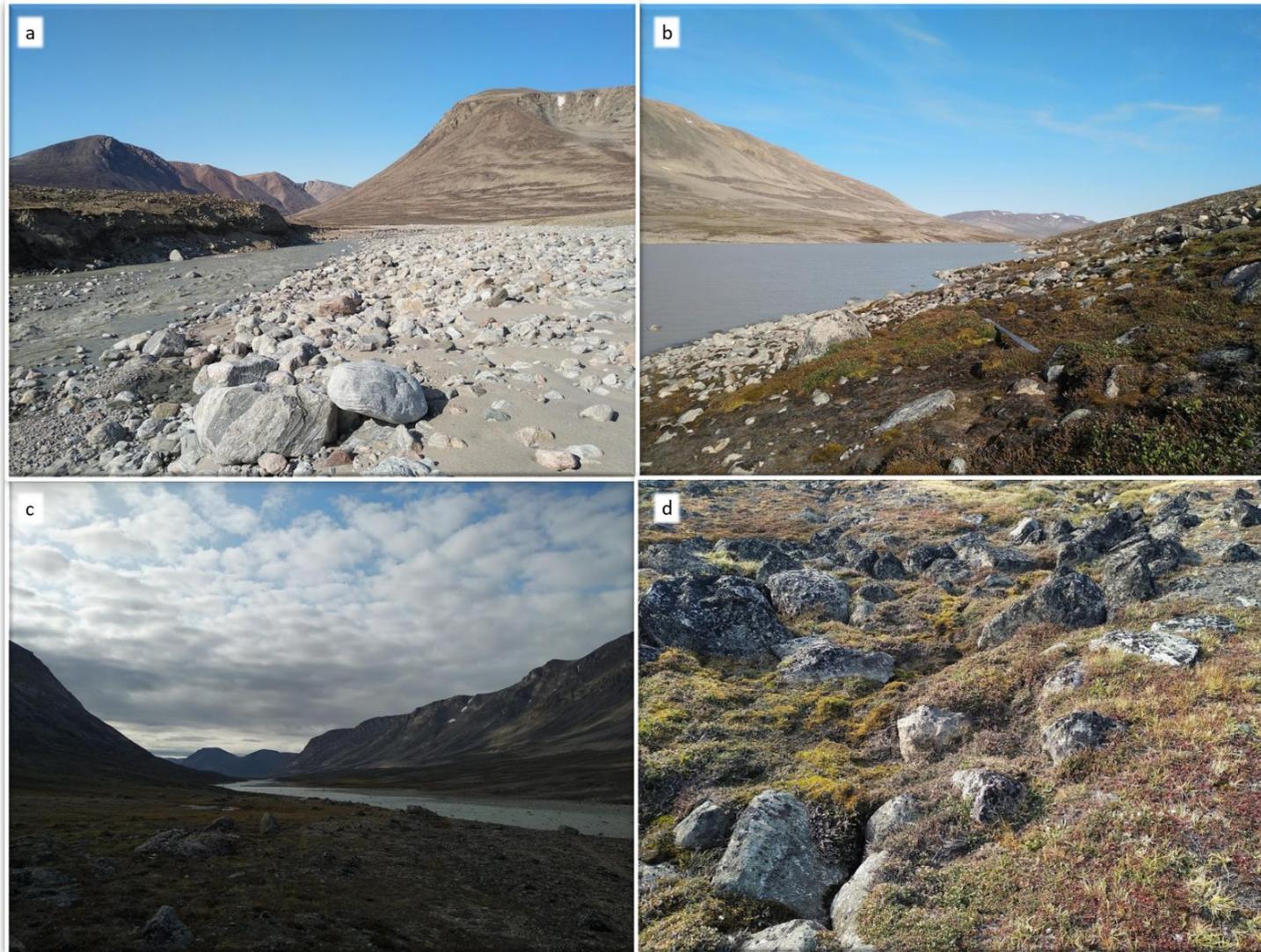


Figure 3.23. (a) Meltwater stream draining a glacier at the A.P. Olsen Land ice cap; (b) Store Sødal valley; (c) Zackenberg river; (d) collecting a soil sample.

Comparing samples from the three field sites (Sweden, Iceland and Greenland) allows for a broader understanding of accumulation of soil derived OC in deglaciated catchments in northern latitudes. The catchments are at different stages of deglaciation: Tarfala valley in Sweden being the youngest, Vatnajökull ice cap in Iceland being an older one, and Zackenberg valley in Greenland being the catchment, which has been ice free for the longest period.

## **3.4 Water data**

This sub-section provides supplementary data of water analysis to see the differences and similarities in water chemistry by country and by transect within Sweden and Iceland.

### **3.4.1 Differences and similarities between Sweden, Iceland and Greenland based on meltwater analysis**

Bedrock lithology has a direct influence on meltwater chemistry of glacial runoff, both as dissolved and particulate load (Eiriksdottir et al., 2015). Principal component analysis using major ions (Appendix 10), dissolved (Appendix 11) and particulate metals (Appendix 12) concentrations was conducted on water samples collected from Sweden, Iceland and Greenland (Figure 3.24) to see the differences and similarities in water chemistry between the countries. It seems both principal component (PC) 1 and PC2 had stronger correlation with particulate metals compared to dissolved metals and ions. This could be due to the fact that dissolved metals have limited water-rock interaction (Eiriksdottir et al., 2015).

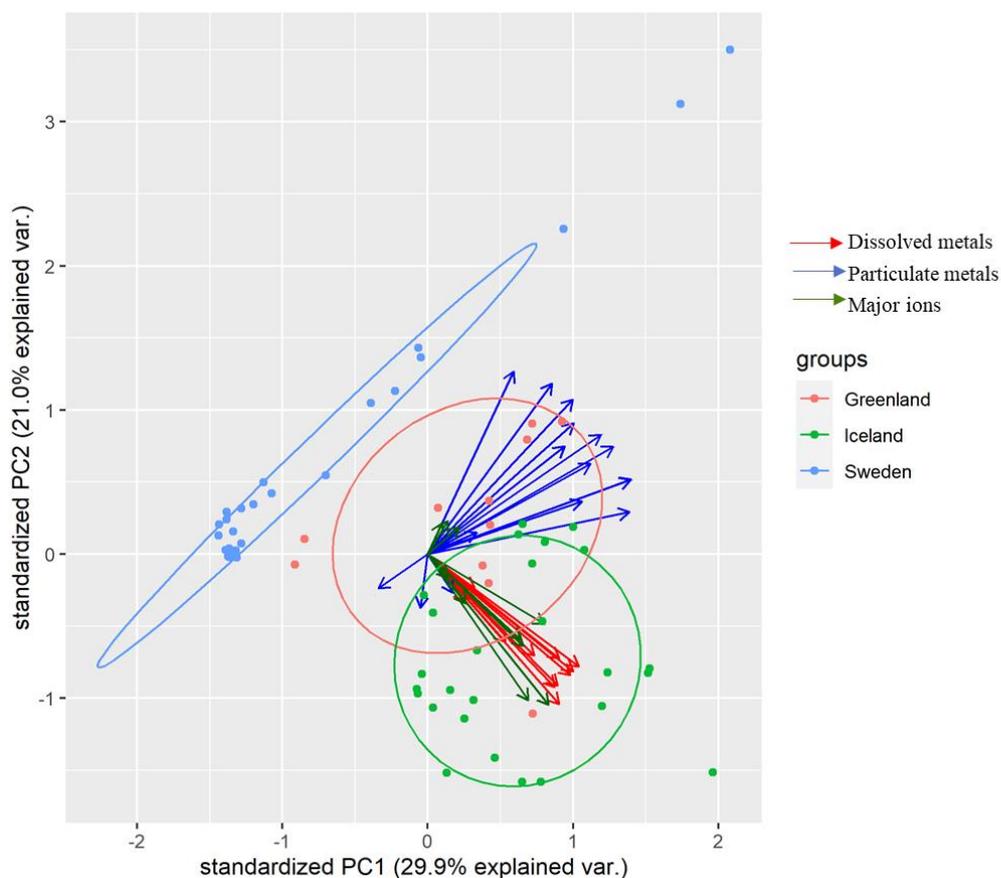


Figure 3.24. PCA ordination plot illustrating the discrimination between water samples from Sweden, Iceland and Greenland according to chemical properties (dissolved metals, particulate metals and major ions) by country. The input data was standardised to have zero mean and variance one. The ellipses represent the core area added by confidence interval of 68%.

Despite some overlap between samples from Iceland and Greenland, the PCA plot shows that samples from different countries form distinct clusters. Moreover, PERMANOVA analysis also showed that there is difference in water chemistry between countries ( $p$ -value  $< 0.05$ ). Samples from Sweden created a cluster along the particulate metals. Similarly, samples from Greenland also cluster along the particulate metals, though they are also influenced by dissolved metals and major ions. Samples from Iceland formed a more dispersed group, indicative of more variation in chemistry. PCA plots based on individual water chemistry parameters (Figure 3.25) also show that there is distinct difference between countries by dissolved metals and major ions (Figure 3.25a and Figure 3.25b) and a small difference by particulate metals (Figure 3.25c). This observation aligns with the expectation that each country in this study has varying meltwater chemistry, as chemical content of glacier meltwater is controlled by the basement rock type, i.e., geology of the study area (Ahmad and Hasnain, 2000). Interestingly, despite having different bedrock

geology, there is a large overlap by particulate metals (Figure 3.25c), especially between Iceland and Greenland.

Bedrock geology is one of the factors contributing to soil development. For instance, OC accumulation rates are higher in soils containing volcanic material, such as tephra fall out, compared to other mineral soils (Eswaran et al., 1993 in Dahlgren et al., 2004). Moreover, according to Vilmundardóttir et al. (2018), even though plant succession at the Hekla volcano in South Iceland has been hindered by tephra fall events and wind erosion, soils developed and accumulated OC over time. This suggests that the volcanic bedrock of Iceland promotes soil development faster than the metamorphic and sedimentary bedrocks in Sweden and Greenland.

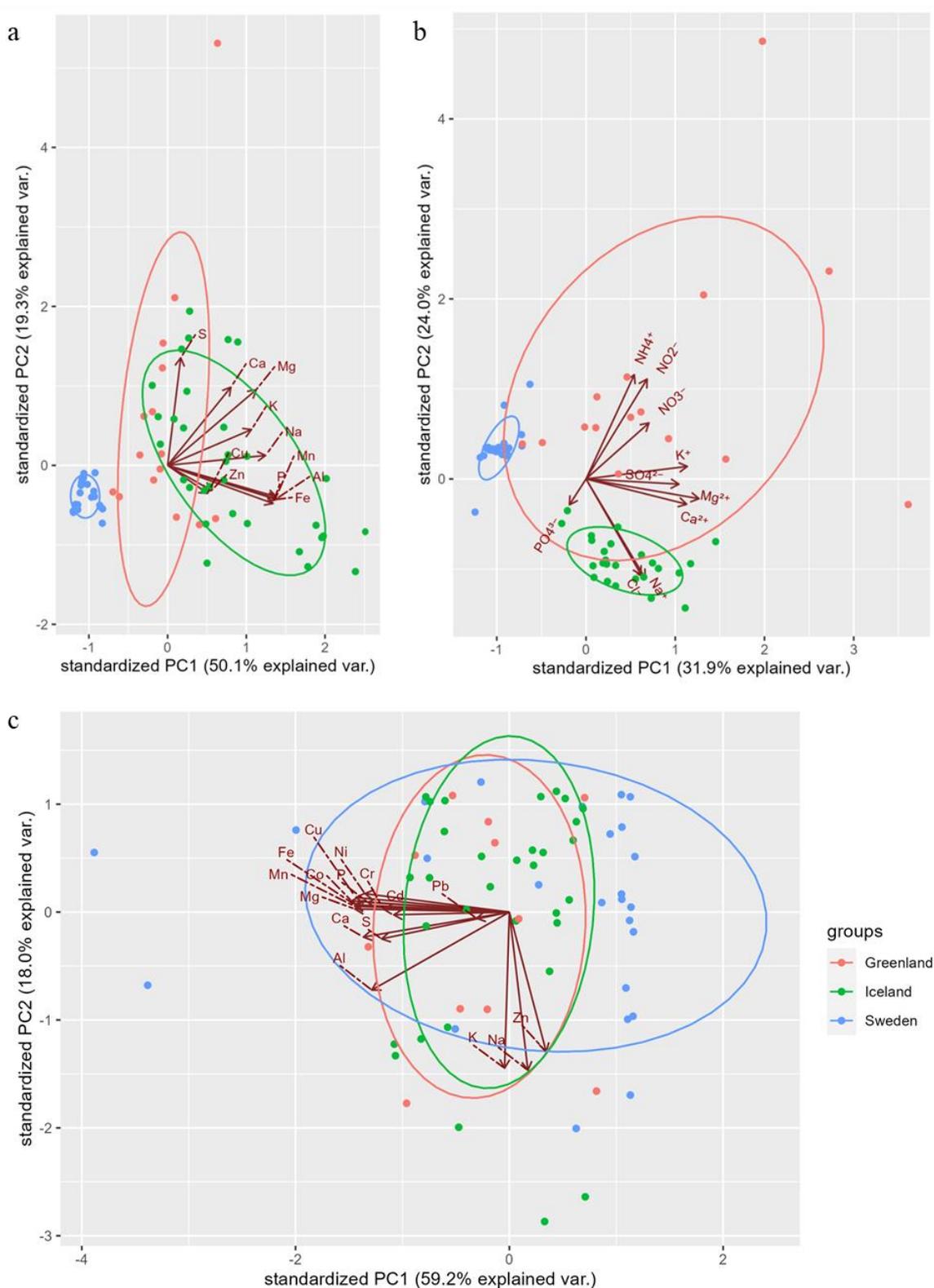


Figure 3.25. PCA ordination plot illustrating the discrimination between water samples from Sweden, Iceland and Greenland according to (a) dissolved metals, (b) major ions and (c) particulate metals by country. The input data was standardised to have zero mean and a standard deviation of one. The ellipses represent the core area added by confidence interval of 68%.

### 3.4.2 Differences and similarities between the transects at the Tarfala valley

To see the differences and similarities in water chemistry and therefore, in geology (Ahmad and Hasnain, 2000; Eiriksdottir et al., 2015) of transects in Sweden, a PCA plot was constructed using dissolved and particulate metals and major ions of meltwater samples from the Tarfala valley (Figure 3.26). PC1 is correlated with particulate metals and some dissolved metals, while PC2 is correlated with some dissolved metals and major ions.

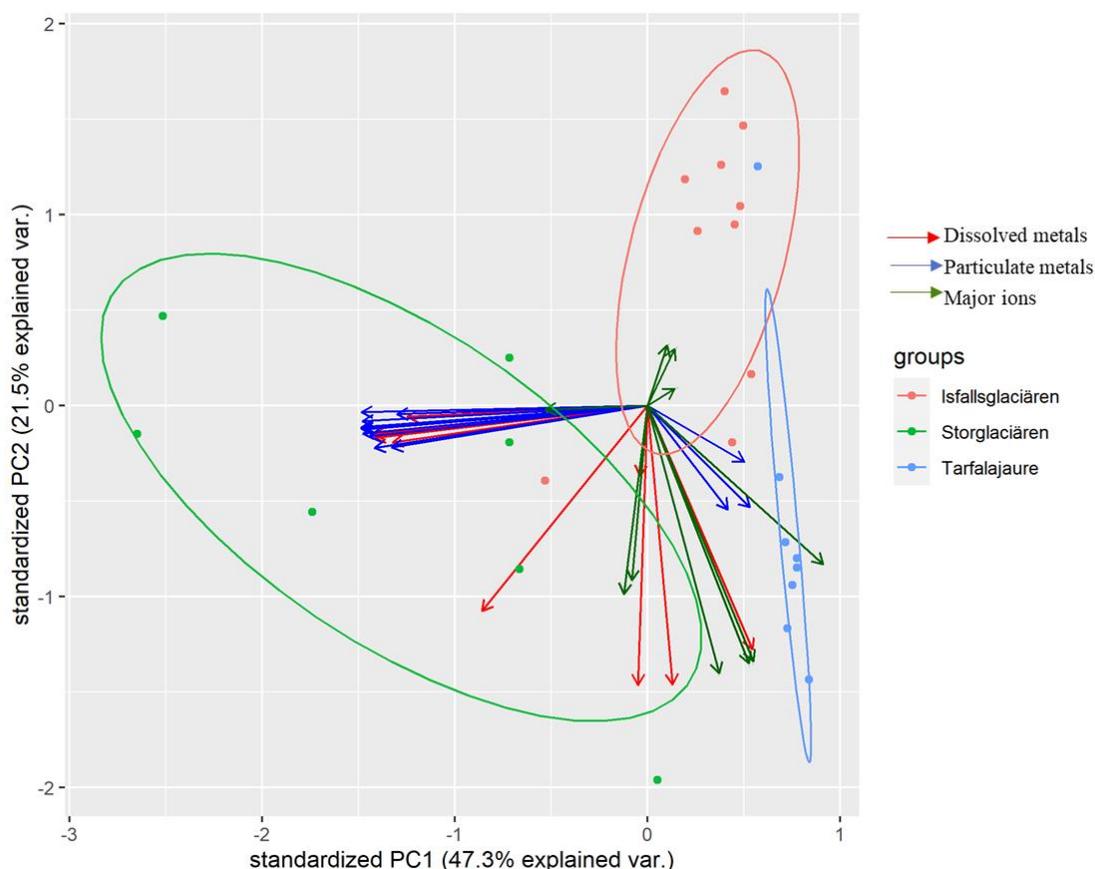


Figure 3.26. PCA ordination plot illustrating the discrimination between water samples from the Tarfala valley according to chemical properties (dissolved metals, particulate metals and major ions) by transect. The input data was standardised to have zero mean and variance one. The ellipses represent the core area added by confidence interval of 68%.

Figure 3.26, as well as PERMANOVA analysis ( $p$ -value  $< 0.05$ ) shows difference between the transects. Samples from the Tarfalajaure transect are stretched along PC2, indicative of little variation by PC1 (particulate metals and some dissolved metals), which can also be seen in PCA plots in Figure 3.27a and Figure 3.27b constructed using dissolved and

particulate metal data, respectively. Samples from Storglaciären and Isfallsglaciären form larger clusters, indicative of more variation in water chemistry. PCA plots based on individual water chemistry parameters also show differences between the transects based on dissolved (Figure 3.27a) and particulate metals (Figure 3.27b), while major ions had some overlap, samples from Storglaciären overlapping with both, Isfallsglaciären and Tarfalajaure (Figure 3.27c). This suggests that despite the very close proximity, transects in Sweden differ by water chemistry and, therefore, might have different rates of accumulation of soil derived OC.

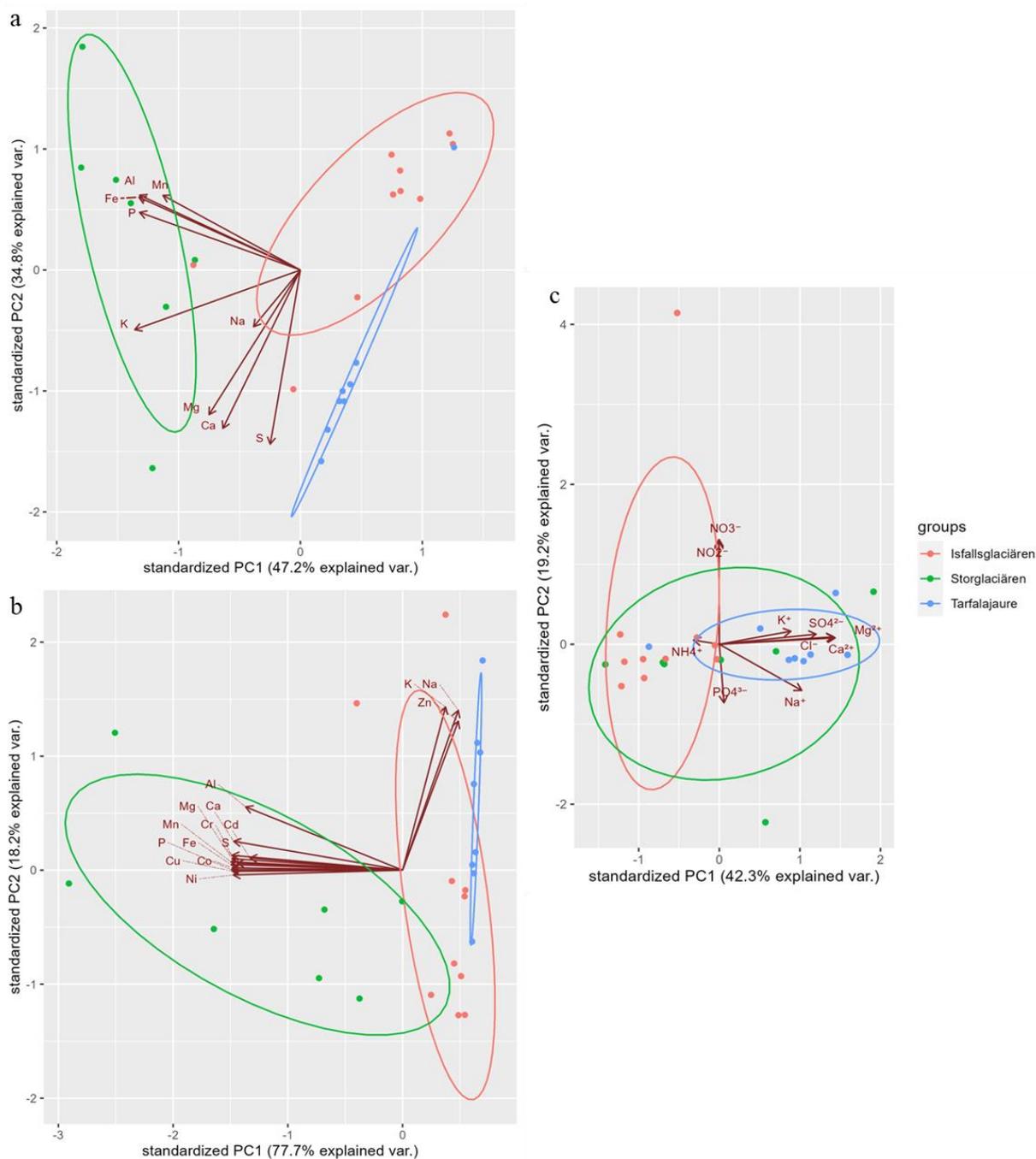


Figure 3.27. PCA ordination plot illustrating the discrimination between water samples from the Tarfala valley according to (a) dissolved metals, (b) particulate metals and (c) major ions by transect. The input data was standardised to have zero mean and variance one. The ellipses represent the core area added by confidence interval of 68%.

### 3.4.3 Differences and similarities between the glaciers at the Vatnajökull ice cap

Water samples from the Vatnajökull ice cap were analysed for dissolved and particulate metals and major ions. As water chemistry depends on bedrock lithology (Ahmad and Hasnain, 2000; Eiriksdottir et al., 2015), PERMANOVA analysis was performed and a PCA plot was constructed to see differences and similarities between glaciers in Iceland (Figure 3.28). PERMANOVA analysis showed that there is statistically significant difference in the chemistry of water samples between the glaciers in Iceland ( $p$ -value  $< 0.05$ ). As for the PCA, PC1 is mostly aligned with eigenvectors representing particulate metals and some major ions, while PC2 is mostly correlated with dissolved metals.

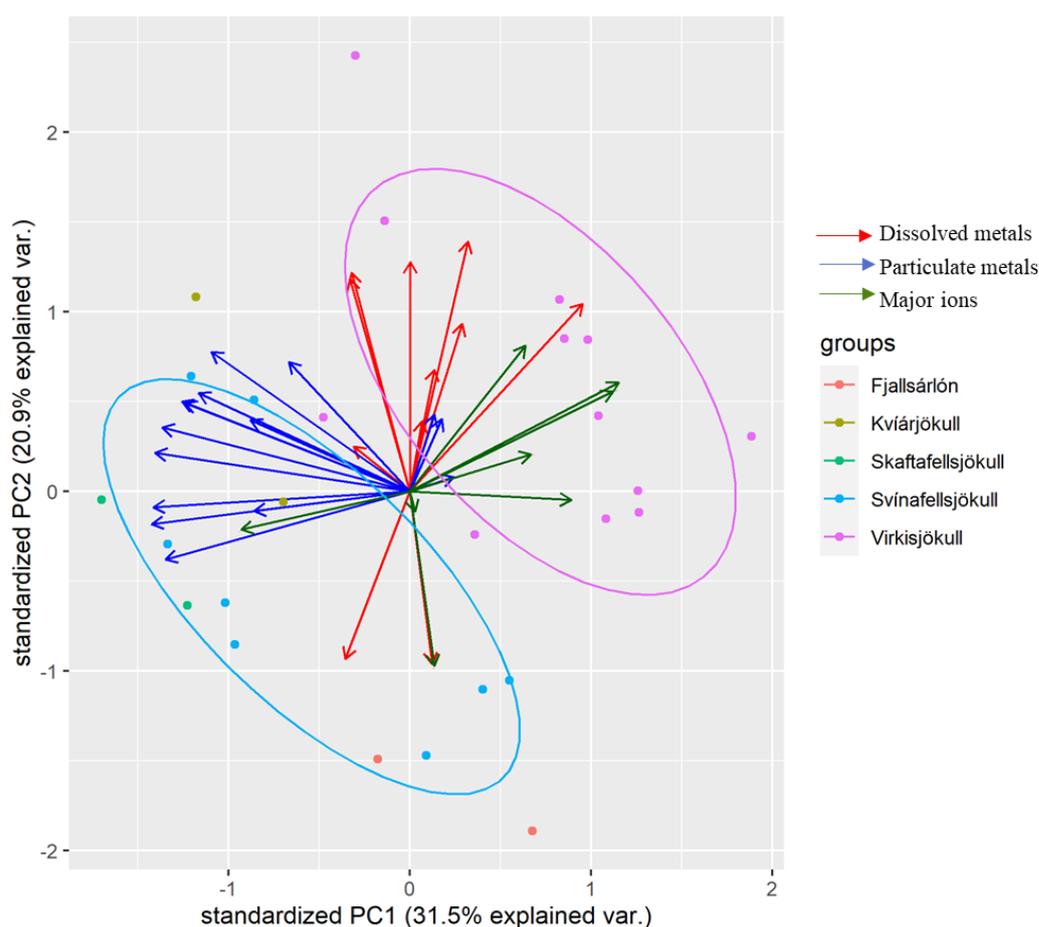


Figure 3.28. PCA ordination plot illustrating the discrimination between water samples from the Vatnajökull ice cap according to chemical properties (dissolved metals, particulate metals and major ions) by glacier. The input data was standardised to have zero mean and variance one. The ellipses represent the core area added by confidence interval of 68%.

Two main glaciers studied in Iceland, Svínafellsjökull and Virkisjökull, form distinct parallel clusters at the bottom-left and top-right halves of the plot, accordingly (Figure 3.28). This suggests that despite close proximity, the glaciers have different chemical properties. It seems samples from Fjallsjökull, Kviarjökull and Skaftafellsjökull have some overlap with samples from Svínafellsjökull. In order to investigate this further, separate PCAs were constructed based on each parameter (Figure 3.29). These PCA plots show that despite some overlap, there is difference between the glaciers based on dissolved metals (Figure 3.29a). Meanwhile, there is a larger overlap between the glaciers based on particulate metals (Figure 3.29b), while there is a clear difference between the glaciers based on major ions (Figure 3.29c). Interestingly, a water sample from the Háalda river, which is not a glacial sample, is located at the top of the plot (Figure 3.29a), away from glacial meltwater samples, suggesting that it has a different chemistry due to the origin of the sample. The overall observations suggest that glaciers at the Vatnajökull ice cap have some differences by meltwater chemistry despite sitting within the same bedrock, comprised of basaltic lava and hyaloclastite, with tephra fallout on flanks (Roberts and Guðmundsson, 2015) (Figure 3.9), though a non-glacial water chemistry is different from clusters of glacial samples.

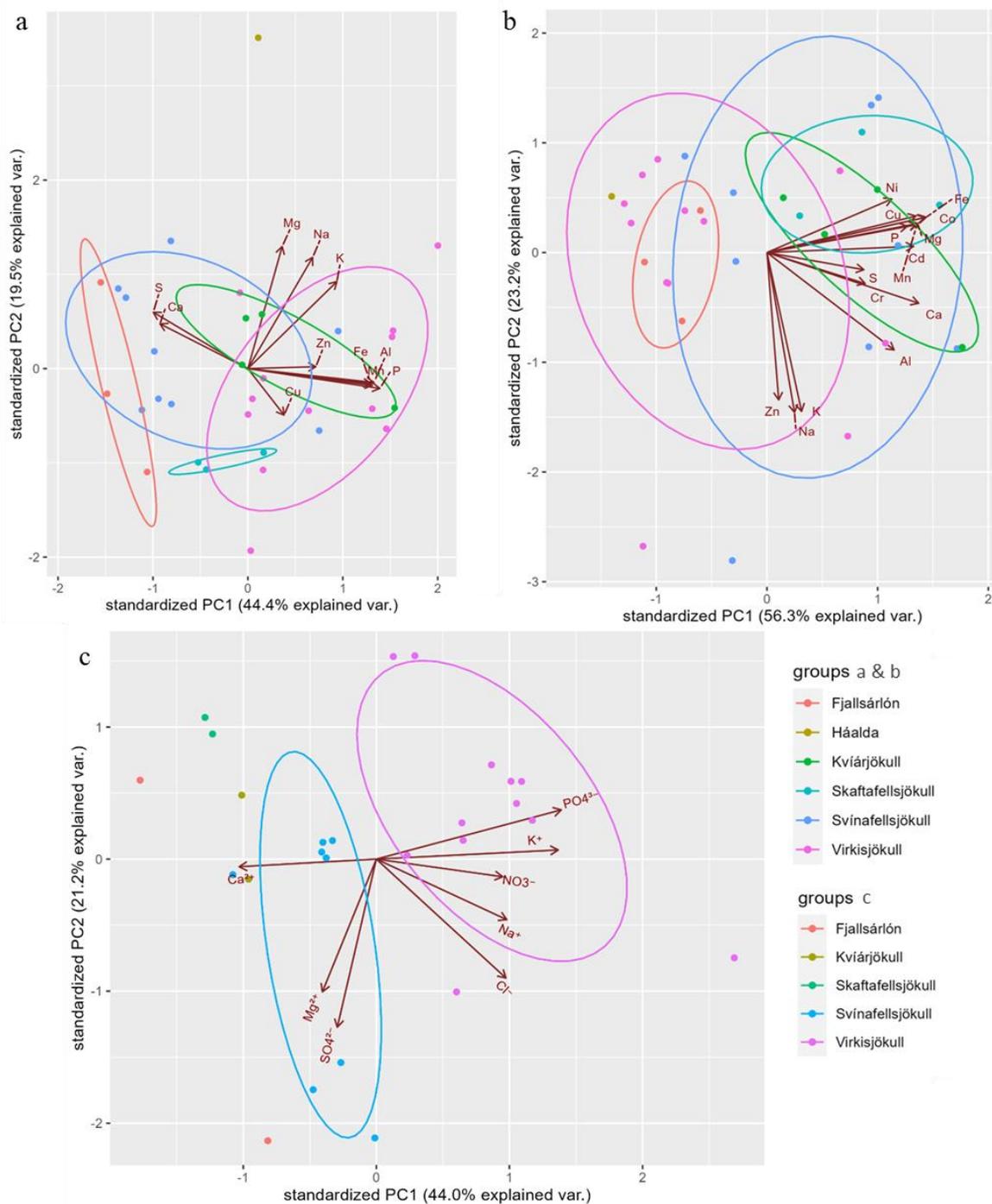


Figure 3.29. PCA ordination plot illustrating the discrimination between water samples from the Vatnajökull ice cap according to (a) dissolved metals, (b) particulate metals and (c) major ions by glacier. The input data was standardised to have zero mean and variance one. The ellipses represent the core area added by confidence interval of 68%.

## 4 Accumulation of soil derived OC at the Tarfala valley, Sweden

### 4.1 Introduction

This chapter investigates accumulation of soil derived OC in a bare catchment, Tarfala valley in Sweden. The Tarfala valley is the youngest catchment out of the three study areas. Within the Tarfala valley, though glaciers are in a close proximity to each other, they have been deglaciated for different periods of time (Figure 3.1). Glacier forelands of Storglaciären and Isfallsglaciären have been developing since the glacier maximum in 1910 (Holmlund et al., 2005), Kebnepakteglaciären since 1940s, while surroundings of the Tarfalajaure lake have been ice free for a longer period (Svenonius, 1910; Schytt, 1959 in Kirchner et al., 2019), perhaps as long as 9000-8500 years BP (Karlen, 1979).

General environmental chemistry parameters were measured on all three transects but, since Storglaciären and Isfallsglaciären are at the similar stages of development, detailed organic biomarker and microbial community analyses were focused on Tarfalajaure and Storglaciären as representatives of accumulation of soil OC from well-established and less established soils.

The main aim of this chapter was to understand the accumulation of soil derived OC at the Tarfala valley by looking at catchment chemistry, organic biomarkers and microbial community along transects along the valley away from the glacier front. This was achieved by carrying out the following objectives:

1. To determine TOC, C/N and major metals along three different transects: Storglaciären, Isfallsglaciären, and Tarfalajaure (including Kebnepakteglaciären);

2. To determine concentration and distribution of BHPs and GDGTs and their proxies ( $R'_{\text{soil}}$ , MAT and soil pH) in samples selected based on the results of objective 1 along Storglaciären and Tarfalajaure transects;
3. To determine microbial community structure in samples selected based on the results of objective 1 from Storglaciären and Tarfalajaure transects;
4. To establish a relationship between organic biomarker distribution and microbial community composition in samples from Storglaciären and Tarfalajaure transects.

Based on these objectives the following hypotheses were tested:

1. Older samples along the Tarfalajaure transect have higher TOC content due to the accumulation of OC over time, while younger samples from Storglaciären and Isfallsglaciären have less OC.
2. Since Tarfalajaure had longer time of exposure, samples along this transect have higher TOC and organic biomarker concentrations, as well as higher diversity of microbial community compared to Storglaciären foreland.
3. There is a strong correlation between OC, BHPs and GDGTs, and as a result a similar trend of OC, BHP and GDGT accumulation along downstream transects.
4. The  $R'_{\text{soil}}$  has an upward trend along the downstream transects, indicating accumulation of soil marker BHPs.
5. Bacterial community becomes more abundant and more diverse away from the glacier front.
6. Within each transect, soils are more abundant in OC, organic biomarkers, and bacteria, compared to sediments.

## **4.2 Results**

### **4.2.1 Environmental chemistry**

TC, TOC, TN, C/N (see Appendix 13 for sample names, raw data, mean concentration, and standard deviation (SD)) and metal concentrations (see Appendix 14 for mean and SD) were measured for 26 sediment and soil samples from the Tarfala valley, Sweden. Nine samples were collected along the Tarfalajaure transect (four soils and five sediments), nine from

Storglaciären (four soils and five sediments), and eight from Isfallsglaciären (three soils and five sediments). Most samples were collected in triplicates.

#### **4.2.1.1 Variation of chemical properties in different sample types and transects**

First, principal component analysis on environmental data was conducted on all soil and sediment samples collected at the Tarfala valley (Figure 4.1). The first and second principal components explained 54.2% and 21.6% of total variance, respectively. PC1 had strong positive correlation with most metals (Mn, Fe, P, Zn, Na, S, Co, Mg, Ca, Cd, and Al), while PC2 had strong negative correlation with TOC, TC, N, and some metals (Pb, Ni, Cr, and Cu). PCA plots show that there was less separation between sediments and soils (Figure 4.1a) than between different transects (Figure 4.1b).

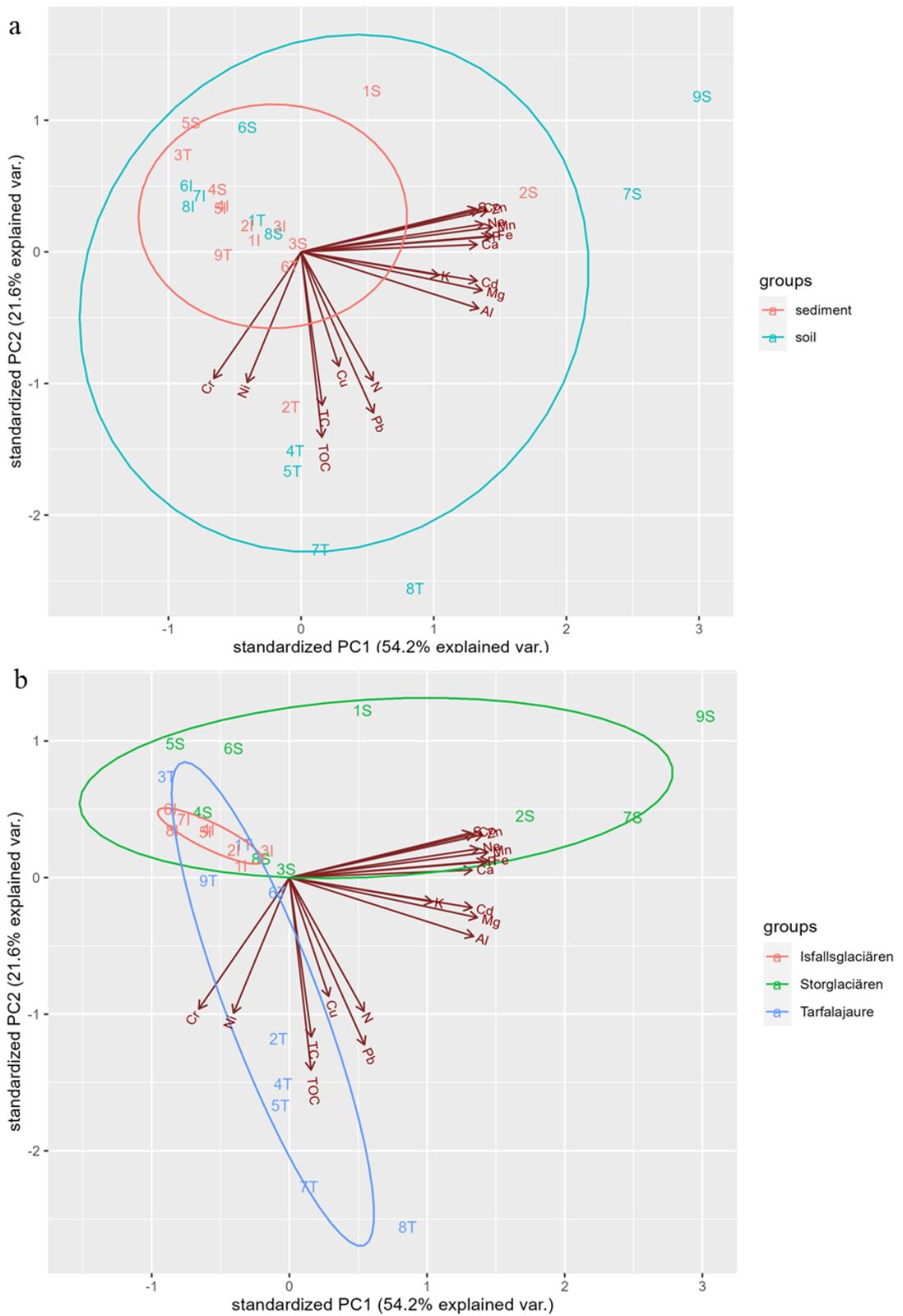


Figure 4.1. PCA ordination plots illustrating the discrimination between all samples from the Tarfala valley, Sweden according to chemical properties by the (a) sample type and (b) downstream transect. The input data was standardised to have zero mean and variance one. The ellipses represent the core area added by confidence interval of 68%. Sample codes are given in Table 4.1.

Table 4.1. Codes of sample names from Tarfala valley, Sweden shown on the PCA plots above (Figure 4.1). T stands for Tarfalajaure, S - Storglaciären, I - Isfallsglaciären.

Symbol	Sample	Type	Symbol	Sample	Type
1T	CB-1408	soil	5S	SG-MX-1008	sediment
2T	KG-BI-1308	sediment	6S	SG-OM-0808	soil
3T	KG-MW-1308	sediment	7S	SG-M2-0808	soil
4T	T-L-1308	soil	8S	SG-VP-0808	soil
5T	TL-RB-1408	soil	9S	SG-RB-0808	soil
6T	MS-TL-1408	sediment	1I	IG-NS-1208	sediment
7T	MS+IG-1908	soil	2I	IG-SS-1208	sediment
8T	ML-1908	soil	3I	IG-EL-1208	sediment
9T	MS-1008	sediment	4I	IG-DL-1108	sediment
1S	SG-BI-0808	sediment	5I	IG-DR-1108	sediment
2S	SG-R-0808	sediment	6I	IG-LITMOR-1808	soil
3S	SG-SS-1008	sediment	7I	IG-LM-1208	soil
4S	SG-DS-1008	sediment	8I	IG-OM-1808	soil

PERMANOVA analysis showed that there was no statistically significant difference between soil and sediment samples ( $p$ -value > 0.05). The PCA plot in Figure 4.1a indicates that sediment samples clustered to the top left, away from C and other elements, though some soil samples (samples number 1T, 6S, 8S, 6I, 7I, and 8I, see Table 4.1 for sample names) also occupied top left half of the plot, indicating little difference in chemical composition between soils and sediment samples. This could be due to low OC concentration of these soil samples from the Tarfala valley. Soil samples overlapping with sediment samples on the PCA plot were mostly collected from young catchments (Storglaciären and Isfallsglaciären), which have had little time to accumulate organic matter, therefore, being similar to the sediments in terms of chemical composition.

When viewed by transect/glacier (Figure 4.1b) the three transects formed distinct groups with PERMANOVA analysis showing a statistically significant difference ( $p$ -value < 0.05) between samples from the different transects. Samples from Isfallsglaciären clustered together and had very little variation in chemistry, forming a tight cluster situated within the Storglaciären group. The Storglaciären samples formed a more dispersed group, particularly along PC1, indicative of more variation in chemistry. The distribution along PC1 indicated that some samples from Storglaciären were different based on overall chemistry – for instance, three samples located at the right top corner of the plots (2S, 7S and 9S) had

higher metal concentrations. In contrast, samples from the Tarfalajaure transect were distributed along PC2, with five samples (negatively loaded on PC2) being characterised by higher carbon. The overlap between the Tarfalajaure samples and Storglaciären samples was largely for sediment samples, which also had a similar chemical composition to samples (both soils and sediment) from Isfallsglaciären.

#### 4.2.1.2 Total organic carbon concentration

Total organic carbon (TOC; %) was measured on 26 samples along three downstream transects (Figure 4.2). TOC concentration in most samples collected at the Tarfala valley were very low (mean TOC of field replicates was less than 0.1% in 19 samples, Figure 4.2, Appendix 13). At Storglaciären the TOC concentration ranged from below detection limit (BDL) to  $0.13\pm 0.11\%$  ( $n=3$ ) with a mean of  $0.04\pm 0.07\%$  ( $n=9$ ). These values were similar to those seen at Isfallsglaciären where concentrations ranged from 0.01 % to  $0.06\pm 0.03\%$  ( $n=3$ ) with a mean of  $0.03\pm 0.03\%$  ( $n=8$ ). In contrast, the Tarfalajaure transect had a wider range of concentrations, from a minimum of  $0.01\pm 0.01\%$  ( $n=3$ ) to a maximum of  $1.08\pm 0.09\%$  ( $n=3$ ), giving a mean of  $0.46\pm 0.49\%$  ( $n=9$ ).

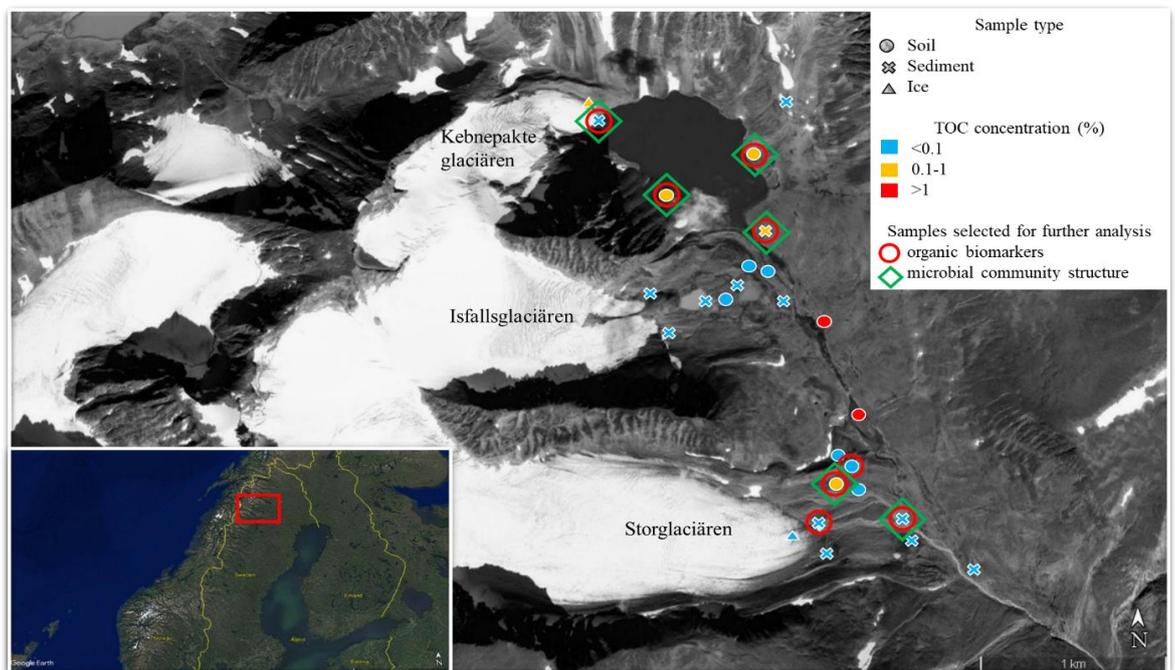


Figure 4.2. Sampling locations and total organic carbon (%) distribution along downstream transects of the Storglaciären and Isfallsglaciären glaciers and Tarfalajaure transect (including samples from the Kebnepakteglaciären and Sydöstra Kaskatjåkkoglaciären – not shown, to the top right corner of the map – glaciers), Sweden (Modified from Google Earth, 2012).

Figure 4.3 shows distribution of mean TOC concentration (%) in relation to the Tarfalajaure, Storglaciären and Isfallsglaciären downstream transects at the Tarfala valley. Samples from Tarfalajaure had the highest levels of TOC, but also the greatest range. Surprisingly, the basal ice sample (KG-BI-1308) had relatively high TOC concentration, while sediment samples from meltwater streams (CB-1408 and KG-MW-1308) had low values. Even though there was no particular pattern in TOC changes along the transect, samples collected along the lake and the river had relatively high TOC concentrations. Overall, lower levels of TOC ( $0.14\pm 0.20\%$ ) were associated with sediments, while higher levels ( $0.81\pm 0.47\%$ ) were associated with more developed soils. Samples collected along the Storglaciären and Isfallsglaciären transects had low TOC concentrations both in sediments and soils and did not vary along the downstream transects.

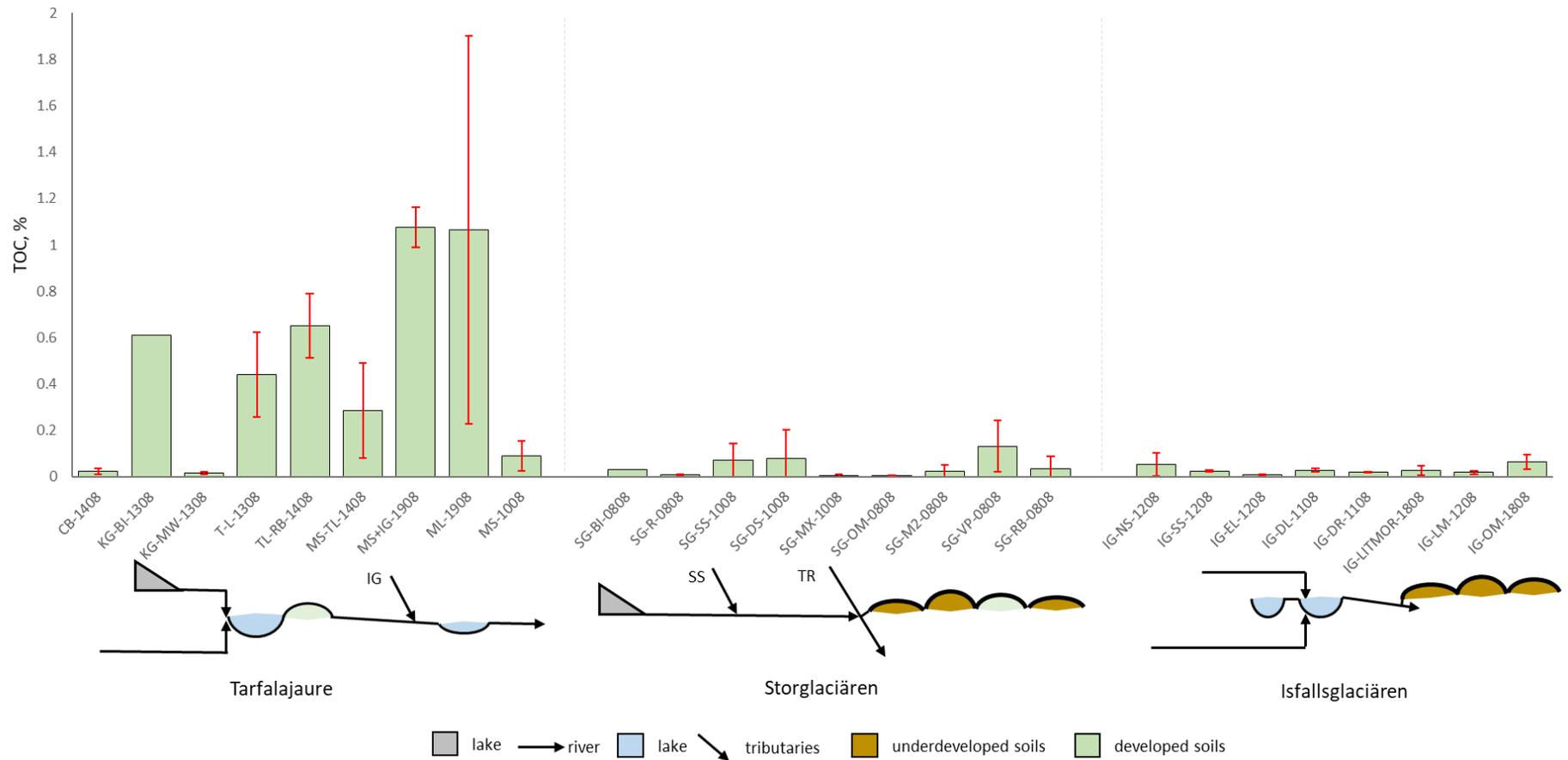


Figure 4.3. Mean distribution of TOC (%) along the Tarfalajaure, Storglaciären and Isfallsglaciären transects in Sweden. Error bars represent ± one standard deviation on triplicate samples collected from individual sampling locations at the Tarfala valley. Charts at the bottom of the graph show downstream (left to right) transect profiles. IG stands for Isfallsglaciären, SS – Storglaciären south stream, TR – Tarfalajaure.

### 4.2.1.3 Total nitrogen and C/N ratio

Total nitrogen (TN; %) concentrations were consistently very low, having a mean of  $0.03 \pm 0.01\%$  (n=9) at Storglaciären,  $0.01 \pm 0.01\%$  (n=8) at Isfallsglaciären and  $0.04 \pm 0.03\%$  (n=9) at Tarfalajaure (Appendix 13). C/N ratios varied across the three transects: from  $0.03 \pm 0.04$  (n=3) to  $4.57 \pm 3.81$  (n=3) with a mean of  $1.80 \pm 2.99$  (n=9) at Storglaciären, from  $0.50 \pm 0.25$  (n=3) to  $4.69 \pm 6.46$  (n=3) with a mean of  $2.84 \pm 3.40$  (n=8) at Isfallsglaciären, and Tarfalajaure again showing a greater range (from  $1.57 \pm 1.53$ , n=3 to  $20.57 \pm 7.48$ , n=3) and higher mean than the other transects ( $9.89 \pm 7.38$ , n=9). The higher C/N ratios along the Tarfalajaure transect are due to changing TOC values, rather than changing TN values, which showed little variation. Further, when TOC (%) and TN (%) are correlated, the nitrogen is likely to be organic sourced. The gradient of such a relationship can be diagnostic for carbon source. Linear regression of TOC (%) versus total TN (%) across sample points from Storglaciären revealed no relationship ( $R^2=5 \times 10^{-7}$ , Pearson correlation=0), a weak positive relationship for samples from Isfallsglaciären ( $R^2=0.11$ , Pearson correlation=0.34) and a strong positive relationship for samples from the Tarfalajaure transect ( $R^2=0.55$ , Pearson correlation=0.74) (Figure 4.4), suggesting that TN is organic in nature only in samples collected from the Tarfalajaure transect.

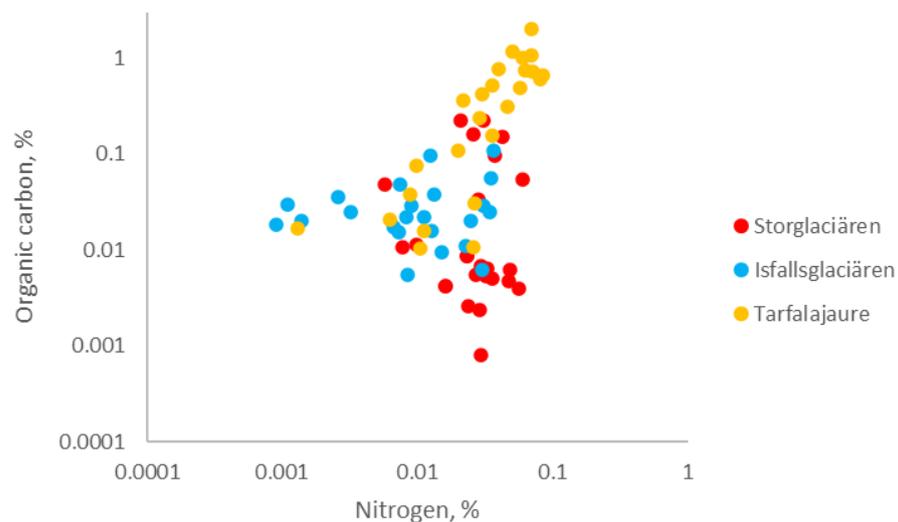


Figure 4.4. Relationship between TOC (%) and total nitrogen (%) for three different transects at the Tarfala valley (logarithmic axes).

#### 4.2.1.4 Metal concentration

Figure 4.5 shows the distribution of the mean of total and individual metal concentrations (mg/kg) along the Tarfalajaure, Storglaciären and Isfallsglaciären downstream transects. Total concentration of metals was highest in samples collected along the Storglaciären transect ( $15808 \pm 6163$  mg/kg,  $n=9$ ), followed by Tarfalajaure ( $12063 \pm 2582$  mg/kg,  $n=9$ ) and Isfallsglaciären ( $10775 \pm 1639$  mg/kg,  $n=8$ ) transects. Mean and SD of individual metal concentrations in samples from each transect are shown in Table 4.2 (see Appendix 14 for more details). Fe, Al, Ca and Mg had the highest concentrations, followed by Na, K, P, Mn and S, while Cu, Cr, Zn, Ni, Co, Pb and Cd had lower concentrations (Table 4.2).

Table 4.2. Mean and standard deviation (mg/kg) of metal concentrations in samples from Tarfalajaure, Storglaciären and Isfallsglaciären transects in Sweden.

Meta l	Tarfalajaure		Storglaciären		Isfallsglaciären	
Fe	4788	$\pm 1022$	6766	$\pm 3115$	3989	$\pm 663$
Al	2978	$\pm 727$	3290	$\pm 1034$	2691	$\pm 435$
Ca	2299	$\pm 554$	3271	$\pm 1218$	2372	$\pm 366$
Mg	1238	$\pm 266$	1414	$\pm 459$	1070	$\pm 155$
Na	298	$\pm 77$	495	$\pm 407$	286	$\pm 75$
K	206	$\pm 59$	189	$\pm 109$	153	$\pm 44$
P	129	$\pm 14$	156	$\pm 51$	109	$\pm 12$
Mn	77	$\pm 19$	125	$\pm 52$	68	$\pm 14$
S	18	$\pm 14$	72	$\pm 96$	9	$\pm 12$
Cu	11.88	$\pm 3.34$	8.75	$\pm 3.4$	9.80	$\pm 0.83$
Cr	9.38	$\pm 2.39$	6.18	$\pm 3.53$	9.48	$\pm 1.56$
Zn	3.56	$\pm 0.89$	7.23	$\pm 5.12$	2.57	$\pm 0.2$
Ni	3.52	$\pm 0.89$	2.47	$\pm 0.68$	3.27	$\pm 0.29$
Co	3.11	$\pm 0.53$	4.51	$\pm 1.45$	2.95	$\pm 0.39$
Pb	0.51	$\pm 0.36$	0.20	$\pm 0.17$	0.14	$\pm 0.12$
Cd	0.37	$\pm 0.09$	0.39	$\pm 0.18$	0.30	$\pm 0.07$

The PCA plot in Figure 4.1b indicates that samples from Storglaciären were influenced by PC1, which had strong positive correlation with most metals (Mn, Fe, P, Zn, Na, S, Co, Mg, Ca, Cd, and Al). Samples from Storglaciären had the highest concentration of these metals compared to other two transects (Table 4.2). Samples from Isfallsglaciären were also affected by PC1 but formed a tighter cluster. Overall, metal concentrations were lower and had little variation (lower SD values in Table 4.2) compared to samples from Storglaciären. On the contrary, samples from the Tarfalajaure transect varied along PC2, which had strong

negative correlations with the remaining metals (Pb, Ni, Cr, and Cu). Concentrations of these metals were higher in samples from Tarfalajaure compared to Storglaciären, while it was the opposite for the metals correlated with PC1.

Concentration of individual metals did not vary along the transects (Figure 4.5, Appendix 14). Soil samples generally had higher metal concentrations compared to sediments. There was no particular pattern in total metal concentrations along the transects either: total metal concentration along the Tarfalajaure transect increased downstream, while it decreased along the Isfallsglaciären transect. However, there was some variation between sediment and soil samples. Soil samples from Tarfalajaure and Storglaciären had higher concentrations of all measured metals compared to sediment samples. On the contrary, at Isfallsglaciären, soil samples had lower metal concentrations compared to sediments. There was no pattern in sediment or soil concentrations along the transects. Overall, sediment sample KG-MW-1308 had the lowest total metal concentration (7784 mg/kg), while soil samples collected at Storglaciären had the highest values (25105 mg/kg in SG-RB-0808 and 24570 mg/kg in SG-M2-0808).

#### 4 Soil development at Tarfala valley, Sweden

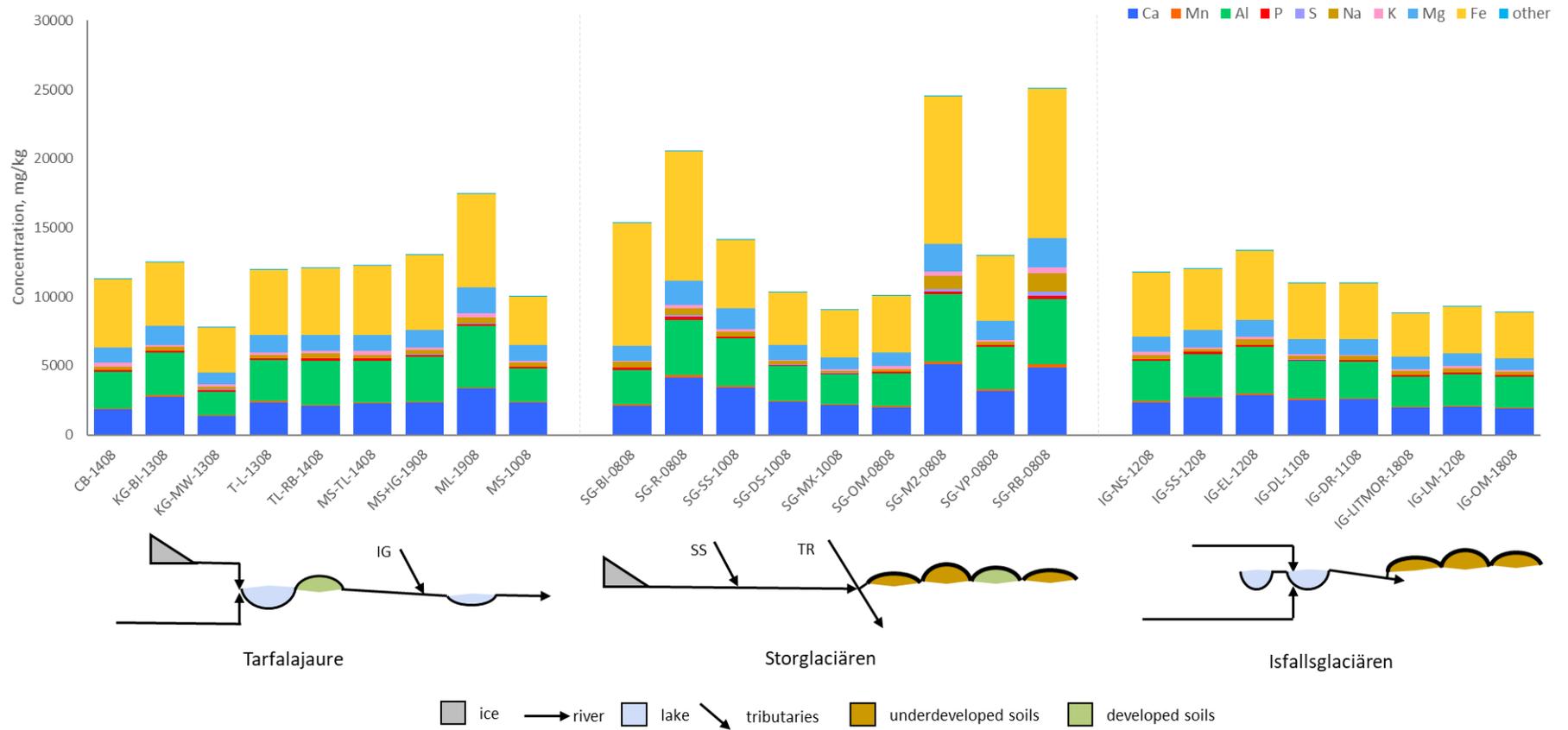


Figure 4.5. Mean concentration of metals (mg/kg) along the Tarfalajaure, Storglaciären and Isfallsglaciären transects in Sweden. Other metals include Cr, Co, Ni, Cu, Zn, Pb and Cd. Charts at the bottom of the graph show downstream (left to right) transect profiles. IG stands for Isfallsglaciären, SS – Storglaciären south stream, TR – Tarfalajaure.

## 4.2.2 Biomarkers

TOC concentrations in many samples from the Tarfala valley were too low for the successful extraction of organic biomarkers. Therefore, to avoid performing analysis on samples with low to no detectable amounts of organic biomarkers, only selected samples were chosen for further analysis. Biomarker analysis was carried out on samples collected along the Tarfalajaure transect, representing an older catchment, as well as some other samples at Storglaciären, representing a recent, younger catchment.

Samples from the Tarfalajaure transect were chosen to represent soil development along the lake transect (see Figure 4.2 for location of selected samples on a map): from the meltwater inflow (KG-MW-1308, mean TOC=0.01±0.006%, n=3) to the sediment side of the lake bank (T-L-1308, mean TOC=0.44±0.18%, n=3), a vegetated patch at the other bank (TL-RB-1408, mean TOC=0.65±0.14%, n=3) and further to the river outflow (MS-TL-1408, mean TOC=0.29±0.21%, n=3). These samples are believed to represent soil derived OC accumulation from recently deglaciated areas (sample KG-MW-1308) to areas that have been exposed for a long time (samples T-L-1308, TL-RB-1408 and MS-TL-1408).

Samples from Storglaciären (and Isfallsglaciären) had very low TOC values (< 0.14%). Mean TOC value of samples from Storglaciären (0.04±0.07%, n=9) were not significantly different than those from Isfallsglaciären (0.03±0.03%, n=8). Therefore, it was decided to investigate only some samples from Storglaciären for organic biomarkers. At Storglaciären, one replicate from samples with low TOC (SG-R-0808, mean TOC=0.008±0.003%, n=3; SG-DS-1008, 0.08±0.12%, n=3; SG-M2-0808, 0.02±0.03%, n=3) and highest TOC (SG-VP-0808, mean TOC=0.13±0.11%, n=3) values were chosen for further organic biomarker analysis (see Figure 4.2 for location of selected samples on a map).

### 4.2.2.1 BHP concentration and distribution pattern

Overall, up to 28 individual BHPs were identified in soil and sediment samples from the Tarfala valley, with the relative concentration of total BHPs ranging from 30 to 8705 µg/g TOC with a mean of 4301±3427 µg/g TOC (no BHPs were identified in sample SG-DS-1008) (Appendix 15), while mean absolute concentration was 12±15 µg/g sediment. The BHPs were grouped in five main classifications: BHT, amino, sugar, aminosugar and soil markers

(specified in Appendix 15). Among the non-soil specific BHPs, BHT was the most abundant individual BHP, with a mean of  $1932 \pm 1418$   $\mu\text{g/g}$  TOC (11 to 3679  $\mu\text{g/g}$  TOC), followed by Me-BHT ( $536 \pm 332$   $\mu\text{g/g}$  TOC), 35-aminotriol ( $394 \pm 350$   $\mu\text{g/g}$  TOC), BHT glucosamine ( $325 \pm 211$   $\mu\text{g/g}$  TOC) and BHT cyclitol ether ( $293 \pm 241$   $\mu\text{g/g}$  TOC). Adenosylhopane ( $535 \pm 346$   $\mu\text{g/g}$  TOC), followed by adenosylhopane type 3 ( $259 \pm 165$   $\mu\text{g/g}$  TOC) were the main soil marker BHPs. Other adenosylhopane type structures were present in smaller concentrations.

BHPs were present in all analysed samples from the Tarfalajaure transect (Figure 4.6, Appendix 15). Total BHP concentration along the Tarfalajaure downstream transect increased from a glacier meltwater stream (KG-MW-1308) to a lake bank (T-L-1308) and further to the vegetated patch at the lake (TL-RB-1408) and the riverbank outflowing from the Tarfala lake (MS-TL-1408) (Figure 4.6). The  $R'_{\text{soil}}$ , used to trace accumulation of soil marker BHPs, had a consistent upward trend along the downstream transect at the valley, increasing from 0.12 to 0.34 (Figure 4.6), indicating increasing relative/fractional concentrations of soil markers.

In contrast, BHPs were only detected in some samples from the Storglaciären glacier (Figure 4.6, Appendix 15). No BHPs were detected in the sediment sample from the downstream location of the river SG-DS-1008, sediment samples SG-R-0808 and SG-M2-0808 had low relative concentration of total BHPs of 30 and 84  $\mu\text{g/g}$  TOC, respectively, while the soil sample SG-VP-0808 had much higher BHP concentration of 8705  $\mu\text{g/g}$  TOC. The  $R'_{\text{soil}}$  was higher in the SG-VP-0808 (0.21) than SG-R-0808 (0.07), indicative of accumulation of soil derived OC at the vegetated riverbank (SG-VP-0808) compared to the sediment sample from the river (SG-R-0808) (Figure 4.6).

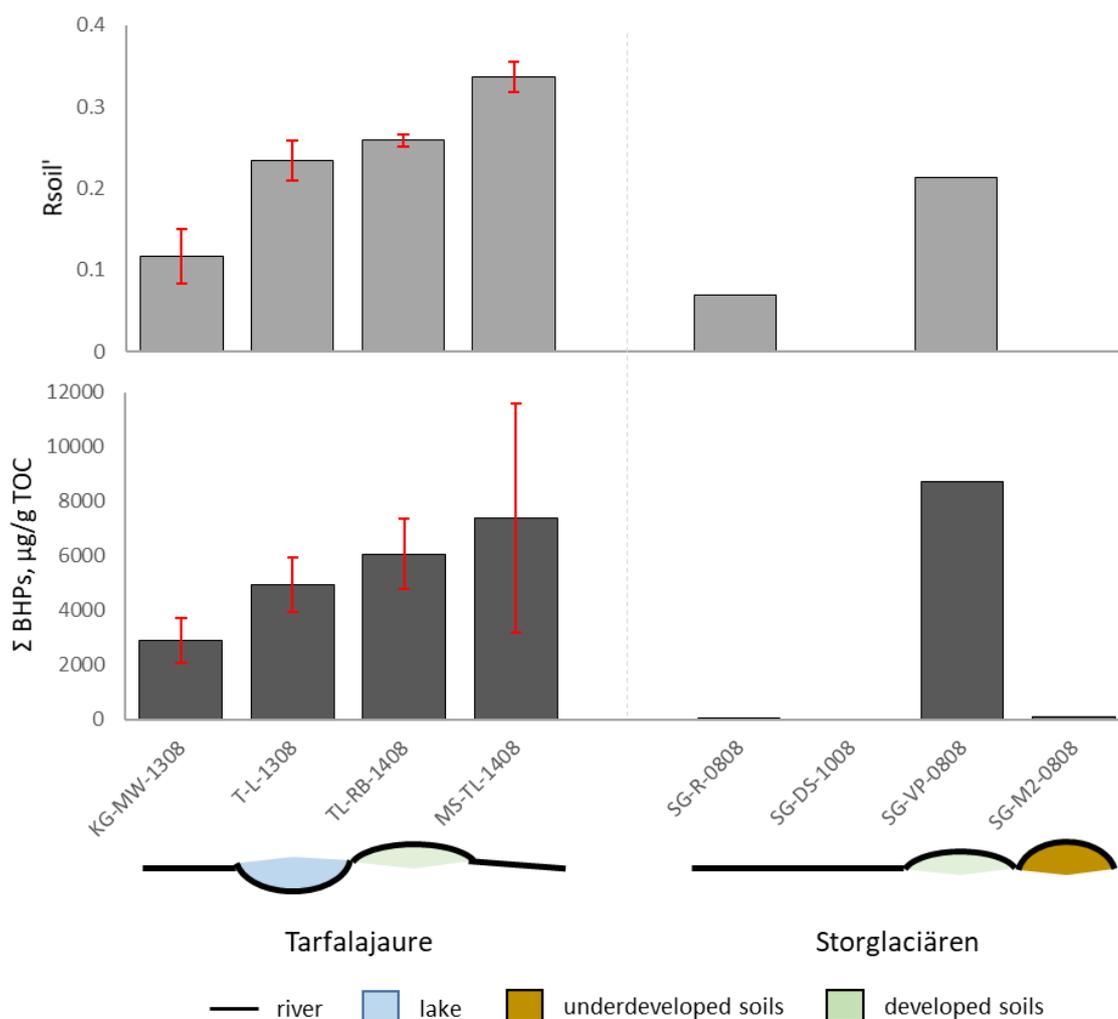


Figure 4.6. Average distribution of total BHPs ( $\mu\text{g/g}$ ) and  $R'_{soil}$  in selected samples along the Tarfalajaure and the Storglaciären glacier transects in Sweden. Error bars represent  $\pm$  one standard deviation on three field replicate samples collected from individual sampling locations at the Tarfalajaure. Only one field replicate per sampling location from Storglaciären was analysed for organic biomarkers. Charts at the bottom of the graph show downstream (left to right) transect profiles.

Samples from the Tarfalajaure transect consisted of all five main groups (Figure 4.7). BHT was the most abundant individual compound in all samples with a mean relative concentration of  $2123 \pm 1908 \mu\text{g/g TOC}$  (Appendix 15), which constituted 36-56% of total BHPs. After an initial increase from  $1624 \mu\text{g/g TOC}$  at KG-MW-1308 to  $2761 \mu\text{g/g TOC}$  at T-L-1308, BHT concentration stayed roughly the same ( $2667$ - $2761 \mu\text{g/g TOC}$ ) (Appendix 15), suggesting an increase in BHPs due to the accumulation of soil marker BHPs. Soil BHPs varied depending on sample location in the range of  $219$ - $1563 \mu\text{g/g TOC}$  (mean value of  $610 \pm 623 \mu\text{g/g TOC}$ ), equivalent to 8-21% of total BHPs. Following the same pattern, the concentration of soil marker BHPs increased along the downstream transect (Figure 4.7).

Soil marker BHPs were dominated by adenosylhopane (up to 861  $\mu\text{g/g}$  TOC) and adenosylhopane type 3 (up to 498  $\mu\text{g/g}$  TOC).

As for the samples from Storglaciären, BHT concentration in samples where BHPs were identified varied in the range of 11 to 4592  $\mu\text{g/g}$  TOC, equivalent of 37-100% of total BHPs (only BHT was identified in sample SG-M2-0808 from a larger moraine (Appendix 15, Figure 4.7)). Soil BHPs constituted only 0-12% of total BHPs (Figure 4.7).

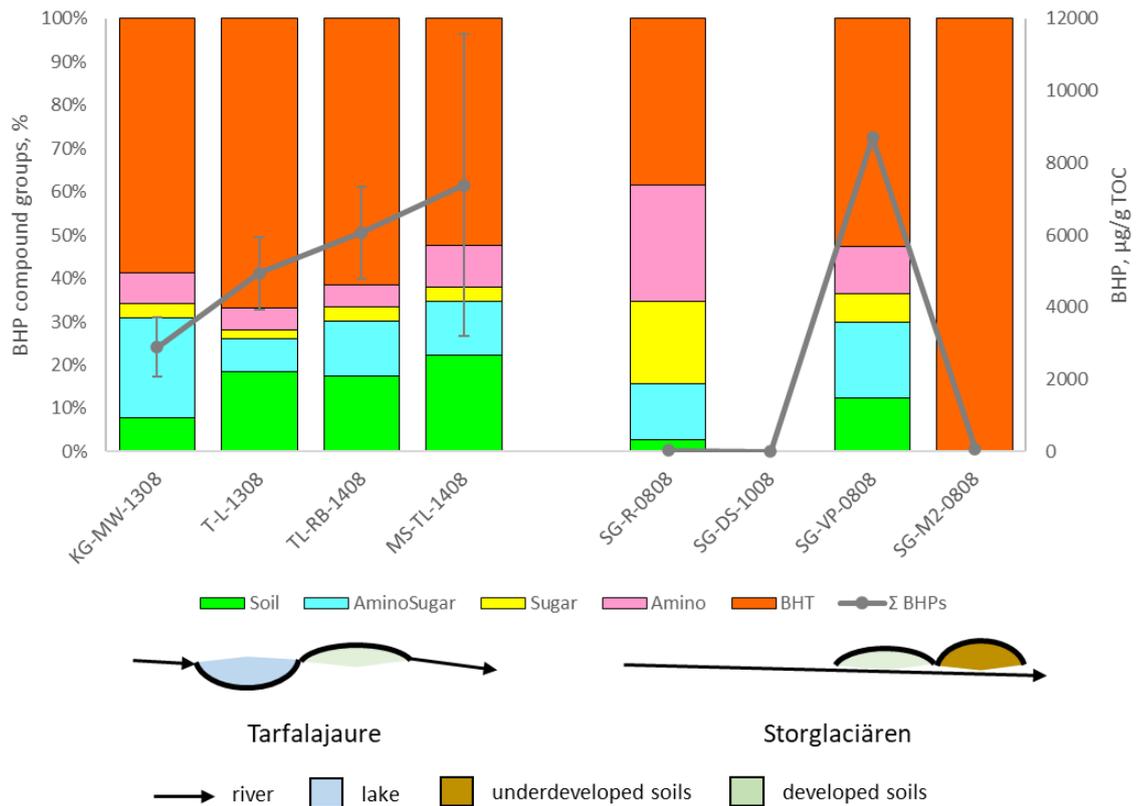


Figure 4.7. Relative concentration of total BHPs (grey circles and lines) and fractional abundance of BHP compound groups (BHTs, aminos, sugars, aminosugars and soil BHPs; coloured bars) in selected samples along the Tarfalajaure and the Storglaciären glacier transect in Sweden. Error bars represent  $\pm$  one standard deviation on three field replicate samples collected from individual sampling locations at the Tarfalajaure. Only one field replicate per sampling location from Storglaciären was analysed for organic biomarkers. Charts at the bottom of the graph show downstream (left to right) transect profiles.

#### 4.2.2.2 GDGT concentrations and distribution along the transects

Besides BHPs, GDGTs were also investigated. In samples with detectable GDGTs, relative concentration of total GDGTs varied from 164 to 525  $\mu\text{g/g}$  TOC, having a mean of  $403 \pm 163$   $\mu\text{g/g}$  TOC (Appendix 15), while mean absolute concentration was  $0.77 \pm 1.19$   $\mu\text{g/g}$  sediment.

There was a wide range of branched GDGTs and crenarchaeol GDGT (cren) across the samples from the Tarfala valley (Figure 4.8), though GDGTs were not detected in all samples, with no GDGTs detected in samples KG-MW-1308, SG-R-0808, SG-DS-1008 and SG-M2-0808.

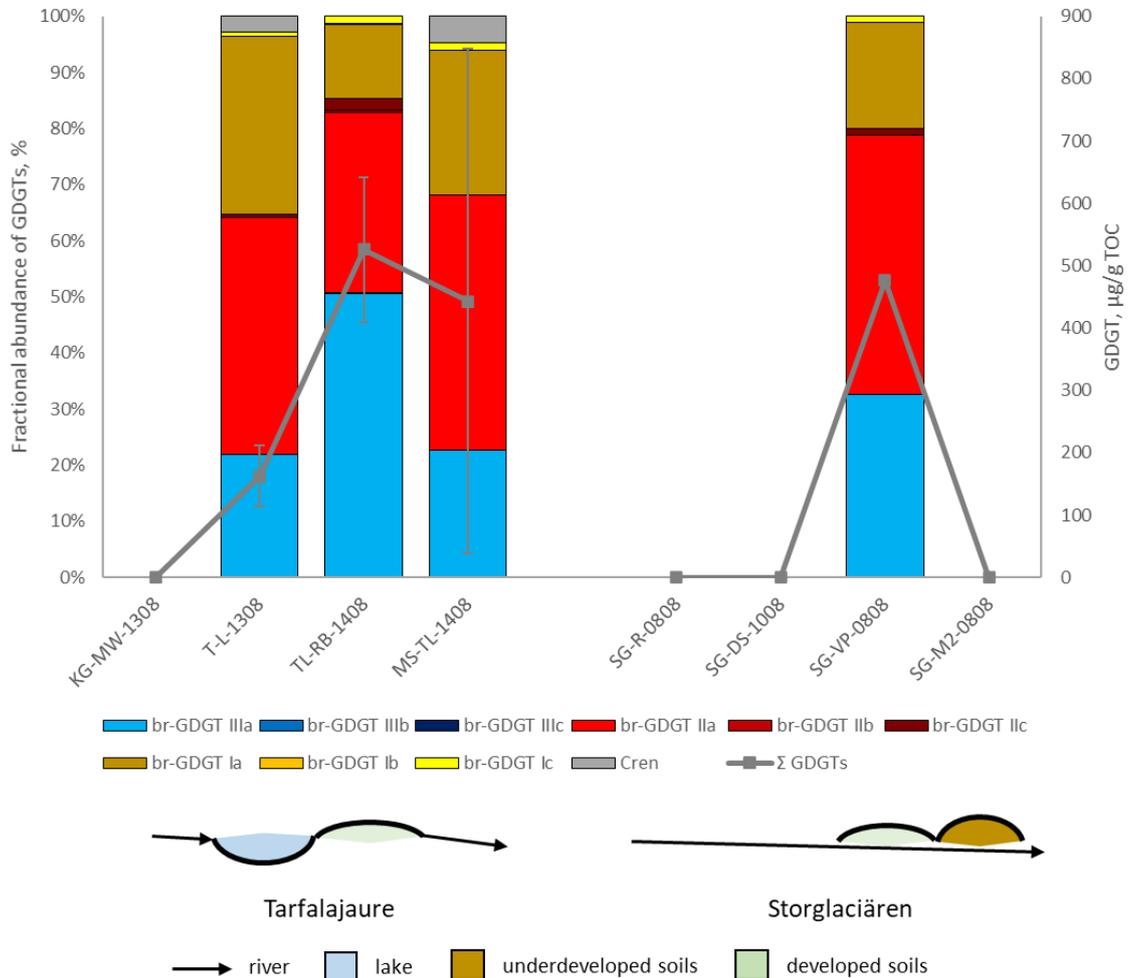


Figure 4.8. Relative concentration of total GDGTs (grey circles and lines) and fractional abundance of branched GDGT compounds and crenarchaeol GDGT (coloured bars) in selected samples along downstream transects of the Tarfalajaure and the Storglaciären glacier in Sweden. TL-RB-1408, T-L-1308, MS-TL-1408 SG-VP-0808 and SG-M2-0808 are soil samples; KG-MW-1308, SG-R-0808 and SG-DS-1008 – sediment. Error bars represent  $\pm$  one standard deviation on three field replicate samples collected from individual sampling locations at the Tarfalajaure. Only one field replicate per sampling location from Storglaciären was analysed for organic biomarkers. Charts at the bottom of the graph show downstream (left to right) transect profiles.

Ia, IIa and IIIa compounds were the most abundant brGDGTs with relative concentrations varying in the range of 52-114 (mean value of  $81 \pm 27$  µg/g TOC), 69-221 (mean= $165 \pm 68$  µg/g TOC) and 36-266 (mean= $139 \pm 97$  µg/g TOC) µg/g TOC, respectively. Ib, IIb, Ic, IIc and IIIc

compounds were present in smaller quantities in the range from 1 to 11  $\mu\text{g/g}$  TOC. Some samples also had minor amount of crenarcheol (samples T-L-1308 and MS-TL-1408).

Three out of four samples from Tarfalajaure transect had detectable amounts of GDGTs. The highest concentration was detected in sample TL-RB-1408, collected at the vegetated bank at the Tarfala lake. When plotted against the downstream distribution of samples, no overall trend in GDGTs along the transect was observed (Figure 4.8). As for the samples from Storglaciären, GDGTs were detected only in sample SG-VP-0808.

#### **4.2.2.2.1 GDGT proxies**

GDGTs can be used as a proxy to study paleoenvironmental changes, therefore, GDGT-based soil pH and mean annual temperature (MAT) were analysed. Soil pH varied from 4.4 to 6.3, while MAT fluctuated between  $-0.3$  and  $1.8$   $^{\circ}\text{C}$  (Figure 4.9). Along the Tarfalajaure transect temperature decreased downstream. Distribution of either of the proxies along the Storglaciären transect could not be seen because GDGTs were detected only in one sample from Storglaciären. Given that GDGTs were only detected in some samples, it might not be viable to use these proxies.

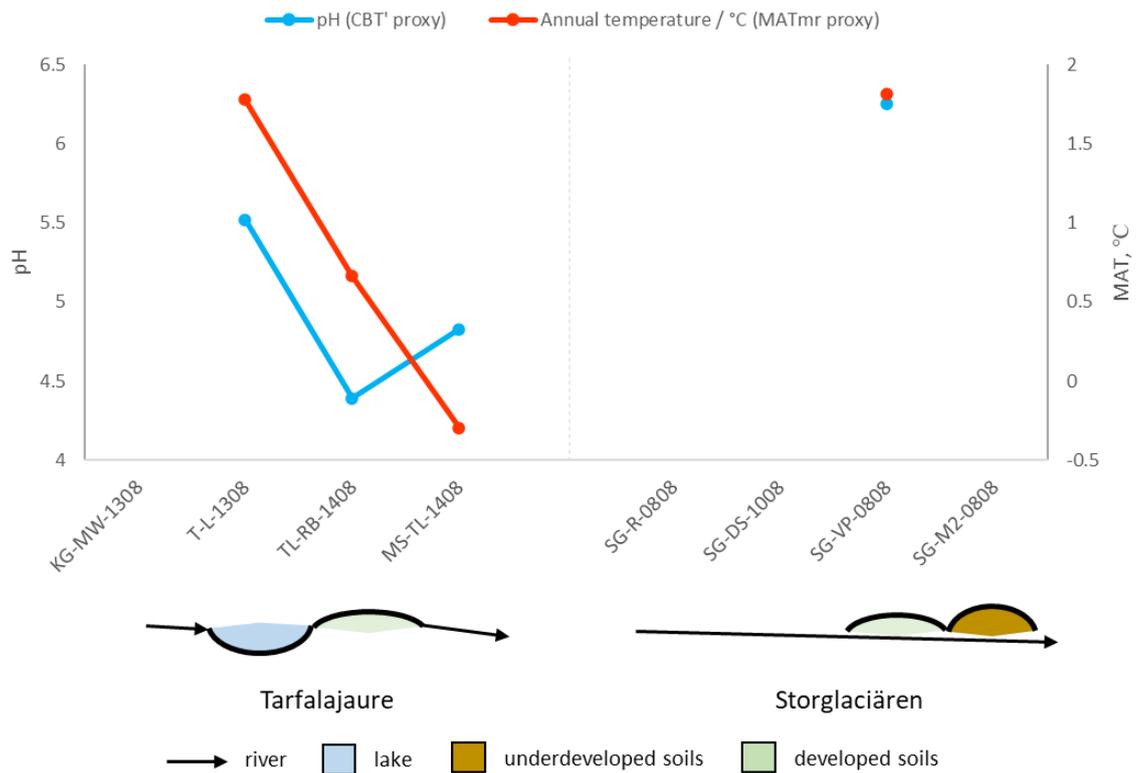


Figure 4.9. Downstream distribution of GDGT proxies, soil pH and annual mean air temperature (MAT, °C) along the Tarfalajaure and Storglaciären transects in Sweden.

#### 4.2.2.3 Relationship between TOC, BHPs and GDGTs

In order to determine if organic biomarkers are related to OC concentrations a comparison between TOC and absolute concentration of organic biomarkers was plotted (Figure 4.10). There was a linear relationship between TOC (%) and absolute concentration of total BHPs ( $\mu\text{g/g}$  sediment) (Figure 4.10a) across sites in Tarfala valley, Sweden with an  $R^2$  value of 0.96 and Pearson correlation of 0.98, indicative of a strong correlation between the two variables. Similarly, there was a strong positive relationship between TOC and total brGDGTs (Figure 4.10b) with an  $R^2$  value of 0.80 and Pearson correlation of 0.89. Linear regression of total BHPs ( $\mu\text{g/g}$  sediment) versus total brGDGTs ( $\mu\text{g/g}$  sediment) across all sample points (Figure 4.10c) also revealed strong positive relationship ( $R^2=0.88$ , Pearson correlation=0.94).

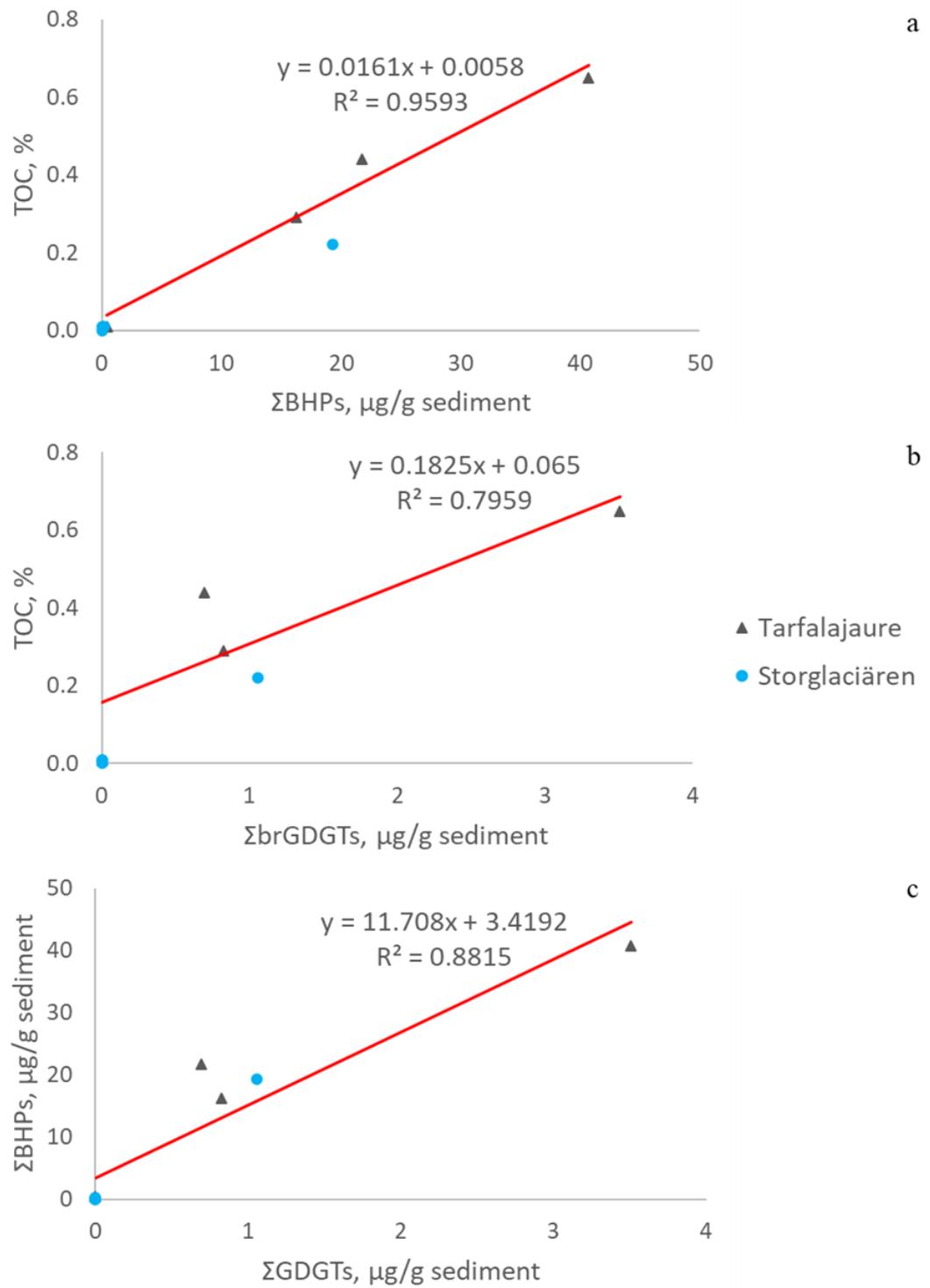


Figure 4.10. Relationship between total organic carbon (TOC) (%) and (a) total BHPs ( $\mu\text{g/g}$  sediment), (b) total brGDGTs ( $\mu\text{g/g}$  sediment) and (c) total BHPs ( $\mu\text{g/g}$  sediment) and total brGDGTs ( $\mu\text{g/g}$  sediment) across sites at the Tarfala valley, Sweden.

### 4.2.3 Microbial analysis

#### 4.2.3.1 Sample selection and DNA concentration

As with organic biomarkers, only selected samples from the Tarfala valley were investigated for microbial communities, with 10 samples from Tarfalajaure, including some replicates, and three samples from Storglaciären. From the Tarfalajaure transect, the same samples as the ones for organic biomarker analyses were selected for microbial analysis, while one replicate of sediment sample (SG-DS-1008) and two replicates of soil sample (SG-VP-0808) were chosen from Storglaciären (see Figure 4.2 for the location of selected samples on a map). Only a few samples from Storglaciären were selected for microbial analysis based on the earlier findings of low OC content and lack of detectable organic biomarkers, as well as low DNA count and lack of visible PCR bands. Samples that were selected for analysis of microbial community had sufficient DNA and produced a visible band in PCR. The majority of samples (11) were soil samples, with two being sediments.

DNA concentration of selected samples from the Tarfala valley ranged from BDL to 30.4 ng/ml (Appendix 18). Generally, sediment samples had lower values (BDL) compared to soils ( $13.59 \pm 10.66$  ng/ml), having a statistically significant difference by the type of sample (Welch's t-test, p-value < 0.05).

#### 4.2.3.2 Chemical characterisation of soils and sediments investigated for microbial community structure

To identify chemical differences along transects and between soil and sediment samples, PCA was conducted on the environmental data from those samples specifically chosen for further microbial analysis (Figure 4.11). PC1 explained 43.9% of total variance and had the strongest negative correlation with Al, TOC, TC, Fe, Zn, Na and Cd, followed by Pb, N, P, Ni, Cr and Mg. PC2 (23.0% of total variance) had a strong negative correlation with Ca, Mn and Mg, negative correlation with Co and Cr, and positive correlation with Pb, S, Cu and P.

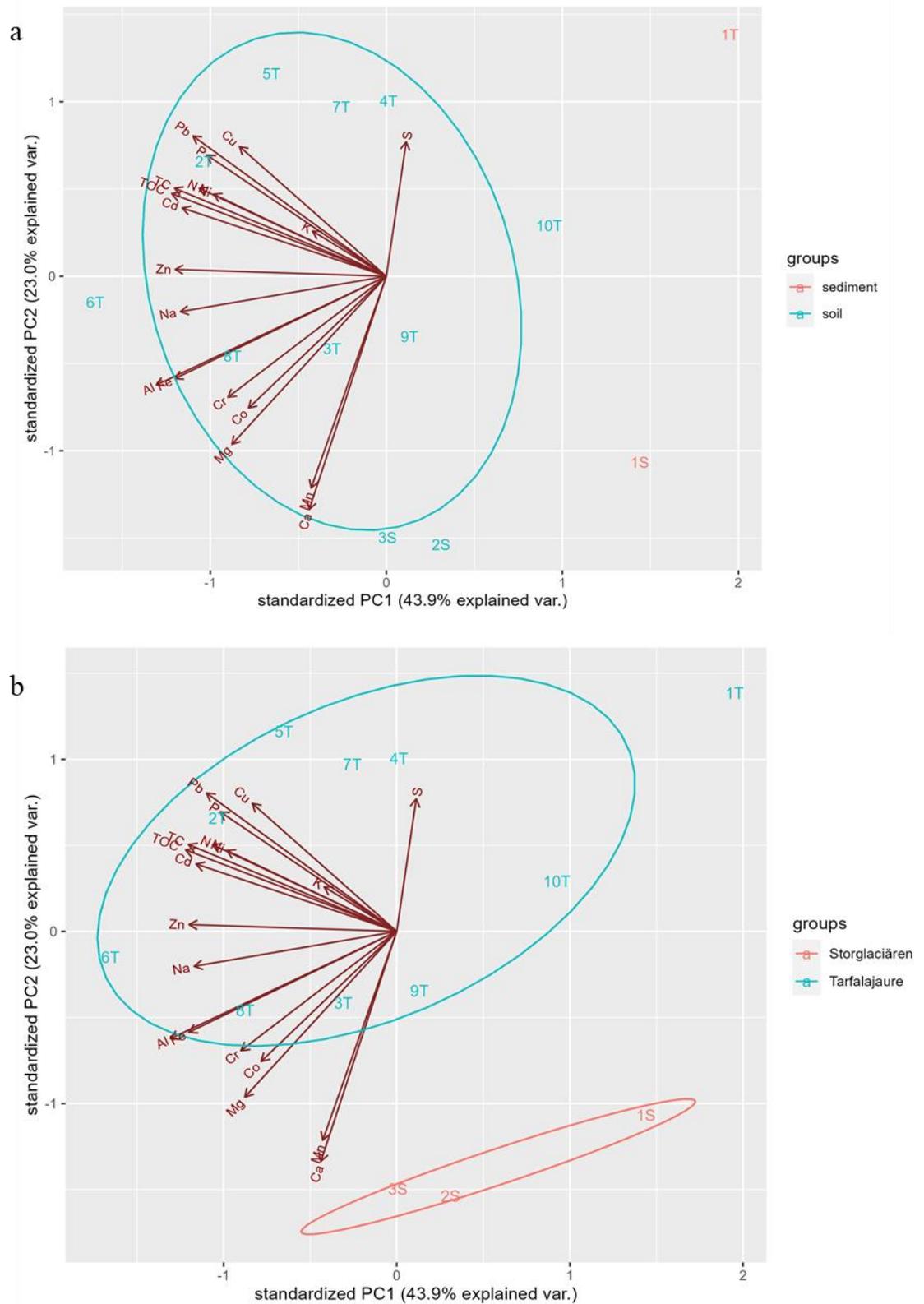


Figure 4.11. PCA ordination plots illustrating the discrimination between samples investigated for microbial community structure from Tarfalajaure and Storglaciären transects according to chemical properties by the (a) sample type and (b) downstream transect. The input data was standardised to have zero mean and variance one. The ellipses represent the core area added by confidence interval of 68%. Sample codes are given in Table 4.3.

Table 4.3. Codes of selected sample names from Tarfala valley, Sweden shown Figure 4.11, Figure 4.12 and Figure 4.13. T stands for Tarfalajaure, S – Storglaciären.

Cod e	Sample	Type	Cod e	Sample	Type
1T	KG-MW-1308_2	sediment	8T	MS-TL-1408_1	soil
2T	T-L-1308_1	soil	9T	MS-TL-1408_2	soil
3T	T-L-1308_2	soil	10T	MS-TL-1408_3	soil
4T	T-L-1308_3	soil	1S	SG-DS-1008_3	sediment
5T	TL-RB-1408_1	soil	2S	SG-VP-0808_1	soil
6T	TL-RB-1408_2	soil	3S	SG-VP-0808_2	soil
7T	TL-RB-1408_3	soil			

Moreover, even though only two sediment samples were chosen for further microbial community analysis, PCA indicated that sediment samples were plotted to the right of soil samples (Figure 4.11a), being significantly different (PERMANOVA,  $p$ -value < 0.05).

When viewed by transect, PCA showed that samples collected along the Tarfalajaure transect were different to the ones collected at the Storglaciären (Figure 4.11b), though according to PERMANOVA analysis there was no statistically significant difference between the samples from two transects ( $p$ -value > 0.05). These observations on the sub-set of samples due to microbial analysis were contrary to what was detected when analysing all samples collected from the Tarfala valley (Figure 4.1). Samples from Tarfalajaure were scattered throughout the plot, above samples from Storglaciären. Interestingly, replicates (2T, 3T and 4T) were not clustered together (Figure 4.11b, Table 4.3).

### 4.2.3.3 Microbial community structure

#### 4.2.3.3.1 NMDS

NMDS ordination plot of family-level bacterial communities (Figure 4.12) showed that soil samples formed a tight cluster at the top right corner of the plot, while sediment samples were more widely dispersed. PERMANOVA analysis also showed that there was a statistically significant difference between family-level bacterial communities of soil and sediment samples ( $p$ -value < 0.05).

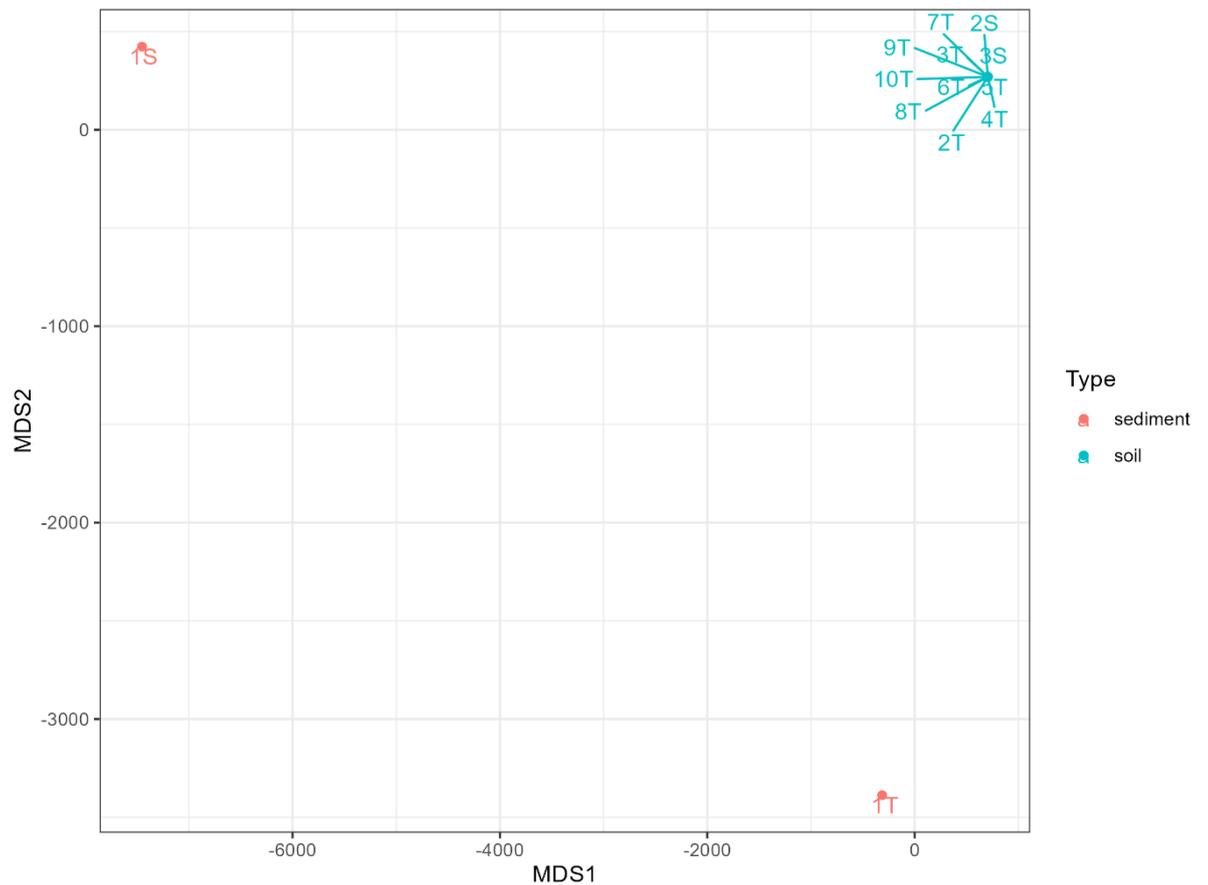


Figure 4.12. Two-dimensional NMDS plots of family-level bacterial communities in samples from Tarfala valley by sample type. When the data values were larger than common abundance class scales, Wisconsin double standardisation was performed; when values looked very large, sqrt transformation was also performed; and distance matrix based on Bray–Curtis dissimilarity was obtained. Sample codes are given in Table 4.3.

Further, NMDS ordination plot (Figure 4.13) also demonstrated clear separation of bacterial family-level distribution of samples by transect. Samples from Storglaciären were located in the top right corner of the plot compared to samples from Tarfalajaure, which were positioned mostly in the bottom half of the plot (Figure 4.13b). There was a statistically significant difference between family-level bacteria in samples by transect (PERMANOVA analysis,  $p$ -value < 0.05). Compared to PCA plots, replicates in the NMDS plot (Figure 4.13b) were located closer to each other (2T, 3T and 4T; 5T, 6T and 7T; 8T, 9T and 10T; 2S and 3S, see Table 4.3 for sample names).

4 Soil development at Tarfala valley, Sweden

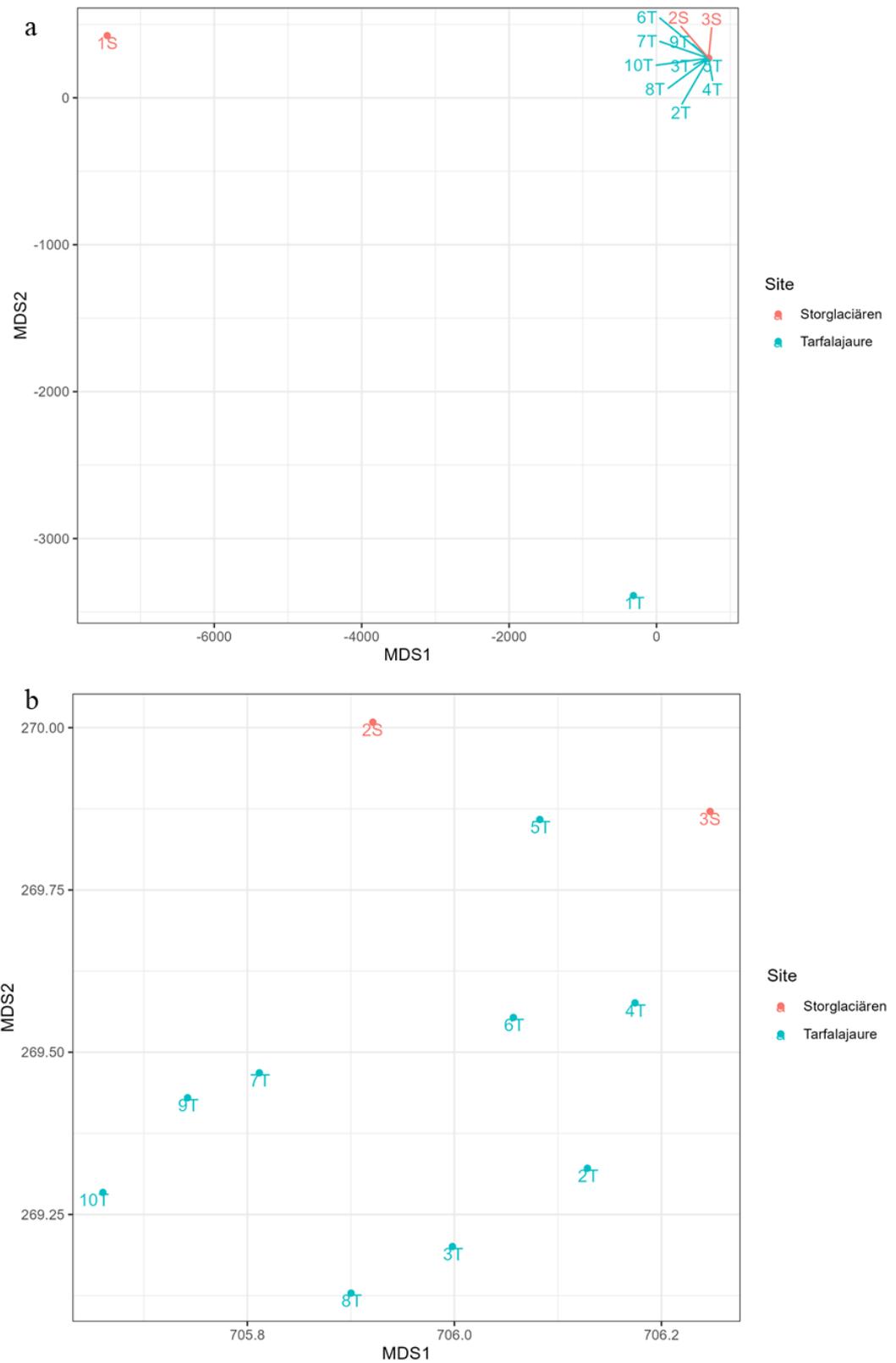


Figure 4.13. (a) Full scale and (b) zoomed-in two-dimensional NMDS plots of family-level bacterial communities in samples from Tarfala valley in Sweden by site. When the data values were larger than common abundance class scales, Wisconsin double standardisation was performed; when values looked very large, sqrt transformation was also performed; and distance matrix based on Bray–Curtis dissimilarity was obtained. Sample codes are given in Table 4.3.

#### 4.2.3.3.2 Alpha diversity

In order to evaluate the microbial alpha diversity in each sample, Shannon–Weiner diversity index and Chao1 species richness estimate values were plotted (Figure 4.14). The Chao1 species richness estimate indicated that sediment samples had low bacterial diversity, while the Shannon–Weiner diversity index indicated low diversity only in the sediment sample from Storglaciären (Figure 4.14). Moreover, both the Shannon–Weiner diversity index and Chao1 species richness estimate showed that one of the soil samples (SG-VP-0808\_2) from Storglaciären had higher bacterial diversity compared to all the other samples. As for the samples from Tarfalajaure, sample TL-RB-1408 had the highest diversity, followed by MS-TL-1408 and T-L-1308.

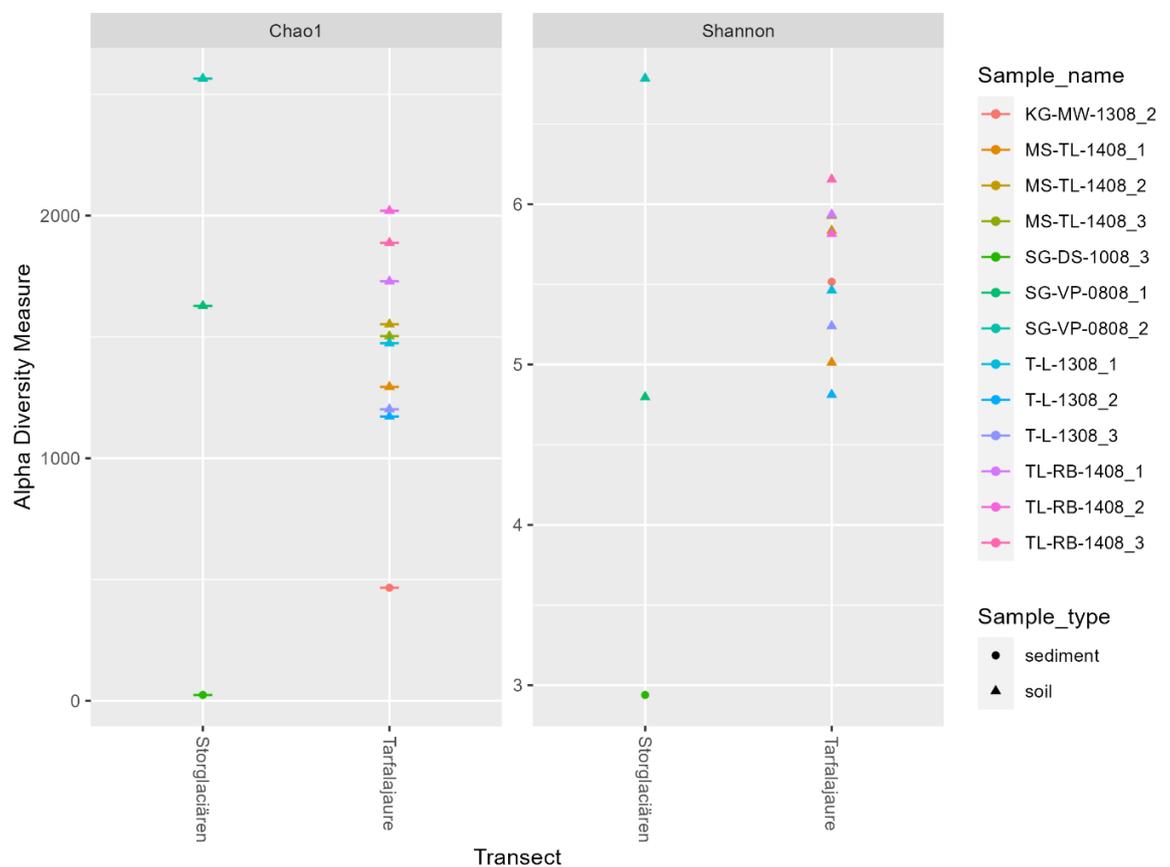


Figure 4.14. Alpha diversity (Shannon) and richness (Chao1) variation in soil and sediment samples from Tarfalajaure and Storglaciären transects in Sweden.

#### 4.2.3.3.3 Phylum-level community composition

Bacterial 16S rRNA gene derived phyla distribution along the Tarfalajaure and Storglaciären transects is presented in Figure 4.15a. The phyla that appeared consistently among all

samples were candidate phylum AD3, Acidobacteria, Actinobacteria, Chloroflexi, Planctomycetes and Proteobacteria. The most abundant phyla in samples from Sweden were candidate phylum AD3 ( $16.8 \pm 13.6\%$ ), Acidobacteria ( $17.2 \pm 10.5\%$ ), Chloroflexi ( $15.7 \pm 6.7\%$ ), Proteobacteria ( $15.4 \pm 13.6\%$ ), and Planctomycetes ( $7.0 \pm 3.5\%$ ). There was a clear difference in phyla distribution between the two transects. In samples from the Tarfalajaure the most abundant phyla were candidate phylum AD3 ( $21.3 \pm 12.3\%$ ), Chloroflexi ( $17.7 \pm 5.7\%$ ), Acidobacteria ( $15.2 \pm 4.2\%$ ), and Proteobacteria ( $11.7 \pm 9.1\%$ ). Meanwhile, Proteobacteria ( $27.8 \pm 21.1\%$ ), Acidobacteria ( $24.0 \pm 22.1\%$ ), Actinobacteria ( $10.9 \pm 5.1\%$ ), and Chloroflexi ( $9.2 \pm 6.2\%$ ) were dominant phyla in samples from Storglaciären. Additionally, PERMANOVA analysis showed a significant difference ( $p$ -value  $< 0.05$ ) in distribution of phyla between the two transects. Sediment samples had significantly different phyla distribution compared to soils (PERMANOVA,  $p$ -value  $< 0.05$ ), having higher relative abundance of Proteobacteria ( $44.2 \pm 11.2\%$ ) and Bacteroidetes ( $12.3 \pm 5.2\%$ ).

#### **4.2.3.3.4 Family-level community composition**

Bacterial family-level distribution is presented in Figure 4.15b. The most abundant family-level bacteria across the samples from the Tarfala valley were unclassified at family-level ABS-6 (candidate phylum AD3) ( $11.3 \pm 11.4\%$ ), unclassified RB41 (Acidobacteria) ( $6.2 \pm 10.1\%$ ), unclassified JG37-AG-4 (candidate phylum AD3) ( $4.6 \pm 5.0\%$ ). The “other” category refers to a total of family-level bacteria that had less than 0.5% relative abundance in all samples collected in Sweden. Unclassified at family-level ABS-6 (candidate phylum AD3) ( $14.3 \pm 11.3\%$ ), unclassified JG37-AG-4 (candidate phylum AD3) ( $6.0 \pm 4.9\%$ ) and Ktedonobacteraceae (Chloroflexi) ( $3.7 \pm 4.1\%$ ) were the most abundant in samples from the Tarfalajaure transect, while unclassified RB41 (Acidobacteria) ( $15.2 \pm 20.6\%$ ), Comamonadaceae (Proteobacteria) ( $9.7 \pm 13.9\%$ ) and S24-7 (Bacteroidetes) ( $4.4 \pm 7.6\%$ ) were abundant at Storglaciären. Similar to phyla, bacterial family-level distribution of samples at the Tarfalajaure was significantly different from that at Storglaciären (PERMANOVA analysis,  $p$ -value  $< 0.05$ ). Sediment samples had higher relative abundance of Comamonadaceae (Proteobacteria) ( $16.8 \pm 12.6\%$ ), as well as lower relative abundance of other bacteria, having statistically significant difference compared to soil samples from the Tarfala valley (PERMANOVA,  $p$ -value  $< 0.05$ ).

Replicates of samples from Tarfalajaure transect (T-L-1308, TL-RB-1408 and MS-TL-1408) had similar phyla (Figure 4.15a) and family-level bacterial compositions (Figure 4.15b), which was expected based on their close proximity to each other on the NMDS plot (Figure 4.13b). Similarly, though replicate SG-VP-0808\_1 had higher concentrations of unclassified at family-level RB41 (Acidobacteria), replicates of this soil sample from Storglaciären (SG-VP-0808) seemed to have similar phyla and family-level bacteria.

#### 4 Soil development at Tarfala valley, Sweden

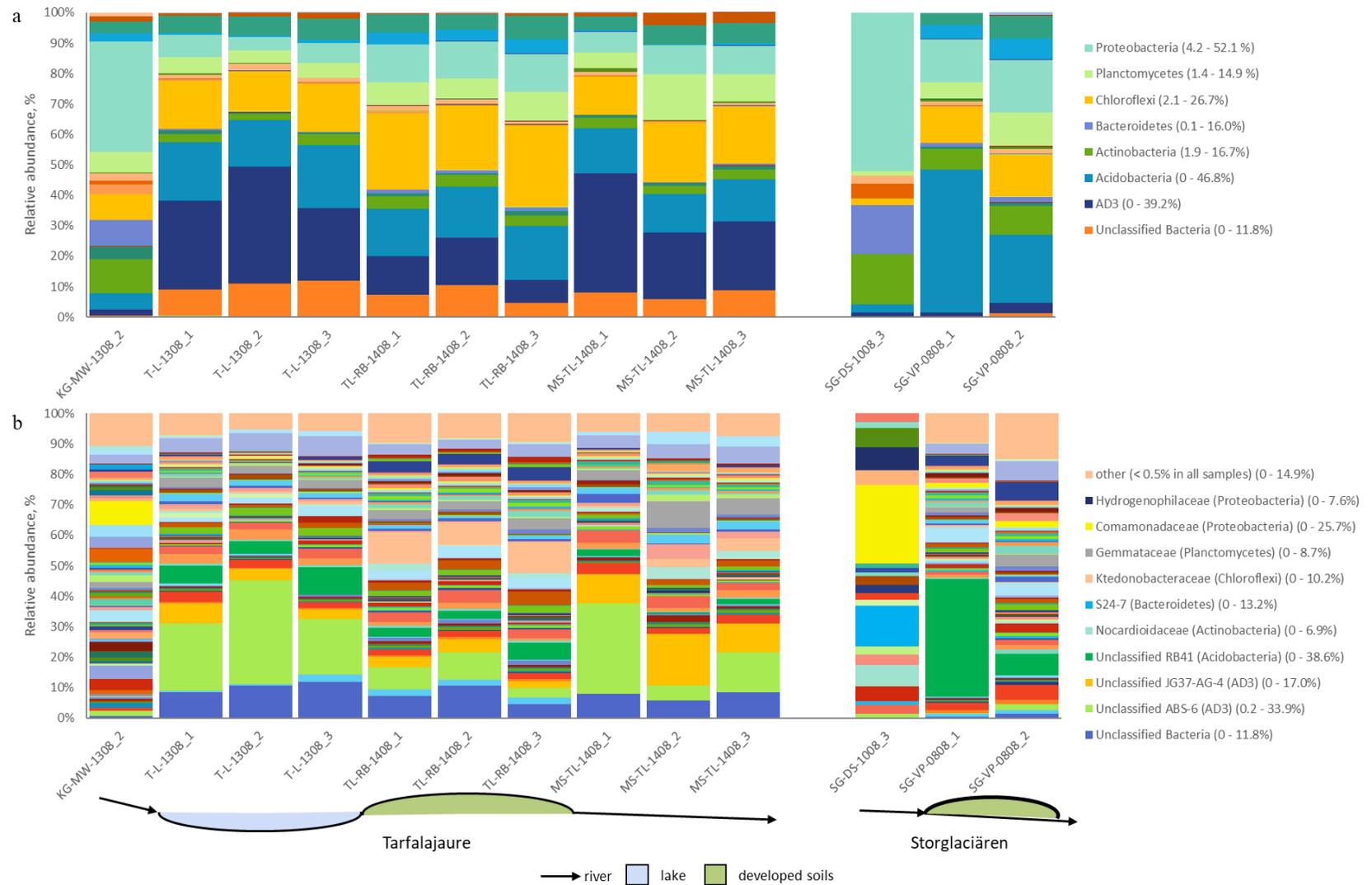


Figure 4.15. Relative abundance of bacterial taxa: (a) all assigned taxa at phylum level where possible and (b) all assigned taxa shown to the highest resolution possible down to the family level where possible (phylum level given in parentheses) for each sample along the Tarfalajaure and Storglaciären transects in Sweden. Selected taxa of high abundance given in parentheses.

## 4.3 Discussion

The overall aim of this study was to understand the distribution of OC, N, metals, organic biomarkers, and microbial community in deglaciated areas, which can inform the knowledge of accumulation of soil derived OC in the catchments. This chapter seeks to understand how these parameters behave in the barren catchment of the Tarfala valley. Within the catchment, three transects were studied, with Storglaciären and Tarfalajaure studied in greater depth.

In this chapter, the following hypotheses were tested: (1) since Tarfalajaure has been deglaciated for the longest period, samples from the Tarfalajaure transect have higher TOC, TN, and organic biomarker concentrations, as well as higher bacterial diversity, compared to samples collected from the more recently deglaciated Storglaciären glacier foreland; (2) concentration of OC and organic biomarkers increase along the downstream transects, from sediments to soils; (3) in older soils microbial community diversity is higher, with bacterial taxa more representative of stable soil systems compared to sediments.

### 4.3.1 TOC, TN, and C/N compared to other Arctic catchments

Analysis of TOC concentrations of soil and sediment samples along the Tarfala valley indicated that the investigated landscape is poor in TOC (0 - 1.08±0.09%) compared to other glacier forelands. For example, Wietrzyk et al. (2018) reported TOC concentration of 0.69-3.81% in samples from Svalbard collected 0-96 years after the glacier retreat, while Prietzel et al. (2013) found TOC concentrations of 0.15-7.97% in Switzerland after 15 to over 700 years of glacier retreat and 0.20-41.23% in Tibetan plateau after 0-120 years of glacier recession. The reason for Sweden having a lower TOC concentration may be due to it being a bare landscape, with thin soil cover. Wietrzyk et al. (2018) suggested that TOC concentration is a function of time and distance from the glacier forehead, i.e., time of exposure after deglaciation. However, since the time of exposure at the Tarfala valley is similar to that in the abovementioned studies, accumulation of soil OC might be due to geology, rather than age of the catchment. In line with this, Prietzel et al. (2013) suggested that OC content is dependent on soil-forming moraine material and physical exposure

processes these moraines go through. This highlights the importance of investigating samples in more detail to gain a better understanding of processes within the catchment.

Moreover, as expected, soil samples, especially the ones with some vegetation had higher TOC (0 - 1.08±0.09%), while sediment samples had very low values (0 - 0.29±0.21%). This finding is in line with findings in Guzella et al. (2016), reporting that OC was lower in sediment samples (0.2 - 1.6%) from the Mt. Everest region compared to soil samples (0.4 - 7.6%), suggesting that soils accumulate more OC than sediments.

Overall, TOC concentration was higher at Tarfalajaure (0.46±0.49%) compared to that at Storglaciären (0.04±0.07%) and Isfallsglaciären (0.03±0.03%). Since geology of the catchment doesn't change much from transect to transect, this was expected as older soils were sampled from the Tarfalajaure transect, demonstrating that soils exposed for longer time have higher OC values. However, there was no overall trend of OC build-up along the downstream transects. This is in contrast to, for example, a study by Wietrzyk et al. (2018), where an increase in TOC from low (0.69%) in newly exposed soils to higher (3.81%) in older soils in glacier forelands in Svalbard were observed. This suggests that build-up of OC along the transects is slow in bare catchments.

TN values were also low across all three transects (0-0.07%), compared to findings in glacier forelands from Svalbard (0.04-0.15%, Wietrzyk et al., 2018), Switzerland (0.02-0.6%, Prietzel et al., 2013) and Tibetan plateau (0.37-2.12%, Prietzel et al., 2013). TOC and TN were correlated only at the Tarfalajaure transect ( $R^2=0.55$ , Pearson correlation=0.74), suggesting that nitrogen is organic in nature (Stein and Rack, 1995 in Huon et al., 2002) in samples from this transect but not the others. Organic nitrogen can be transformed to ammonia (NH<sub>3</sub>) and ammonium (NH<sub>4</sub>), becoming available for vegetation utilization (Brust, 2019), and hence development of organic soils. Once again, this suggests that soils along the Tarfalajaure transect are more organic, while glacier forelands of Storglaciären and Isfallsglaciären do not have enough organic matter that could be utilized by plants.

Moreover, as OC concentrations were low, C/N ratios at Storglaciären (0.03±0.04 - 4.57±3.81) and Isfallsglaciären (0.50±0.25 to 4.69±6.46) were lower than C/N at Tarfalajaure (1.57±1.53 to 20.57±7.48). Since C/N ratio can be used to elucidate the source of organic matter (Yu et al., 2010), comparing these values to other studies showed that

samples from Storglaciären and Isfallsglaciären were lower than in soil environments elsewhere ( $8.9\pm 1.1 - 17.9\pm 3.6$ , Yu et al., 2010), while C/N at Tarfalajaure was descriptive of soil/plant material ( $8.9\pm 1.1 - 17.9\pm 3.6$  in soils and  $22.7\pm 11.6 - 24.6\pm 9.4$  in plants, Yu et al., 2010). This suggests that similar to TOC concentrations and relationship between TOC and TN, samples collected along the Tarfalajaure were more representative of more organic soils compared to samples collected at Storglaciären and Isfallsglaciären, that have been exposed for shorter period.

### 4.3.2 No variation in metal concentration

Principal component analysis of chemical properties (TOC, TC, N, and metals) showed that most metals, as well as TOC, TC, and N played an important role in the distribution of samples by transect (Figure 4.1). This indicated that concentration of OC and other elements should be taken into account when analysing and grouping the samples. Despite some overlap, PCA showed that there was a difference between the transects, especially between Tarfalajaure and Storglaciären. Samples from Isfallsglaciären were clustered together, indicative of little variation in chemistry. This was expected as Isfallsglaciären glacier foreland had less variation in landscape, i.e. less vegetation, but also lower metal concentrations as the metal analysis showed. Samples from Storglaciären were distributed along PC1, which was correlated with most metals, indicating variation in chemistry. This was expected as metal analysis showed that samples from Storglaciären had higher concentration of most metals compared to other two transects, while having little vegetation (low TOC and TN concentrations). Meanwhile, samples from Tarfalajaure were distributed along PC2, indicative of sample variation by OC and TN, as well as some metals. This was expected as Tarfalajaure was exposed for a longer period compared to Storglaciären and Isfallsglaciären (Holmlund et al., 2005; Svenonius, 1910; Schytt, 1959 in Kirchner et al., 2019). This finding is in line with other studies, confirming that time elapsed after deglaciation plays an important role in accumulation of soil derived OC (Wietrzyk et al., 2018).

For instance, soil samples from Tarfalajaure and Storglaciären had higher concentration of metals compared to sediments, while soil samples from Isfallsglaciären had lower metal concentrations compared to sediments. However, a PCA plot, as well as PERMANOVA

analysis showed no particular difference between soil and sediment samples. On the contrary, Colombo et al. (2020) indicated that OC and element concentrations were different in proglacial soil and sediment samples from a proglacial environment in the Italian Alps. The lack of difference between the sampling environments in this study is most likely due to low TOC values, suggesting that the catchment is in its early stages of development.

Analysis of metal concentrations in soil and sediment samples from the Tarfala valley indicated that Fe ( $5227 \pm 2230$  mg/kg), Al ( $2998 \pm 791$  mg/kg), Ca ( $2658 \pm 905$  mg/kg), and Mg ( $1247 \pm 342$  mg/kg) were the most abundant metals. Similarly, these elements were abundant in glacier foreland soils at the Tibetan Plateau (Bing et al., 2016). Fe, Al, and Ca were also abundant in a glacier foreland in Svalbard, along with K and Zn (Venkatachalam et al., 2021), which were less abundant in samples collected in Sweden.

Moreover, metal concentrations recorded in Sweden were much lower compared to that at the Tibetan plateau and in Svalbard. For instance, at the Tibetan plateau concentration of Fe ranged from  $8800 \pm 2400$  to  $28400 \pm 6300$  mg/kg, Al – from  $15400 \pm 4800$  to  $50400 \pm 7600$  mg/kg, Ca – from  $19100 \pm 3600$  to  $33000 \pm 2200$  mg/kg, and Mg – from  $4000 \pm 600$  to  $14600 \pm 3100$  mg/kg (Bing et al., 2016). In Svalbard, concentrations of Fe (from 25547 to 53774 mg/kg) and Al (from 12561 to 39008 mg/kg) were much higher than that detected in Sweden, though concentration of Ca (from 537 to 2352 mg/kg) was comparable to Sweden (Venkatachalam et al., 2021). This suggests that while similar elements make up the majority of total metals, their concentration varies by country/location depending on chemical properties, i.e., geology, of the catchment.

No particular trend was observed in distribution of metal concentrations along the investigated transects in Sweden. This is similar to findings in Venkatachalam et al. (2021), where concentration of metals in soil samples collected along 1.8 km long glacier foreland chronosequence that has been deglaciated for up to 1900 years had varying concentrations but did not follow a particular pattern along the downstream transects of glacier foreland in Svalbard. Therefore, it seems that unlike OC, metals do not have a tendency to either accumulate or decrease in concentration, suggesting that their concentration is not affected by the time of exposure but rather by the geology of the catchment.

### 4.3.3 Organic biomarkers

It was expected that TOC concentration would play an important role in organic biomarker distribution along the downstream transects, as content and composition of organic biomarkers in soils is dependent on carbon content (Höfle et al., 2015; see Figure 4.10a and b). Moreover, both, TOC and BHPs ( $R^2=0.96$ , Pearson correlation=0.98), and TOC and brGDGTs ( $R^2=0.80$ , Pearson correlation=0.89) were correlated, suggesting that samples with low TOC will have low organic biomarker content and vice-versa.

In previous studies, BHP and GDGT distribution patterns were used to trace terrestrial OC input to aquatic systems (e.g., Bischoff et al., 2016; De Jonge et al., 2015; Doğrul Selver et al., 2012; Doğrul Selver et al., 2015). This is a first study aiming to use these organic biomarkers to understand accumulation of soil derived OC in deglaciated catchments.

#### 4.3.3.1 Soil-specific biomarkers compared to other Arctic catchments

##### 4.3.3.1.1 Soil marker BHPs

Soil marker BHPs are abundant in soils but not aquatic sediments (Cooke et al., 2008; Doğrul Selver et al., 2012; Höfle et al., 2015), and therefore, in this study they were used as indicators of soil development in deglaciated areas. BHP analysis showed the presence of soil-specific biomarkers in six out of eight samples from the Tarfala valley, suggesting that two samples with no detectable amounts of BHP were either very low in TOC and/or representative of sediments rather than soils. BHPs were detected in three out of four samples from the Storglaciären, and only two samples had detectable amounts of soil marker BHPs. Samples with no detectable amount of BHPs were a river sediment sample SG-DS-1008 with 0.01% of carbon content, and a sample from a moraine SG-M2-0808 with no detectable carbon content had no detectable amount of soil marker BHPs. On the contrary, BHPs in general and soil marker BHPs specifically were detected in all samples from Tarfalajaure that had higher TOC content. This suggests that samples with no to low TOC concentrations lack BHPs and specifically soil marker BHPs.

Soil marker BHPs made up 8-21% of total BHPs at the Tarfalajaure, while at Storglaciären relative abundance of soil marker BHPs was lower (0-12%), suggesting that Tarfalajaure

accumulated more soil derived OC compared to Storglaciären. This was expected as Tarfalajaure has been exposed for a longer period of time allowing accumulation of soil marker BHPs. Moreover, soil marker BHPs in Sweden were less abundant compared to estuary sediment samples in the Arctic (from 15% at the Mackenzie river to 37% at the Indigirka river systems, Cooke et al., 2009), soils in Northern England (20-48%, Cooke et al., 2008) and soils from permafrost regions in the Arctic (20-73% at the Bayelva River catchment, Rethemeyer et al., 2010; up to 82% in Siberia, Doğrul Selver et al., 2015), suggesting that the Tarfala valley accumulated less SOC than most soils and even some sediments.

Similar to the relative abundance of soil marker BHPs, the  $R'_{\text{soil}}$  index was higher in samples from the Tarfalajaure transect (0.12-0.34) compared to samples from the Storglaciären glacier foreland (0-0.21), strengthening the previous observation of Tarfalajaure accumulating more soil derived OC than Storglaciären. The low content of soil marker BHPs was also reflected in the  $R'_{\text{soil}}$  index (0-0.34) used to trace soil derived OC in the Arctic region (Doğrul Selver et al., 2012; Doğrul Selver et al., 2015). This value was similar to aquatic sediments in Northern Sweden ( $R'_{\text{soil}}$  index=0.08-0.30, Doğrul Selver et al., 2012) but lower than the  $R'_{\text{soil}}$  in other Arctic permafrost catchments (e.g., 0.57 in Kolyma River in Northeast Siberia, Doğrul Selver et al., 2015; 0.16-0.62 in river sediment samples from Yenisei River in Siberia, De Jonge et al., 2016). This suggests that accumulation of soil OC in glacier foreland in this study is similar to that found in aquatic sediments in Northern Sweden (Doğrul Selver et al., 2012). Moreover, as expected, lower  $R'_{\text{soil}}$  in proglacial environments in the Tarfala valley compared to permafrost catchments in the Arctic indicate higher organic matter content in permafrost, known for its high OC storage (total of 1672 Pg of OC in the northern circumpolar permafrost region (Tarnocai et al., 2009), compared to 6 Pg of OC in glacier ice (Hood et al., 2015)).

#### **4.3.3.1.2 BrGDGTs**

BrGDGTs produced by anaerobic soil bacteria are mostly found in terrestrial environments (Hopmans et al., 2004; Weijers et al., 2006; Doğrul Selver et al., 2012), also making them a good indicator of soil development. However, only half of the samples had detectable amounts of GDGTs. No GDGTs were detected in a sediment sample KG-MW-1308 from a

glacier front at the Tarfalajaure transect, while only sample SG-VP-0808 from Storglaciären, which had the highest TOC content among other samples from this transect, had detectable amount of GDGTs. This could be due to the lack of bacteria producing brGDGTs – Acidobacteria are presumably a potential source of brGDGTs (Weijers et al., 2009). Moreover, though there was a strong correlation between GDGTs and BHPs ( $R^2=0.88$ , Pearson correlation=0.94), no GDGTs were detected in samples from a meltwater stream and a moraine at Storglaciären (SG-R-0808 and SG-M2-0808) that had detectable BHP signals. Other than the assumption that few/no GDGTs were produced, this could be due to very low TOC values (< 0.01%), reinforcing the importance of carbon content for organic biomarkers, as organic biomarkers are one of the compounds that make up OC.

Where detected, the relative concentration of total brGDGTs at the Tafala valley went up to 525  $\mu\text{g/g}$  TOC, ranging from BDL to 525  $\mu\text{g/g}$  TOC in samples from Tarfalajaure, and from BDL to 478  $\mu\text{g/g}$  TOC in samples from Storglaciären. This suggests that even though generally Storglaciären had lower SOC content, sample SG-VP-0808 is comparable to soil samples from Tarfalajaure, despite being exposed for a shorter time. This was also observed from the TOC concentrations, sample SG-VP-0808 having the highest TOC content ( $0.13\pm 0.11\%$ ) compared to other samples from Storglaciären. Samples T-L-1308 and MS-TL-1408 from Tarfalajaure also had some crenarchaeol (5 and 21  $\mu\text{g/g}$  TOC, respectively). Relative concentrations of GDGTs in samples from Sweden in this study were much higher than that in Northern Sweden (total brGDGTs ranged between 0.26-0.91  $\mu\text{g/g}$  TOC and crenarchaeol between 0.09-0.74  $\mu\text{g/g}$  TOC, Doğrul Selver et al., 2012), Kolyma River (total brGDGTs – 3.2-4.5, crenarchaeol – 1.8-5.4  $\mu\text{g/g}$  TOC, Doğrul Selver et al., 2015) and Yenisei River (total brGDGTs – 0.1-35, crenarchaeol – 0.06-11  $\mu\text{g/g}$  POC, De Jonge et al., 2015). Such high relative concentration of total GDGTs in samples from Sweden seem quite unrealistic and is most likely caused by very low TOC concentrations in Sweden ( $0 - 1.08\pm 0.09\%$ ) compared to other sites (e.g., 8-13.6% in samples from Kolyma River, Vonk et al., 2010 in Doğrul Selver et al., 2015), since the organic biomarker concentrations were normalized by TOC. Moreover, no GDGTs were detected in sediment samples or any soil samples with very low TOC content, which is surprising because brGDGTs are widely detected in river and lake sediments (Blaga et al., 2009, Tierney and Russell, 2009, Zink et al., 2010, Loomis et al., 2011, Sun et al., 2011, Zhang et al., 2012, Shanahan et al., 2013,

Ajioka et al., 2014a, Zell et al., 2014a, Hanna et al., 2016, Freymond et al., 2017, Peterse and Eglinton, 2017 in Cao et al., 2018). Absence of GDGTs in these samples could be due to low productivity, causing low TOC content (0-0.02%) and low/no GDGTs.

Distribution of brGDGTs can be used as a proxy to study paleotemperatures (Weijers et al., 2007) and past soil pH (Peterse et al., 2012). Therefore, GDGT proxy values in this chapter were compared to the existing literature to ensure whether calculating proxies would provide information on past soil pH and temperature. Proxies calculated using GDGTs seem to be reasonable. GDGT proxy soil pH varied from 4.4 to 6.3, which is similar to the soil pH<sub>H2O</sub> reported in samples from the Tarfala valley (4.24±0.02 to 6.22±0.02, Agnelli et al., 2021). Annual mean air temperature (MAT) fluctuated between -0.3 and 1.8 °C. However, according to Ingvander et al. (2013), mean annual temperature at the Tarfala valley was -3.5°C (1965-2011). Similarly, De Jonge et al. (2016) reported that MAT in Yenisei watershed soils was also above the annual mean air temperature and most likely representative of mean air temperature in summer. This suggest that GDGT proxies, especially MAT should be considered with care, and account for some fluctuations from the actual readings.

#### **4.3.3.2 Source and nature of organic biomarkers**

The samples with detectable amounts of GDGTs were dominated by brGDGTs, which are produced in soils and indicative of terrestrial OC (Weijers et al., 2006). This is applicable to sampling locations in this study site, as GDGTs were detected only in some soil samples but not sediments from the Tarfala valley. Small quantities of crenarchaeol, which is mainly produced in aquatic systems (Hopmans et al., 2004; Zell et al., 2013), was also detected in some samples.

Crenarchaeol was found in soil samples T-L-1308 and MS-TL-1408 from Tarfalajaure, which were collected close to the Tarfala lake and river, suggesting fluvial in-situ production. This is most likely due to the location of samples next to the lake and river and the seasonal increase in water levels. Moreover, according to Peterse et al. (2014), brGDGTs can also be produced in rivers and lakes. The fact that samples from the Tarfalajaure transect analysed in this study contain brGDGTs and some crenarchaeol strongly suggests that distribution of GDGTs at the Tarfalajaure transect have mixed origin and is derived mostly from soils but also in-situ production in the Tarfala lake and the river.

Only one sample from the Storglaciären glacier (SG-VP-0808) which was a soil sample, had detectable amounts of brGDGTs and no crenarchaeol, suggesting that the brGDGTs in the sample is derived from soil producing bacteria (Weijers et al., 2009).

Despite different chemical composition and the time of exposure, samples from the Tarfalajaure and Storglaciären transects had similar main BHP compounds. For instance, adenosylhopane and adenosylhopane type 3 were the main soil marker BHPs detected in samples from both Tarfalajaure and Storglaciären transects. Soil marker BHPs, adenosylhopane and related structures are produced by purple non-sulfur bacteria, *Nitrosomonas europaea*, and *Bradyrhizobium japonicum* (as cited in table 2.2a, Cooke, 2011), while Acidobacteria is thought to produce brGDGTs (Weijers et al., 2009). Other individual BHPs are also produced by specific bacteria and therefore, can be used as biomarkers characteristic of certain environments (Killops and Killops, 2005). The most abundant BHP structures in samples from Tarfalajaure were BHT contributing to 36-56% of total BHPs, soil marker BHPs (8-21%), aminotriol (4-10%), Me-BHT (1-13%), BHT glucosamine (3-7%) and BHT cyclitol ether (2-8%). Similarly, samples from Storglaciären also had high relative abundance of BHT (BDL-100%), soil marker BHPs (BDL-12%), aminotriol (BDL-10%), Me-BHT (BDL-8%), BHT glucosamine (BDL-8%) and BHT cyclitol ether (BDL-13%). BHT, aminotriol and BHT cyclitol ether are produced by various organisms and can be found in different environments (Table 4.4), therefore, presence of these BHP compounds in samples from Sweden was not surprising. Interestingly, some of the bacteria producing Me-BHT and BHT glucosamine also produce BHT, aminotriol and BHT cyclitol ether. Therefore, it is expected that these bacteria (Cyanobacteria, *Methylobacterium organophilum*, Methylophilic bacteria, *Zymomonas mobilis*) are present in samples from Sweden and produce more than one BHP compound. There was also a small amount of aminotetrol and methylated aminopentol, likely produced by methanotrophs (Table 4.4).

BHT glucosamine and methylated aminopentol which were previously reported in sediments (Table 4.4, as cited in table 2.2b, Cooke, 2011) but not soils were also present in soil samples in this study. Moreover, they were one of the abundant BHP compounds. This provides new information about the environment these compounds can be found in.

Table 4.4. Major BHPs, their source organism and environment they are found in (Adapted from Cooke, 2011) .

BHP	Source organism	Environment
Adenosylhopane and related structures	Purple non-sulfur bacteria <sup>1</sup> , <i>Nitrosomonas europaea</i> <sup>1</sup> , <i>Bradyrhizobium japonicum</i> <sup>1</sup>	Sediment <sup>2</sup> , soils <sup>2</sup> , peats <sup>2</sup>
BrGDGTs	Acidobacteria <sup>32</sup>	Soils <sup>32</sup> , peats <sup>32</sup>
BHT	<i>Acetobacter</i> <sup>19,28</sup> , <i>Acetobacter xylinum</i> <sup>18</sup> , <i>Anabaena cylindrica</i> <sup>30</sup> , <i>Azotobacter vinelandii</i> <sup>31</sup> , <i>Bacillus acidocaldarius</i> <sup>18</sup> , <i>Beijerinckia indica</i> <sup>31</sup> , <i>Beijerinckia mobilis</i> <sup>31</sup> , <i>Calothrix</i> <sup>30</sup> , <i>Chlorogloeopsis fritschii</i> <sup>30</sup> , <i>Chroococcidiopsis</i> <sup>30</sup> , <i>Cyanobacteria</i> <sup>25,30</sup> , <i>Cyanothece</i> <sup>30</sup> , <i>Desulfovibrio</i> <sup>5</sup> , <i>Frankia</i> <sup>3,21</sup> , <i>Frateuria aurantia</i> <sup>11</sup> , <i>Gloeocapsa sp.</i> <sup>30</sup> , <i>Gluconacetobacter</i> <sup>28</sup> , methylotrophs <sup>25</sup> , <i>Methylobacterium organophilum</i> <sup>4,8,12,20,29</sup> , <i>Methylocystis parvus</i> <sup>24</sup> , <i>Methylosinus trichosporium</i> <sup>15</sup> , <i>Microcystis aeruginosa</i> <sup>30</sup> , <i>Nostoc muscorum</i> <sup>4,30</sup> , <i>Oscillatoria amphigranulata</i> <sup>30</sup> , <i>Phormidium sp.</i> <sup>30</sup> , <i>Prochlorothrix hollandica</i> <sup>23</sup> , Purple non-sulfur bacteria <sup>25</sup> , Purple non-sulfur bacterium <i>Rhodomicrobium vannielii</i> <sup>14</sup> , <i>Rhodopseudomonas acidophila</i> <sup>8</sup> , some gram-positive and gram-negative bacteria <sup>25</sup> , <i>Zymomonas mobilis</i> <sup>8,9,13</sup>	Sediment <sup>2</sup> , soils <sup>2</sup> , water column <sup>2</sup> , peat <sup>2</sup> , hot springs <sup>2</sup>
BHT cyclitol ether	<i>Acetobacter xylinum</i> <sup>10</sup> , <i>Anacystis montana</i> <sup>30</sup> , <i>Azotobacter vinelandii</i> <sup>31</sup> , <i>Burkholderia cepacia</i> <sup>27,28</sup> , <i>Chlorogloeopsis</i> <sup>26,30</sup> , <i>Chroococcidiopsis</i> <sup>30</sup> , <i>Desulfovibrio</i> <sup>5</sup> , <i>Frateuria aurantia</i> <sup>11</sup> , <i>Gloeocapsa sp.</i> <sup>30</sup> , <i>Methylobacterium</i> <sup>12</sup> , <i>Methylobacterium organophilum</i> <sup>17,20</sup> , <i>Trichodesmium erythraeum</i> <sup>30</sup> , <i>Zymomonas mobilis</i> <sup>17,24</sup>	Sediments <sup>2</sup> , soils <sup>2</sup> , water column <sup>2</sup> , peat <sup>2</sup>
Aminotriol	<i>Anacystis montana</i> <sup>30</sup> , <i>Beijerinckiaceae</i> <sup>31</sup> , <i>Bradyrhizobium japonicum</i> <sup>6</sup> , cyanobacteria: <i>Microcystis sp.</i> <sup>30</sup> , <i>Chroococcidiopsis sp.</i> <sup>30</sup> , <i>Desulfovibrio</i> <sup>5</sup> , <i>Methylomicrobium album</i> <sup>24</sup> , <i>Methylobacterium organophilum</i> <sup>8</sup> , <i>Methylocystis parvus</i> <sup>24</sup> , <i>Methylosinus trichosporium</i> <sup>24</sup> , <i>Microcystis aeruginosa</i> <sup>30</sup> , <i>Nitrosomonas europaea</i> <sup>22</sup>	Sediments <sup>2</sup> , soils <sup>2</sup> , water column <sup>2</sup> , hot springs <sup>2</sup>
Me-BHT	<i>Cyanobacteria</i> <sup>1</sup> , <i>Rhodopseudomonas palustris</i> <sup>1</sup> , <i>Methylobacterium organophilum</i> <sup>1</sup>	Sediments <sup>2</sup> , soils <sup>2</sup> , water column <sup>2</sup> , hot springs <sup>2</sup>
BHT glucosamine	Methylotrophic bacteria <sup>1</sup> , Thermoacidophilic bacteria <sup>1</sup> , <i>Zymomonas mobilis</i> <sup>1</sup>	Sediments <sup>2</sup>
Aminotetrol	Methanotrophs <sup>1</sup>	Sediments <sup>2</sup> , soils <sup>2</sup> , water column <sup>2</sup> , peat <sup>2</sup> , hot springs <sup>2</sup>
Me-aminopentol	Methanotrophs <sup>1</sup>	Sediments <sup>2</sup> , hot springs <sup>2</sup>

References: <sup>1</sup>as cited in table 2.2a, Cooke, 2011; <sup>2</sup>as cited in table 2.2b, Cooke, 2011; <sup>3</sup>Berry et al., 1991; <sup>4</sup>Bisseret et al., 1985; <sup>5</sup>Blumenberg et al., 2006; <sup>6</sup>Bravo et al., 2001; <sup>7</sup>Cvejic et al., 2000; <sup>8</sup>Flesch and Rohmer, 1988; <sup>9</sup>Flesch and Rohmer, 1989; <sup>10</sup>Herrmann et al., 1996; <sup>11</sup>Joyeux et al., 2004; <sup>12</sup>Knani et al., 1994; <sup>13</sup>Moreau 1997; <sup>14</sup>Neunlist et al., 1985; <sup>15</sup>Neunlist and Rohmer, 1985a; <sup>16</sup>Neunlist and Rohmer, 1985b; <sup>17</sup>Neunlist et al., 1988; <sup>18</sup>Ourisson and Rohmer 1992; <sup>19</sup>Peiseler and Rohmer, 1992; <sup>20</sup>Renoux and Rohmer, 1985; <sup>21</sup>Rosa- Putra et al., 2001; <sup>22</sup>Seemann et al., 1999; <sup>23</sup>Simonin et al., 1996; <sup>24</sup>Talbot et al., 2001; <sup>25</sup>Talbot et al., 2003a; <sup>26</sup>Talbot et al., 2003b; <sup>27</sup>Talbot and Farrimond, 2007; <sup>28</sup>Talbot et al., 2007a; <sup>29</sup>Talbot et al, 2007b; <sup>30</sup>Talbot et al, 2008; <sup>31</sup>Vilcheze et al., 1994; <sup>32</sup>Weijers et al., 2009.

#### 4.3.3.3 Evidence for downstream soil development

Previously,  $R_{\text{soil}}$  and  $R'_{\text{soil}}$  indexes have been used to trace OC transport along the land to aquatic environment transects (e.g., Bischoff et al., 2016; Cooke et al., 2008; Doğrul Selver et al., 2012; Doğrul Selver et al., 2015; Spencer-Jones et al., 2015; Zhu et al., 2011), no studies have used these proxies to understand soil OC accumulation in post-glaciated areas before. This is a first study that uses  $R'_{\text{soil}}$  to study nature and source of soil OC in glacier forelands. Rethemeyer et al. (2010) found an increase in soil marker BHPs with depth in the soil profiles in soils from Spitsbergen, suggesting preservation of these compounds in soils. In this study it is expected that soil marker BHPs accumulate over time after being exposed, with concentrations of soil marker BHPs and  $R'_{\text{soil}}$  index increasing along the glacier downstream transects. As predicted, there was a clear pattern of increase in  $R'_{\text{soil}}$  along the Tarfalajaure transect (Figure 4.6), which could also be seen from Figure 4.7, where soil marker BHPs increased with the age of samples.

Along the Storglaciären transect, it was only possible to calculate  $R'_{\text{soil}}$  index at two locations, due to the lack of detectable soil marker BHPs. It is therefore difficult to make any firm conclusions, though the available data does suggest accumulation of soil marker BHPs over time after being exposed.

Likewise, brGDGTs are abundantly produced in soils (Weijers et al., 2006) and their distribution as biomarkers of terrestrial OC has previously been studied along transects from land to sea/ocean (Bischoff et al., 2016; De Jonge et al., 2015; Doğrul Selver et al., 2012; Doğrul Selver et al., 2015). However, the use of distribution of GDGTs to study accumulation of soil derived OC in glacier forelands, as in this study, has not been previously investigated. In a similar manner to the accumulation of soil marker BHPs, it was expected that relative concentration of GDGTs would also increase along the downstream

transects. Contrary to the expectation, there was no increase, or any specific pattern of GDGT distribution along the transects. This suggests that while brGDGT can be used as a tool confirming the presence of terrestrial OC in glacier forelands, the lack of pattern of GDGT downstream distribution suggest that this biomarker may not be suitable for analysing soil development. The difference in distribution of soil marker BHPs and brGDGTs could be because these organic biomarkers are produced by different bacteria (Table 4.4). When comparing the distribution of soil marker BHPs and brGDGTs in the soil profile of soils from a river watershed in France, Kim et al. (2011) reached a similar conclusion that brGDGTs were produced by anaerobic bacteria, while BHPs were produced by bacteria inhabiting more aerated topsoil profile. This calls for investigation of the current microbial community composition to gain a better understanding of the relationship between organic biomarkers and organisms producing them.

Moreover, no GDGTs were detected in sediment samples or a soil sample with very low TOC content, which is surprising because brGDGTs are widely detected in river and lake sediments (Tierney and Russell, 2009; Loomis et al., 2011; Zell et al., 2014; Peterse and Eglinton, 2017). The absence of GDGTs in these samples could be due to the low TOC content (0-0.02%) in samples from the Tarfala valley compared to other studies. For instance, TOC concentration ranged from 0.5 to 9.4% in river and lake sediment samples from Indonesia (Tierney and Russell, 2009), 0.1-16.6% in river and lake sediments from Uganda (Loomis et al., 2011), 0-13.7% in river and marine sediments from the Amazon Shelf (Zell et al., 2014) and 0.1-6.0% in river sediments from Canada, Costa Rica, Switzerland, New Zealand, and USA (Peterse and Eglinton, 2017), demonstrating that sufficient TOC concentration is essential to detect organic biomarkers.

Previous studies combining the investigation of both biomarkers highlighted the importance of using multi-proxy approaches in order to compare one to another and understand the fate and transport of terrigenous OC (Doğrul Selver et al., 2012; Doğrul Selver et al., 2015). In this study, it seems that soil marker BHPs and brGDGTs had different concentrations and different distribution along downstream transects in glacier forelands in this study. While studying the concentration and distribution of both BHPs and GDGTs might be important as soil-tracing biomarkers, absence of GDGTs in some samples indicated that this biomarker might not be useful to understand processes involved in the

accumulation of soil derived OC along the transects. Based on the investigation of organic biomarkers in samples from Sweden, it can be summarized that factors controlling the organic biomarker distribution are OC and distance along the transects at the Tarfala valley, hence time of exposure after deglaciation for soil marker BHPs and OC content for brGDGTs. Soil marker BHPs accumulate along the downstream transects, indicative of soil development over time. Meanwhile, though brGDGTs confirm presence of terrestrial OC in glacier forelands in Sweden, this biomarker does not provide information about soil development.

#### **4.3.4 Microbial community diversity in samples from the Tarfala valley**

While organic biomarkers function as a signal of past changes, it is also important to investigate the current microbial communities inhabiting sediments and soils in the Tarfala valley and their potential influence on the system. Therefore, whether microbial communities and their diversity change along the downstream transects in the same way as organic biomarkers was studied further.

##### **4.3.4.1 Role of chemical composition of samples**

Microbial analyses were carried out on a subset of the sites. Very few sediment samples were suitable for microbial analysis as they had very little DNA compared to soils, hence the microbial samples were dominated by soils. Also, only three samples from Storglaciären were analysed for bacterial community composition as they had less DNA than samples from Tarfalajaure and some were not amplified with PCR. Once again this suggests that samples from Storglaciären had less organic components.

First, the differences in chemical compositions of samples were investigated. PCA of samples selected for further microbial community studies indicated that soil and sediment samples had some difference from each other, high concentration of metals, TOC, TC and N having a noticeable impact on the clustering of soil samples (Figure 4.11a). This was contrary to the analysis of all samples from the Tarfala valley (see above in section 4.3.2). It is difficult to make firm conclusions due to the lack of sediment samples for the reasons outlined above, but based on the few sediment samples analysed, they are distinct from

the soils, as expected, and as found in Colombo et al. (2020), indicating that concentrations of OC and other elements vary in sediment and soil samples from a rock glacier and a glacier front in the Italian Alps. These observations suggest that soil and sediment samples chosen for investigation of microbial communities represent different environments and hence, should be inhabited by different bacteria.

Moreover, the PCA plot (Figure 4.11b) shows a distinct difference between samples from Tarfalajaure and Storglaciären transects. This is likely due to the fact that surroundings of the Tarfalajaure were exposed after deglaciation for a longer period of time (Svenonius, 1910; Schytt, 1959 in Kirchner et al., 2019), compared to Storglaciären (Holmlund et al., 2005), in line with findings in Wietrzyk et al. (2018) suggesting that time of exposure is one of the main factors controlling soil development. However, there was no statistically significant difference between the transects (PERMANOVA,  $p$ -value > 0.05). Therefore, it would be interesting to see whether there is some difference in microbial community habitats at each transect.

#### **4.3.4.2 Bacterial diversity**

Elemental composition of soil and sediment samples plays a major role in the establishment of microbial community structure. This becomes clear comparing soil and sediment samples. The alpha diversity measurements (Chao1 species richness, Figure 4.14) suggested that bacterial community composition of sediment samples was less diverse than soil samples. Considering that sediment samples are representative of recently deglaciated areas, compared to soil samples, this difference in diversity is in line with other studies. For instance, Mapelli et al. (2018) found that bacterial communities vary along the chronosequence of a glacier foreland in Svalbard, barren soils (8, 22, and 43 years old) having lower bacterial diversity (alpha- and beta-diversity) than developing soils (66 and 106 years old) and developing soils lower diversity than mature soils (156 and >1900 years old). Similarly, Venkatachalam et al. (2021) also reported less diverse groups from the recently deglaciated sites from the glacier foreland in Svalbard, compared to older sites with rich alpha diversity. This suggests that it takes time for microbial communities to establish after glacier retreat.

It was expected that diversity would increase along the Tarfalajaure transect from newer to older soils similar to findings in Mapelli et al., 2018 and Venkatachalam et al., 2021. However, sample TL-RB-1408 had higher microbial diversity than sample MS-TL-1408, which is located downstream. Sample TL-RB-1408 was collected from a lake bank that had some vegetation, suggesting that plant material rather than the age of the sample plays an important role in bacterial diversity. Moreover, as it was expected that diversity increases with soil age, it was also expected that the Tarfalajaure samples would have a significantly higher diversity than the Storglaciären samples. However, whereas one replicate of soil sample SG-VP-0808 from Storglaciären had a similar diversity to samples from Tarfalajaure, a second replicate had significantly higher diversity. This is contrary to findings of studies indicating that the time of exposure plays a major role in microbial diversity of soils (Mapelli et al., 2018; Venkatachalam et al., 2021). Again, it is difficult to make firm conclusions based on a limited set of samples, particularly from Storglaciären, and these unexpected results from Storglaciären may be due to the presence of bacteria caused by plant material. Sample SG-VP-0808 was collected from a meltwater stream bank that had some plant material. Mapelli et al. (2018) also found that rhizosphere (area around plant roots) soils had higher diversity than bulk soils in any stage of soil development. Therefore, it would be interesting to see the difference in microbial community structure of replicates with differing alpha diversity.

#### **4.3.4.3 Bacterial Taxa**

Dominant phyla identified in samples from Sweden were candidate phylum AD3, Acidobacteria, Actinobacteria, Chloroflexi, Proteobacteria, and Planctomycetes, all of which appeared consistently in all samples (Figure 4.15a). Relative abundance of some of the phyla in Sweden is similar to that to other cold habitats. For instance, Acidobacteria and Actinobacteria were the dominant phyla in glacier foreland in Spitsbergen (Schütte et al., 2010). Seok et al. (2016) reported that Actinobacteria and Proteobacteria were the dominant phyla in glacier forelands in Svalbard. Venkatachalam et al. (2021) reported that Acidobacteria, Actinobacteria, Chloroflexi and Proteobacteria were dominant phyla in soil samples from a glacier foreland in Svalbard. Moreover, Planctomycetes was also found in other cold habitats (e.g., Costello and Schmidt, 2006; Ji et al., 2016; Mateos-Rivera et al., 2016). Meanwhile, candidate phylum AD3 was usually present in small quantities except in

samples from Mitchell Peninsula (Ji et al., 2016). This suggests that with the exception of the candidate phylum AD3 glacier forelands in Sweden are inhabited by microbial communities similar to other cold environments.

There was a difference in phylum distribution between transects: candidate phylum AD3 (21.3±12.3%), Chloroflexi (17.7±5.7%), Acidobacteria (15.2±4.2%), and Proteobacteria (11.7±9.1%) were the main phyla at Tarfalajaure, while Proteobacteria (27.8±21.1%), Acidobacteria (24.0±22.1%), Actinobacteria (10.9±5.1%), and Chloroflexi (9.2±6.2%) were the dominant phyla at Storglaciären, suggesting that despite the close proximity, bacterial communities evolved differently in systems in Tarfala valley. This was especially true for the candidate phylum AD3 which was abundant in samples from the older Tarfalajaure transect (T-L-1308, TL-RB-1408 and MS-TL-1408). Previously, this phylum was reported to be found in older soils in Norway (Mateos-Rivera et al., 2016). Its dominance in the longer exposed soils in Tarfalajaure, but not in Storglaciären suggests that this bacterial taxon is indicative of older soils.

As discussed earlier, sediment samples had less organic matter, therefore, it was expected that they would have less bacteria associated with them, but also different phyla. Soils had different phyla compared to few sediment samples analysed for bacterial community composition. For instance, soil samples had high relative abundance of candidate phylum AD3 (19.6±12.9%), Acidobacteria (19.6±9.5%), and Chloroflexi (17.6±5.1%) compared to sediments (1.6±0.3%, 4.1±1.8%, and 5.3±4.5%, respectively). On the contrary, Proteobacteria (44.2±11.2%) and Actinobacteria (13.9±3.9%) were the dominant phyla in sediments, while their relative abundance in soils was lower (Proteobacteria (10.2±3.8%) and Actinobacteria (4.1±2.1%)). Also, high relative abundance of Bacteroidetes was observed in sediment samples (12.3±5.2%) compared to soils (0.8±0.5 %). These were similar to other cold habitats, for example along with Actinobacteria, Acidobacteria, and Proteobacteria, Chu et al. (2010) identified Bacteroidetes as the dominant phylum in polar soil samples. This observation suggests that sediment samples may be colonised by bacteria that become less abundant in more developed soils with high bacterial diversity.

To address the distribution of bacteria in samples from the Tarfala valley, each phylum was investigated in more detail. Firstly, Acidobacteria had higher relative abundance in soil

samples, compared to sediments, having the highest relative abundance in the soil sample from Storglaciären (SG-VP-0808\_1). Previously, Acidobacteria has been reported to be one of the phyla with highest relative abundance in soils, that on average makes up 20% of the total microbial community in soils (Janssen, 2006). Similarly, in the present study, mean relative abundance of Acidobacteria was  $15.2 \pm 4.2\%$ . Kielak et al. (2016) suggesting that Acidobacteria easily adapt to oligotrophic conditions and low pH, explaining their abundance in nutrient-poor soils from the Tarfala valley. Moreover, according to Fierer et al. (2005), Acidobacteria have higher abundance in low carbon soils, which is also in line with TOC concentration of samples from the Tarfala valley. A study showed that addition of organic matter to soils decreased the relative abundance of this phyla (Delgado-Balbuena et al., 2016). Therefore, it would be interesting to compare the relative abundance of this phylum with other study sites of this project (Iceland and Greenland).

In contrast to Acidobacteria, Proteobacteria had higher relative abundance in sediment samples compared to soils, which is comparable to findings in Mateos-Rivera et al. (2016), where it was more abundant in recently deglaciated soils. Similarly, the most abundant family member of Proteobacteria, Comamonadaceae, decreased with the age of soil in recently deglaciated areas (Nemergut et al., 2007). Nemergut et al. (2007) suggested that Comamonadaceae lives in or under the glacier, explaining its relatively high abundance in younger soils. *Thiobacillus* genus from the Hydrogenophilaceae family was another abundant member of Proteobacteria in samples collected in Sweden. *Thiobacillus* is chemolithoautotrophic and metabolizes reduced sulphur compounds (Orlygsson and Kristjansson, 2014), explaining its relatively high abundance in a sediment sample from Storglaciären. Therefore, it can be concluded that the presence of these bacteria suggests that sediments are younger than soils and, despite Tarfalajaure being more developed, the soils from both Storglaciären and Tarfalajaure are also characteristic of a young system.

Similar to Proteobacteria, Actinobacteria also had higher relative abundance in sediment samples. Actinobacteria is a large taxonomic group and can be found both in aquatic and terrestrial ecosystems. Most Actinobacteria are aerobic and can be either heterotrophic or chemoautotrophic (Barka et al., 2016). In Sweden Nocardioideae, Intrasporangiaceae and Microbacteriaceae were the dominant family members of Actinobacteria. Interestingly, Nocardioideae had high relative abundance in a sediment sample from

Storglaciären, while Microbacteriaceae in samples from Tarfalajaure. Members of the family Nocardioidaceae have been reported as chemoorganotrophs that can be found in different environments, including sediments (Persina et al., 2018). Members of Microbacteriaceae are suggested to have originated from glacier ecosystems (Mapelli et al., 2018), which could explain its presence in a sediment sample along the Tarfalajaure transect, as this was collected close to a glacier. Intrasporangiaceae was abundant in both transects, similarly, genus *Phycoccus* from the Intrasporangiaceae family was also found in soils from the Tianshan Glacier No. 1 (Wu et al. unpub. in Stackebrandt et al., 2014). Based on these observations it is suggested that Actinobacteria prefer to inhabit sediments without much downstream variation in soil samples and prefer low carbon environments suggesting chemoautotrophic nature.

Another phylum abundant in samples from the Tarfala valley was Chloroflexi, which had been found in different environments, including cold environments (Costello and Schmidt, 2006). Ji et al. (2016) suggested that Chloroflexi could perform photosynthesis in soil. In samples from both Storglaciären and Tarfalajaure, phyla Chloroflexi had higher relative abundance in older soils compared to recently deglaciated sediment samples, in line with findings in Mateos-Rivera et al. (2016) reporting that relative abundance of this phylum increased with increasing soil age. Ktedonobacteraceae and Thermogemmatissporaceae were the most abundant family members of Chloroflexi identified in soil samples from Tarfalajaure, while classified at class-level Ellin6529 was abundant in both Storglaciären and Tarfalajaure samples. Literature indicates that family-level phylogenetic groups of Chloroflexi were poorly studied. Members of class Ktedonobacteria (Ktedonobacteraceae and Thermogemmatissporaceae) are reported to inhabit different environments, including extreme and cold environments (Yabe et al., 2017). Ellin6529 is N<sub>2</sub> fixing member, contributing to enriching deglaciated soils in labile N (Lopes et al., 2015). Findings in this study are in line with this, as in samples from Sweden Chloroflexi was more abundant in soils than sediments and soils contain plant material, and in samples from Tarfalajaure compared to Storglaciären, which had higher C/N ratio.

In Sweden, another phylum of the relatively abundant bacteria, Planctomycetes, had higher relative abundance in soils compared to sediments. They can be found in different environments, including freshwater and soil (Fuerst and Sagulenko, 2011). Planctomycetes

were found in other polar and alpine environments (e.g., Costello and Schmidt, 2006; Ji et al., 2016; Mateos-Rivera et al., 2016). Gemmataceae, Pirellulaceae, and unclassified at family-level N1423WL were the most abundant members of Planctomycetes. Gemmataceae is a strictly aerobic and chemoorganotrophic member of Planctomycetes (Kulichevskaya et al., 2020), which could explain its higher presence in soil samples from Sweden. Classified at order-level N1423WL was reported to be negatively correlated to ammonium ( $\text{NH}_4^+$ ), decreasing with the increase in  $\text{NH}_4^+$  (Mackelprang et al., 2018). Compared to other members of the Planctomycetes, N1423WL had a higher relative abundance in sediment samples, perhaps due to the utilization of ammonium by vegetation in soils (Brust, 2019).

Though Bacteroidetes was reported to be one of the most relatively dominant phyla in polar soils (Chu et al., 2010), it was mainly present in sediment samples from the Tarfala valley. In line with that, Bacteroidetes have been reported to grow in anoxic environments such as proglacial lake and meltwater stream sediments and glacier terminus, fermenting inorganic material of subglacial origin (Sheik et al., 2015). It is also worth mentioning that Bacteroidetes, was a dominant group in snow and ice samples from an Alaskan glacier (Choudhari et al., 2014), confirming that this phylum can be found in sediment samples as well. This suggests that Bacteroidetes metabolizes inorganic material in OC poor sediment samples from Sweden.

While the aforementioned phyla were prominent in other samples from cold environments, the bacterial communities identified at the Tarfala valley had unusually high relative abundance of the candidate phylum AD3. Generally, even when present, the candidate phylum AD3, is not the most dominant phylum. From previous publications, soils with such high relative abundance of candidate phylum AD3 have only been reported ones (Mitchell Peninsula, Ji et al., 2016). Moreover, candidate phylum AD3 was present only in mature soils (LIA) from the Styggedalsbreen glacier foreland in Norway (Mateos-Rivera et al., 2016), in line with the age of soil samples collected along the Tarfalajaure transect.

Though most of the bacteria reported from the Tarfala valley is in line with other studies (Chu et al., 2010; Costello and Schmidt, 2006; Ji et al., 2016; Mateos-Rivera et al., 2016; Schütte et al., 2010; Seok et al., 2016; Venkatachalam et al., 2021), high relative abundance

of the candidate phylum AD3 highlights the uniqueness of this system and calls for further investigation.

#### **4.3.4.4 Bacterial communities along the transects**

Four samples from Tarfalajaure and two samples from Storglaciären were investigated for microbial community analysis, which is too few samples to get a full set of data along a transect. As there was a difference in microbial communities in sediments and soils, and accumulation of soil marker BHPs downstream, it may be expected that microbial communities would also change along the Tarfalajaure transect. However, no particular pattern of microbial community changes along the transect was observed. It is proposed that the lack of variation is due to the fact that bacterial community in older soils from Tarfalajaure stabilised over time. For instance, though Schütte et al. (2009) found that there was a microbial succession along the chronosequence in glacier foreland in Spitsbergen, in their findings from 2010, they concluded that community composition stabilised in older soils (older than 150 years) (Schütte et al., 2009). Therefore, it is believed that microbial communities adapt to the new environment over time and become homogeneous in older soils such as the surroundings of the Tarfalajaure lake that perhaps have been ice free for as long as 9000-8500 years BP (Karlen, 1979). As for the younger soils of Storglaciären that have been developing anew since the glacier maximum in 1910 (Holmlund et al., 2005), the soil sample is young and the bacterial community is still being established. Overall, it seems that in samples from the Tarfala valley the microhabitat from where samples were taken (i.e., soil, sediments, riparian areas, vegetation) is more important than simply distance from the glacier front.

#### **4.3.4.5 Relationship between microbial community and organic biomarkers**

It was discussed earlier that specific biomarkers are associated with particular environments and source organisms, and that the biomarkers identified in this study have been associated with particular bacterial taxa. It may be expected therefore, that the BHPs and the bacterial taxa identified at each site would show a direct relationship. However, only quarter of bacterial taxa that have been identified as biomarker sources listed in Table 4.4 were identified in samples from the Tarfala valley, and even then, had very low relative abundances.

BHPs were detected in all samples from the Tarfala valley, except sediment sample SG-DS-1008. BHT, soil marker BHPs (adenosylhopane and related structures), aminotriol, Me-BHT, BHT glucosamine and BHT cyclitol ether were the main BHP compounds present in samples from Sweden. Adenosylhopane and related structures were of particular interest as they are generally abundant in soils (Cooke et al., 2008; Doğrul Selver et al., 2012; Höfle et al., 2015) and were used as indicators of accumulation of soil derived OC along transects in this study. Adenosylhopane and adenosylhopane type 3 were detected in all samples from Sweden except samples SG-DS-1008 and SG-M2-0808. Adenosylhopane producing Rhodospirillaceae (also known as purple non-sulfur bacteria) (as cited in table 2.2a, Cooke, 2011) was present in small quantities (< 1%) in all samples, except the sediment sample SG-DS-1008. Relative abundance of this bacterium increased from 0.25 to 0.44±0.12% along the Tarfalajaure transect, which is in line with observations of increase of soil marker BHPs. Similarly, family Nitrosomonadaceae of species *Nitrosomonas europaea* which produces soil-specific BHPs (as cited in table 2.2a) was present in samples T-L-1308\_2 and MS-TL-1408\_1 even in smaller quantities (< 0.1%). Based on this observation, it seems that soil marker BHPs in samples from Sweden were mainly produced by Rhodospirillaceae or purple non-sulfur bacteria.

BHT was the dominant BHP compound in samples from Sweden and was detected in all samples except SG-DS-1008. BHTs had higher abundance in soil samples compared to sediments but did not vary along the transects. BHT producing family Acetobacteraceae (species *Acetobacter* (Peiseler and Rohmer, 1992; Talbot et al., 2007a), *Gluconacetobacter* (Talbot et al., 2007a)) was present in small quantities in all soil samples (<1%) and had high relative abundance in the sediment sample KG-MW-1308, which had BHTs, but their concentration wasn't higher than that detected in soils. Family Frankiaceae (specie *Frankia* (Berry et al., 1991; Rosa- Putra et al., 2001)) was present in all samples except sediment samples KG-MW-1308 and SG-DS-1008 (relative abundance was less than 0.1% in samples from Tarfalajaure, less than 1% in samples from Storglaciären) and Hyphomicrobiaceae (specie *Rhodomicrobium vannielii* (Neunlist et al., 1985)) was detected in all samples and their relative abundance increased along the Tarfalajaure transect (from 0.19 to 1.43%). Another family of BHT producing species Nostocaceae (species *Anabaena cylindrica* (Talbot et al, 2008), *Nostoc muscorum* (Bisseret et al., 1985; Talbot et al, 2008)) was detected only

in sample TL-RB-1408\_1 (relative abundance < 0.1%), while Phormidiaceae (*Phormidium* sp. (Talbot et al, 2008)) was found only in sample KG-MW-1308 (relative abundance < 0.1%). BHT is produced by various organisms detected in samples from Sweden, therefore, while all the aforementioned bacteria could be responsible for BHTs detected in the samples, it is difficult to pinpoint which bacteria had the largest influence. Moreover, some bacteria producing BHTs also produce other BHP compounds identified in samples from Sweden (see Table 4.4 for details).

Other BHP compounds with high relative abundances identified in samples from Sweden were aminotriol, Me-BHT, BHT glucosamine and BHT cyclitol ether. The source organisms of these biomarkers produce more than one BHP compound (see Table 4.4 for details). Aminotriol was present in all samples from Tarfalajaure and followed an upward trend along the downstream transect. At Storglaciären, aminotriol was detected in samples SG-R-0808 and SG-VP-0808. BHT and aminotriol producing Methylocystaceae (species *Methylocystis parvus* (Talbot et al., 2001), *Methylosinus trichosporium* (Neunlist and Rohmer, 1985a; Talbot et al., 2001)), was detected in small quantities in all samples (relative abundance < 1%) except sample SG-DS-1008 and had the highest relative abundance in sample KG-MW-1308 (0.3%). Nitrosomonadaceae (*Nitrosomonas europaea*), which was discussed earlier in relation to soil marker BHPs and was found in very small quantities only in couple samples (T-L-1308\_2 and MS-TL-1408\_1), is also a source organism of aminotriol (Seemann et al., 1999). This suggests that while Methylocystaceae and Nitrosomonadaceae are the suggested sources of aminotriol, other bacteria could also have been producing this compound as its relative abundance increases along the Tarfalajaure transect.

Me-BHT was detected in all samples from Tarfalajaure and only sample SG-VP-0808 from Storglaciären. Concentration of Me-BHT also increased along the Tarfalajaure transect. BHT and Me-BHT producing cyanobacteria (as cited in table 2.2a, Cooke, 2011; Talbot et al., 2003a; Talbot et al, 2008) was detected in all samples except sample SG-DS-1008 and had the highest relative abundance in sample KG-MW-1308 (3.03%). This again suggests either that other bacteria could have been producing Me-BHT or that the bacterium mostly produces BHT.

BHT cyclitol ether was present in all samples from Tarfalajaure and samples SG-R-0808 and SG-VP-0808 from Storglaciären. BHT and BHT cyclitol ether producing family Pseudomonadaceae (specie *Azotobacter vinelandii* (Vilcheze et al., 1994)) was found in all samples except sample SG-DS-1008 (<0.1%). BHT cyclitol ether producing family Burkholderiaceae of a specie *Burkholderia cepacia* (Talbot and Farrimond, 2007; Talbot et al., 2007a) was also detected in small quantities in samples TL-RB-1408\_1, MS-TL-1408\_3 and SG-VP-0808 (<0.01%). The concentration of BHT cyclitol ether or the relative abundance of bacteria producing it did not increase along the downstream transects. These observations suggest that Pseudomonadaceae could be responsible for production of BHT cyclitol ether, especially taking into account that both the bacteria and the compound were not detected in sample SG-DS-1008.

BHT glucosamine was detected in all samples from Tarfalajaure and only sample SG-VP-0808 from Storglaciären. Concentration of BHT glucosamine was higher in SG-VP-0808 compared to samples from Tarfalajaure transect. Class-level methanotroph (as cited in table 2.2a, Cooke, 2011) Methylacidiphilae belonging to phylum Verrucomicrobia was observed in small quantities (<1%) in all samples except sediment samples KG-MW-1308 and SG-DS-1008, most likely contributing to production of BHT glucosamine, aminotetrol and methylated aminopentol, which also had low abundance. Aminotetrol had values below detection limit in these samples, as well as samples SG-R-0808 and SG-M2-0808, which were not analysed for microbial analysis. On the contrary, small concentrations of methylated aminopentol were detected only in samples KG-MW-1308 and SG-R-0808. Therefore, Methylacidiphilae could be responsible for producing BHT glucosamine and aminotetrol but not methylated aminopentol in samples from Sweden.

As for GDGTs, Acidobacteria, which are potentially responsible for producing brGDGTs (Weijers et al., 2009), was one of the dominant phyla and had higher relative abundance in soils rather than sediments. In line with this, brGDGTs were only present in soil samples from the Tarfala valley, but not sediments. This suggests that members of Acidobacteria detected in sediments are either do not produce brGDGTs or concentration of GDGTs is BDL. This suggests that while Acidobacteria could be a potential source of brGDGTs in soil samples from Sweden, not all species of this phyla produce brGDGTs. This calls for further

research to identify which members of Acidobacteria are source organisms of this biomarker.

Here, existing microbial populations were looked at alongside existing organic biomarkers. Even though an attempt was made to establish a relationship between specific organic biomarkers and bacteria, the fact that a link was established only between some BHPs with the existing microbial community inhabiting sediments and soils at the Tarfala valley shows that processes taking place in this study area are much more complex and calls for more studies establishing connections between organic biomarkers and specific organisms producing them. Although results indicated that the bacteria described above had low relative abundances, they may still have produced organic biomarkers detected in the samples, especially taking into account that organic biomarkers accumulate over a long period of time. On the other hand, as biomarkers accumulate over time, the microbial community present now might not be representative of the biomarkers produced by different bacteria in the past.

## **4.4 Conclusion**

The Tarfala valley is a bare landscape with no to low vegetation cover and, therefore, low TOC concentrations. Soil samples had higher TOC concentration than sediments, and soils exposed for longer time were more organic rich, reinforcing the hypothesis that time elapsed after deglaciation plays an important role in accumulation of soil derived OC. Moreover, despite the low TOC content, there was a clear trend of increase in soil marker BHPs downstream, indicative of soil development over time. Meanwhile, GDGTs were detectable only in soil samples with relatively high TOC, reinforcing the importance of OC concentration for the distribution of organic biomarkers. Microbial community diversity in the study site in Sweden was also largely affected by TOC concentration, older soils having higher bacterial diversity compared to sediment samples, dominant phyla shifting from Actinobacteria, Bacteroidetes, and Proteobacteria in sediment samples to candidate phylum AD3, Acidobacteria, Chloroflexi, and Planctomycetes in soils. Relatively high abundance of the candidate phylum AD3 in polar regions is rare in literature and highlights the uniqueness of the system in the Tarfala valley. It was initially expected that microbial community diversity would increase downstream the Tarfalajaure transect similar to the

build-up of total and soil marker BHPs. However, no such trend was observed in bacterial diversity. Contrary to the organic biomarkers, microbial community stabilised downstream, becoming homogeneous in soil samples, which suggests that bacteria in deglaciated areas quickly adapts to the new environment. This observation could be used to predict stabilisation in organic biomarker distribution in older and more developed catchments. This study looked at existing microbial populations alongside existing organic biomarkers, BHPs and GDGTs, and though firm relationship between organic biomarkers and existing bacterial community was not established, a link between some bacteria and biomarkers was established.

In conclusion, while older soils with high TOC at the Tarfalajaure transect had higher content of organic biomarkers and may give better representation of processes involved in accumulation of soil derived OC, studying younger transects, such as Storglaciären, is important to understand the initial processes and trends of microbial community establishment and soil development in recently deglaciated areas. The next chapter will investigate accumulation of soil derived OC in the older catchment at the Vatnajökull ice cap in Iceland.

## **5 Accumulation of soil derived OC at the Vatnajökull ice cap, Iceland**

### **5.1 Introduction**

The Vatnajökull ice cap in Iceland is an older catchment compared to that in Sweden but younger compared to the study site in Greenland. Six glaciers (Kviarjökull, Fjallsjökull, Skaftafellsjökull, Skeiðarárjökull, Svínafellsjökull, and Virkisjökull) at the southern margin of the ice cap (Figure 3.10) were investigated.

Samples at the Vatnajökull ice cap in Iceland were generally collected to represent a downstream transect of glacier forelands running across moraines from recently deglaciated areas, the LIA moraines, and older moraines of different ages. For instance, soil samples along the Svínafellsjökull transect were collected from a recent moraine (formed after the LIA), followed by a LIA moraine (Lee, 2016) and the 2500-years-old Stóralda moraine (Guðmundsson et al., 2002). A downstream transect along Virkisjökull ran across a recent moraine due to the glacial retreat since 1990s (Dochartaigh et al., 2007), followed by a LIA moraine (Everest et al., 2017) and a 5000-6000-year-old moraine (Guðmundsson, 1998). Similarly, a transect along the Kvíárjökull glacier foreland was represented by samples collected from a recent lake moraine, followed by the 1964-1980s hummocky moraine that had replaced ice-cored moraine formed during the LIA (Bennet et al., 2010), and the oldest moraine that formed in 3200 BP (Guðmundsson, 1997; Bennet et al., 2010). The Fjallsjökull downstream transect samples were collected from a lake moraine formed in 1965 (Rose et al., 1997), a LIA moraine (Thorarinsson, 1943), the oldest samples being collected from the terminal moraine of the glacial advance in 4500s BP (Rose et al., 1997). At Skaftafellsjökull, soil samples were collected from a recent lake moraine (from 1998 onwards) and an older moraine dating to 1939 (Marren, 2002). At Skeiðarárjökull, samples

were collected from a lake moraine and what is thought a moraine dating to 1900s (Bogacki, 1968), though frequent high-magnitude jökulhlaups deposit sediments, which most likely make up the top part of the stratigraphy at the glacier foreland.

Samples from all six glaciers were investigated for environmental chemistry. Further soil samples from Kviarjökull, Fjallsjökull, Svínafellsjökull, and Virkisjökull were investigated for organic biomarkers, while soil samples from all six glaciers were also analysed for microbial community structure.

The main aim of this chapter was to understand soil development at the Vatnajökull ice cap by looking at catchment chemistry, organic biomarkers and microbial community along glacier foreland transects. To achieve this aim the following objectives were carried out:

1. To determine TOC, C/N and major metals along transects of six glaciers: Kviarjökull, Fjallsjökull, Skaftafellsjökull, Skeiðarárjökull, Svínafellsjökull, and Virkisjökull;
2. To determine concentration and distribution of BHPs and GDGTs and their proxies ( $R'_{\text{soil}}$ , MAT and soil pH) in soil samples selected based on the results of objective 1 along Kviarjökull, Fjallsjökull, Svínafellsjökull, and Virkisjökull transects;
3. To determine microbial community structure in soil samples selected based on the results of objective 1 from Kviarjökull, Fjallsjökull, Skaftafellsjökull, Skeiðarárjökull, Svínafellsjökull, and Virkisjökull transects;
4. To establish a relationship between organic biomarker distribution and microbial community composition in soil samples from Kviarjökull, Fjallsjökull, Svínafellsjökull, and Virkisjökull transects.

Based on these objectives the following outcomes were hypothesised:

1. TOC builds up along the downstream transects of Kviarjökull, Fjallsjökull, Skaftafellsjökull, Skeiðarárjökull, Svínafellsjökull, and Virkisjökull glacier forelands.
2. Within each transect, OC concentrations are higher in soils than sediments.
3. There is a trend of total BHPs, GDGTs and soil marker BHPs accumulation along Kviarjökull, Fjallsjökull, Svínafellsjökull, and Virkisjökull downstream transects.
4. The  $R'_{\text{soil}}$  has an upward trend along the Kviarjökull, Fjallsjökull, Svínafellsjökull, and Virkisjökull downstream transects, indicating accumulation of soil marker BHPs.

5. Bacterial diversity increases with the age of samples away from the current glacier front.
6. Bacterial community stabilizes and is homogeneous in developed soil samples from Kviarjökull, Fjallsjökull, Skaftafellsjökull, Skeiðarárjökull, Svínafellsjökull, and Virkisjökull glacier forelands, similar to Sweden.

## 5.2 Results

### 5.2.1 Environmental chemistry

A total of 45 ice, sediment, and soil samples from the Vatnajökull ice cap in Iceland were analysed for TC, TOC, TN, C/N (see Appendix 13 for TC, TN and TOC raw data, mean concentration, and standard deviation) and metal concentrations (see Appendix 14 for mean values and SD). Total of 45 soil, sediment and ice sediment samples were collected from glaciers at the Vatnajökull ice cap (Table 5.1). Samples from Svínafellsjökull and Virkisjökull were collected in triplicates during two field seasons in 2018 and 2019.

Table 5.1. Summary of samples from glaciers at the Vatnajökull ice cap.

Glacier	Sample type			Total
	Soil	Sediment	Ice sediment	
Svínafellsjökull	7	4	3	14
Virkisjökull	6	6	3	15
Kviarjökull	4	1	-	5
Fjallsjökull	3	2	-	5
Skaftafellsjökull	2	2	-	4
Skeiðarárjökull	2	-	-	2

#### 5.2.1.1 Variation of chemical properties in different sample types and transects

Principal component analysis on chemical composition (TOC, TN, metals) was conducted on mean values of all ice sediment, sediment and soil samples collected at the Vatnajökull ice cap (Figure 5.1). The first and second principal components explained 39.4% and 23.2% of total variance, respectively. PC1 had strong positive correlation with Mn, Mg, Na, Ca, Al, Fe, Co, Cd, and Cu, while PC2 had strong positive correlation with Cr and Ni, strong negative correlation with P and Zn, and some negative correlation with TC, TOC and TN. PCA plots

show that there was more separation by sample type (Figure 5.1a) than between different glaciers (Figure 5.1b).



Table 5.2. Codes of sample names from the Vatnajökull ice cap, Iceland shown on the PCA plots above (Figure 5.1). S stands for Svínafellsjökull, V – Virkisjökull, K – Kviarjökull, F – Fjallsjökull, SK – Skaftafellsjökull, SD – Skeiðarárjökull.

Symbol	Sample	Type	Symbol	Sample	Type
S1	SV-TopIce-3105	sediment	V10	VK-YM-0206	soil
S2	SV-BI-2705	sediment	V11	VK-YM-1909	soil
S3	SV-BI-3105	sediment	V12	VK-M2-1909	soil
S4	SV-PL-2905	sediment	V13	VK-OM-0206	soil
S5	SV-PL-2109	sediment	V14	VK-OM-1909	soil
S6	SV-YM-2009	sediment	V15	VK-RM-2709	soil
S7	SV-R-3005	sediment	K1	KV-PL-2409	sediment
S8	SV-R-2109	soil	K2	KV-R-2409	soil
S9	SV-LM-2905	soil	K3	KV-LM-2409	soil
S10	SV-LM-2109	soil	K4	KV-M2-2409	soil
S11	SV-LIAM-3005	soil	K5	KV-M3-2409	soil
S12	SV-LIAM-2109	soil	F1	FJ-PL-2309	sediment
S13	SV-2.5K-3105	soil	F2	FJ-R-2309	sediment
S14	SV-2.5K-2109	soil	F3	FJ-LM-2309	soil
V1	VK-SIS-0206	sediment	F4	FJ-M2-2309	soil
V2	VK-SI-0206	sediment	F5	FJ-M3-2309	soil
V3	VK-BI-0206	sediment	SK1	SK-PL-2209	sediment
V4	VK-US-2605	sediment	SK2	SK-R-2209	sediment
V5	VK-US-1909	sediment	SK3	SK-TM-2209	soil
V6	VK-PL-2605	sediment	SK4	SK-M2-2209	soil
V7	VK-PL-1909	sediment	SD1	SD-LM-2609	soil
V8	VK-DS-2705	sediment	SD2	SD-M2-2609	soil
V9	VK-DS-2009	sediment			

PERMANOVA analysis showed that there was a statistically significant difference between ice sediment, sediment, and soil samples ( $p$ -value < 0.05). PCA plot demonstrated that despite some overlap, ice sediment samples were generally clustered to the top left of sediment and soil samples away from TC, TN, and TOC (Figure 5.1a). Sediments and soils had larger overlap: sediments formed a tighter cluster along PC1, indicative of little variation in chemistry, while soil samples were located along PC2, indicative of more variation in chemistry. Some samples from Svínafellsjökull (S11, S13 and S14, see Table 5.2 for sample names) occupied the lower right corner of the plot, along the TC, TN, TOC, and Zn PC vectors. On the contrary, samples from Skeiðarárjökull were in the upper right corner of the plot, indicating their mineral nature.

As for the transects, PERMANOVA analysis showed that there was no statistically significant difference between the samples from different transects ( $p$ -value  $> 0.05$ ). Individual testing between transects, revealed that there was a statistically significant difference between Skeiðarárjökull and Kviarjökull ( $p$ -value  $< 0.05$ ), and a marginally statistically significant difference between Skeiðarárjökull and Virkisjökull ( $p$ -value  $\approx 0.055$ ). The PCA plot in Figure 5.1b shows that samples collected along the Skeiðarárjökull transect were different from all other samples (top right corner), samples from Skaftafellsjökull had some overlap with Svínafellsjökull, while there was either some or complete overlap between the samples from all the other glaciers. Generally, samples from Svínafellsjökull and Virkisjökull are distributed along the PC1 and TC, TOC and TN vectors, indicative of variation in C, as well as some metals (Zn, P). Meanwhile, samples from Kviarjökull and Fjallsjökull are stretched along PC2, indicative of their mineral nature. Samples from Skeiðarárjökull are stretched along some metals (Al, Cu, Mg, Ca, Na) at  $90^\circ$  to TC, TOC and TN, indicative of lack of variation in OM content. Overall, samples from Kviarjökull, Fjallsjökull, Skaftafellsjökull, Svínafellsjökull, and Virkisjökull are grouped close to each other, suggesting that they have little variation in chemistry.

### 5.2.1.2 Total organic carbon concentration

Figure 5.2 shows sampling locations and total organic carbon (%) distribution along downstream transects of Kviarjökull, Fjallsjökull, Skaftafellsjökull, Skeiðarárjökull, Svínafellsjökull, and Virkisjökull glacier forelands in Iceland. TOC concentrations in samples collected at the Vatnajökull ice cap varied between 0 and  $5.24 \pm 2.68\%$  ( $n=3$ ) (Appendix 13). At Svínafellsjökull sediment samples had the lowest TOC concentration (mean TOC ranging from 0 to  $0.04 \pm 0.01\%$ ,  $n=3$ ), followed by ice samples (mean TOC varied between 0.05% to  $0.27 \pm 0.46\%$ ,  $n=3$ ), with soil samples having the highest concentration (mean TOC varied from  $0.03 \pm 0.02\%$  to  $5.24 \pm 2.68\%$ ,  $n=3$ ) (Appendix 13, Figure 5.2). Similarly, mean TOC concentrations at the Virkisjökull glacier varied between  $0.06 \pm 0.05\%$  ( $n=3$ ) to 0.08% in ice samples, from below 0.01% to  $0.03 \pm 0.01\%$  ( $n=3$ ) in sediment samples and from  $0.02 \pm 0.01\%$  ( $n=3$ ) to  $4.71 \pm 3.07\%$  ( $n=3$ ) in soil samples. Mean TOC concentrations in the sediment sample from Kviarjökull (KV-PL-2409) were very low  $0.02 \pm 0.01\%$  ( $n=3$ ), while they varied from  $0.05 \pm 0.01\%$  ( $n=3$ ) to  $0.59 \pm 0.12\%$  ( $n=3$ ) in soil samples. These values were lower than

that at Svínafellsjökull and Virkisjökull. Similarly, at Fjallsjökull, sediment samples had low mean TOC and varied between  $0.02\pm 0.01\%$  ( $n=3$ ) to  $0.04\pm 0.04\%$  ( $n=3$ ), while soil samples had higher values from  $0.14\pm 0.06\%$  ( $n=3$ ) to  $1.44\pm 0.43\%$  ( $n=3$ ). At Skaftafellsjökull, sediment samples had low mean TOC concentration ( $0.01\pm 0.01\%$  to  $0.02\pm 0\%$ ,  $n=3$ ) and soil samples varied from a lower ( $0.02\pm 0.01\%$ ,  $n=3$ ) to a higher value ( $0.57\pm 0.1\%$ ,  $n=3$ ), which is similar to the findings in samples from Kviarjökull. Soil samples from the Skeiðarárjökull glacier had the lowest mean TOC concentrations compared to all other sites from the Vatnajökull ice cap, ranging from  $0.03\pm 0.01\%$  ( $n=3$ ) to  $0.2\pm 0.06\%$  ( $n=3$ ) (Appendix 13, Figure 5.2). To summarize, samples from Svínafellsjökull and Virkisjökull had the highest TOC concentrations with the largest range, followed by Fjallsjökull. Samples from Kviarjökull and Skaftafellsjökull had lower TOC values, while the values recorded in soil samples from Skeiðarárjökull were comparable to that in sediments in other glaciers. Generally, sediment and ice samples across sites in Iceland had lower mean TOC ( $0.04\pm 0.96\%$ ,  $n=3$ , ranging from 0 to  $0.27\pm 0.46\%$ ,  $n=3$ ) compared to soil samples ( $0.96\pm 1.62\%$ ,  $n=3$ , ranging from  $0.02\pm 0.01\%$  to up to  $5.24\pm 2.68\%$ ,  $n=3$ ) (Appendix 13). It is worth noting that across all six investigated glaciers, the TOC concentration in soil samples increased along the downstream transect and/or with the age of moraines.

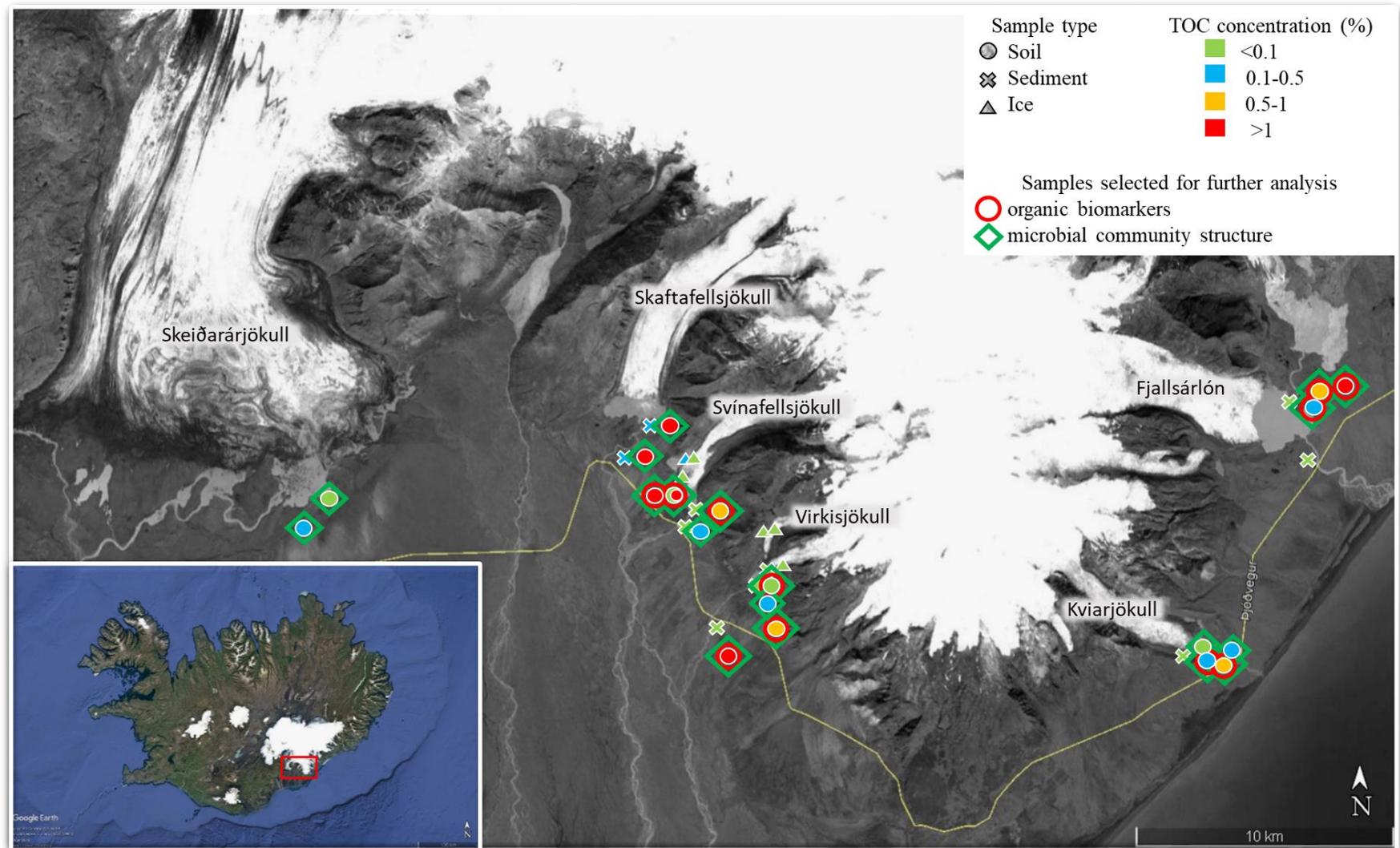


Figure 5.2. Sampling locations and TOC (%) distribution along downstream transects of Svínafellsjökull, Virkisjökull, Kviarjökull, Fjallsjökull, Skaftafellsjökull and Skeiðarárjökull glaciers, SE Iceland (Modified from Google Earth, 2012).

There was an overall trend of TOC increasing in relation to the downstream transects across Virkisjökull, Kviarjökull, Fjallsjökull, Skaftafellsjökull and Skeiðarárjökull glaciers (Figure 5.3). This was especially noticeable across soil samples (younger and older soils). However, Svínafellsjökull downstream transect had an initial upward trend, followed by decrease in the mean TOC concentrations (from SV-LIAM-3005 to SV-2.5K-3105/2109).

There was some variation between TOC concentrations in sediment samples at Svínafellsjökull and Virkisjökull collected in May-June 2018 and September 2019. Mean TOC in sediment samples at the Svínafellsjökull increased from 0.01% to  $0.04 \pm 0.01\%$  ( $n=3$ ) for the lake samples (SV-PL-2905 and SV-PL-2109, respectively) and from  $<0.01\%$  to  $0.28 \pm 0.14\%$  ( $n=3$ ) for the river samples (SV-R-3005 and SV-R-2109). At Virkisjökull, mean TOC concentration increased from  $<0.01\%$  to 0.01% in samples from the meltwater stream (VK-US-2605 and VK-US-1909) and the proglacial lake (VK-PL-2605 and VK-PL-1909), and from 0.01 to  $0.03 \pm 0.01\%$  ( $n=3$ ) in samples from the Virkisá river. As for the soil samples collected from Svínafellsjökull and Virkisjökull in 2018 and 2019, while most samples had similar values (SV-LM-2905 had mean TOC of  $0.54 \pm 0.19\%$ ,  $n=3$  and SV-LM-2109 had values of  $0.75 \pm 0.25\%$ ,  $n=3$ ; SV-2.5K-3105 –  $2.97 \pm 1.8\%$ ,  $n=4$  and SV-2.5K-2109 –  $2.02 \pm 0.9\%$ ,  $n=4$ ; VK-YM-0206 –  $0.07 \pm 0.02\%$ ,  $n=3$  and VK-YM-1909 –  $0.02 \pm 0.01\%$ ,  $n=3$ ; VK-OM-0206 –  $0.58 \pm 0.36\%$ ,  $n=3$  and VK-OM-1909 –  $0.75 \pm 0.07\%$ ,  $n=3$ ), there was a noticeable difference in SV-LIAM-3005 (mean TOC of  $5.24 \pm 2.68\%$ ,  $n=3$ ) and SV-LIAM-2109 (mean TOC of  $0.03 \pm 0.02\%$ ,  $n=3$ ).

5 Soil development at Vatnajökull ice cap, Iceland

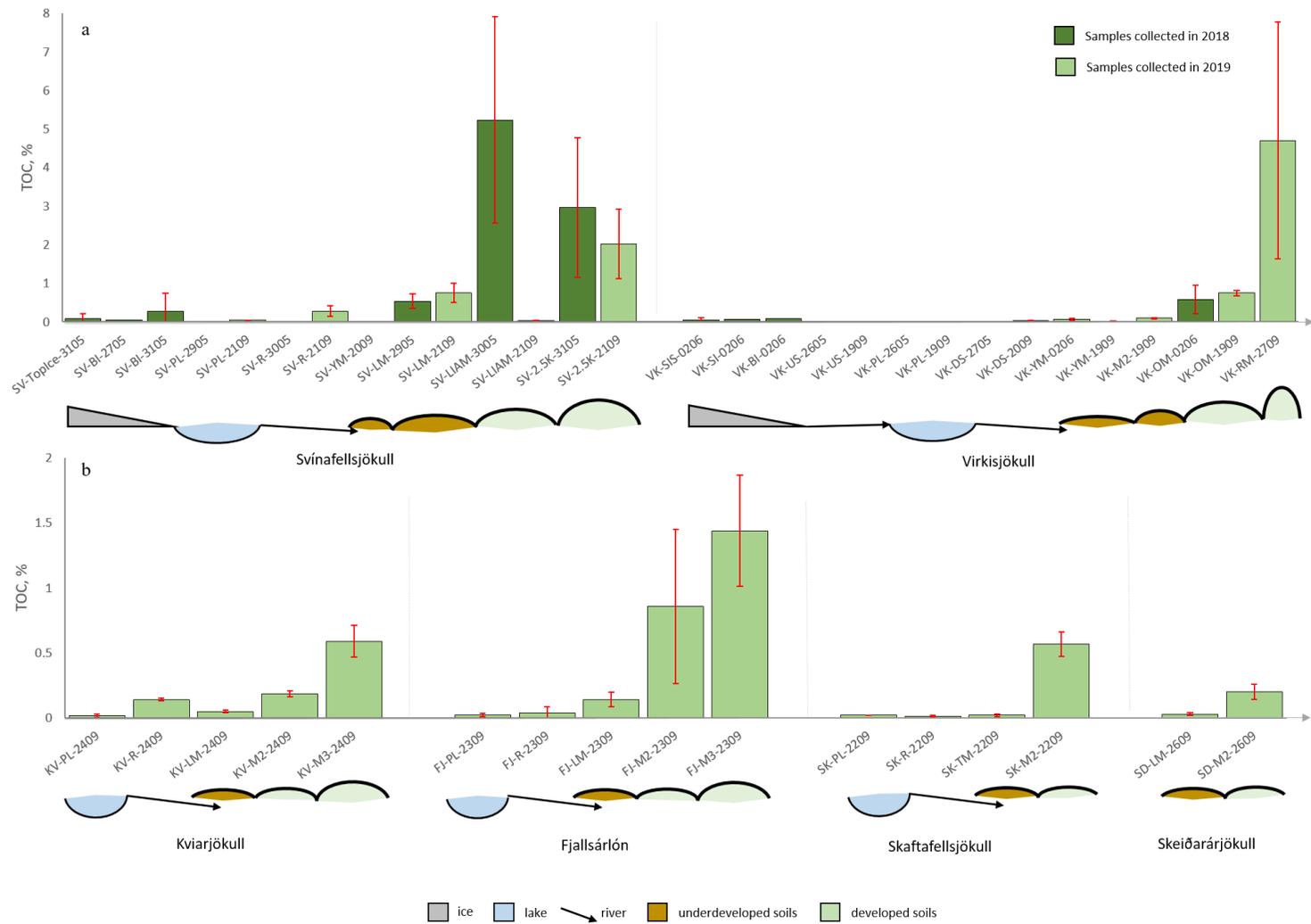


Figure 5.3. Mean distribution of TOC (%) along the (a) Svínafellsjökull and Virkisjökull, (b) Kviarjökull, Fjallsjökull, Skaftafellsjökull and Skeiðarárjökull transects. Error bars represent  $\pm$  one standard deviation on field replicate samples (triplicates for most of the samples, see Appendix 13 for more details) collected from individual sampling locations. Charts at the bottom of the graph show downstream (left to right) transect profiles.

### 5.2.1.3 Total nitrogen and C/N ratio

Across the six sites, TN concentration across all samples ranged from less than  $0.01\pm 0.02\%$  (n=3) to  $0.44\pm 0.19\%$  (n=3), with a mean of  $0.05\pm 0.07\%$  (Appendix 13). Similar to TOC, the highest mean TN concentration was at Svínafellsjökull ( $0.08\pm 0.12\%$ , n=3), followed by Fjallsjökull ( $0.04\pm 0.03\%$ , n=3), Virkisjökull ( $0.03\pm 0.03\%$ , n=3), Kviarjökull ( $0.03\pm 0.02\%$ , n=3), Skaftafellsjökull ( $0.03\pm 0.01\%$ , n=3) and Skeiðarárjökull ( $0.03\pm 0.01\%$ , n=3). C/N ratios ranged along each transect: from  $0.17\pm 0.34$  (n=3) to  $20.41\pm 15.14$  (n=3) at Svínafellsjökull, Virkisjökull –  $0.10\pm 0.15$  (n=3) to  $39.44\pm 33.40$  (n=3), Kviarjökull –  $1.11\pm 1.50$  (n=3) to  $12.19\pm 9.00$  (n=3), Fjallsjökull –  $1.11\pm 1.50$  (n=3) to  $19.32\pm 21.88$  (n=3), Skaftafellsjökull –  $0.67\pm 1.00$  (n=3) to  $12.13\pm 13.40$  (n=3), and Skeiðarárjökull –  $1.00\pm 1.00$  (n=3) to  $10.89\pm 8.67$  (n=3) (see Appendix 13 for more details). The lowest values were those of sediments or the younger soil sample at Skeiðarárjökull, the highest were for the oldest soil samples at each transect. The large variation in range, especially at Virkisjökull, Svínafellsjökull, and Fjallsjökull, was due to the low TN values, while TOC varied much more.

Moreover, there was a strong positive relationship between TOC (%) and TN (%) at Svínafellsjökull ( $R^2=0.91$ , Pearson correlation=0.95), Virkisjökull ( $R^2=0.82$ , Pearson correlation=0.91), Fjallsjökull ( $R^2=0.92$ , Pearson correlation=0.96) and Skaftafellsjökull ( $R^2=0.94$ , Pearson correlation=0.97), weak relationship at Kviarjökull ( $R^2=0.35$ , Pearson correlation=0.59) and no relationship for samples from Skeiðarárjökull ( $R^2=0.05$ , Pearson correlation=-0.22) (Figure 5.4), suggesting that TN was organic in nature in samples collected from sites with high TOC and TN correlation.

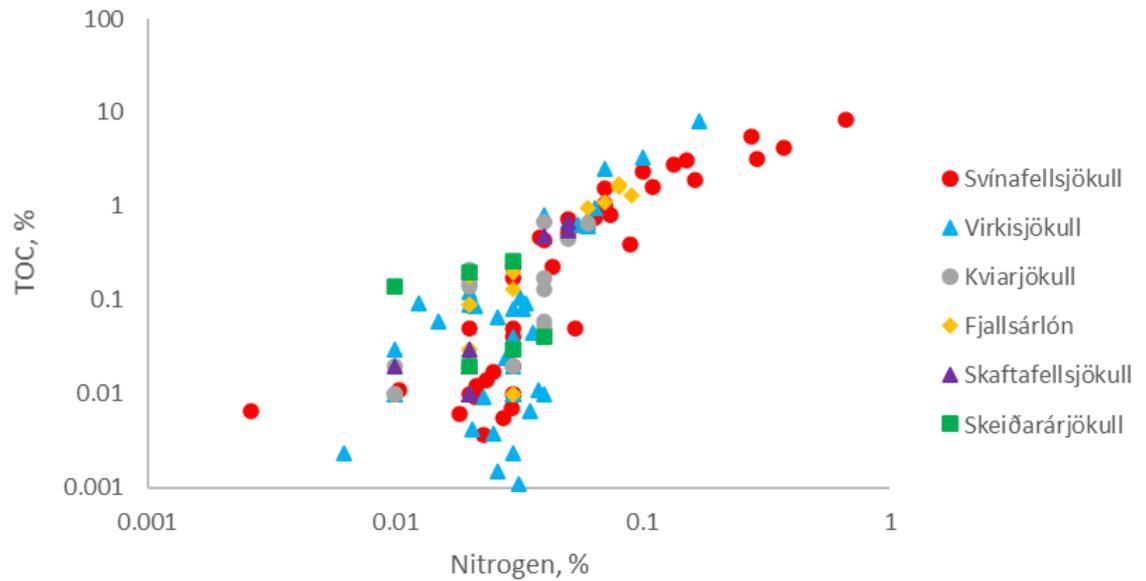


Figure 5.4. Relationship between TOC (%) and total nitrogen (%) for six transects at the Vatnajökull ice cap (logarithmic axes).

#### 5.2.1.4 Metal concentration

Figure 5.5 shows distribution of mean of total and individual metal concentrations (mg/kg) along the Svínafellsjökull, Virkisjökull, Kviarjökull, Fjallsjökull, Skaftafellsjökull, and Skeiðarárjökull downstream transects. Mean total metal concentration of samples collected from each glacier foreland was different, concentrations being much higher at Skeiðarárjökull ( $34882 \pm 1073$  mg/kg,  $n=2$ ) compared to other sites: Skaftafellsjökull ( $22462 \pm 1703$  mg/kg,  $n=4$ ), Fjallsjökull ( $19556 \pm 3040$  mg/kg,  $n=5$ ), Svínafellsjökull ( $18721 \pm 3501$  mg/kg,  $n=14$ ), Virkisjökull ( $16317 \pm 3332$  mg/kg,  $n=15$ ) and Kviarjökull ( $15968 \pm 2278$  mg/kg,  $n=5$ ). Samples collected from Skeiðarárjökull (35640 mg/kg in SD-LM-2609 and 34123 mg/kg in SD-M2-2609) had the highest metal concentrations, while some ice sediment samples from Virkisjökull (9628 mg/kg in VK-SIS-0206 and 9053 mg/kg in VK-SI-0206) and Svínafellsjökull (10384 mg/kg in SV-Toplce-3105) had the lowest values. Mean and SD of individual metal concentrations in samples from each glacier are shown in Table 5.3 (see Appendix 14 for more details). Fe, Al, Ca and Mg had the highest concentrations, followed by Na, K, P, Mn and S, while Zn, Cu, Cr, Co, Ni, Cd, and Pb had lower concentrations (Table 5.3).

Table 5.3. Mean and standard deviation (mg/kg) of metal concentrations in samples from Svínafellsjökull, Virkisjökull, Kviarjökull, Fjallsjökull, Skaftafellsjökull, and Skeiðarárjökull glaciers in Iceland.

<b>Metal</b>	<b>Svínafellsjökull</b>		<b>Virkisjökull</b>		<b>Kviarjökull</b>	
Fe	9486	±2321	8431	±2054	7946	±1168
Al	3349	±641	3041	±880	3108	±555
Ca	3198	±494	2628	±605	2291	±385
Mg	1523	±266	1197	±159	1501	±280
Na	546	±244	491	±193	581	±112
K	199	±63	193	±38	271	±39
P	184	±35	171	±33	119	±22
Mn	146	±26	118	±23	117	±16
S	56	±37	21	±23	7	±7
Zn	11.59	±3.79	10.11	±3.24	10.61	±1.15
Cu	10.23	±2.28	6.52	±1.26	6.4	±1.1
Cr	3.46	±2.64	2.37	±2.44	2.13	±0.34
Co	4.84	±0.88	4.42	±0.77	3.97	±0.43
Ni	2.25	±0.44	1.73	±0.53	2.44	±0.34
Cd	0.6	±0.16	0.54	±0.15	0.55	±0.07
Pb	0.42	±0.28	0.33	±0.31	0.66	±0.68
<b>Metal</b>	<b>Fjallsjökull</b>		<b>Skaftafellsjökull</b>		<b>Skeiðarárjökull</b>	
Fe	10868	±1315	11401	±775	12550	±145
Al	3089	±954	3926	±974	7340	±181
Ca	2701	±813	3751	±758	8114	±651
Mg	1793	±278	1992	±182	4347	±272
Na	541	±306	782	±220	1852	±111
K	157	±54	208	±72	295	±9
P	178	±12	197	±17	95	±8
Mn	137	±14	145	±13	180	±3
S	59	±24	23	±10	60	±2
Zn	13.01	±1.12	12.43	±0.68	10.17	±1.03
Cu	9.56	±1.38	11.65	±0.91	17.62	±0.66
Cr	2.4	±0.25	3.95	±0.73	10.16	±1.14
Co	5.03	±0.46	5.30	±0.25	5.76	±0.36
Ni	2.21	±0.2	3.02	±0.31	4.82	±0.2
Cd	0.75	±0.09	0.75	±0.02	0.8	±0.06
Pb	0.92	±0.83	0.24	±0.06	0.12	±0.17

The PCA plot (Figure 5.1b) indicates that samples from Svínafellsjökull and Virkisjökull are distributed along PC2, which is correlated with Cr, Ni, Zn, P, S and Pb. On the contrary, samples from Kviarjökull, Fjallsjökull and Skaftafellsjökull are distributed along PC1, which is correlated with Mn, Mg, Na, Ca, Al, Fe, Co, Cd, Cu and K. Samples from Skeiðarárjökull

are away from the rest of the samples from other glaciers, being influenced by Mg, Ca, Al, Na, Cu, Ni and Cr.

No particular pattern was observed in concentrations of individual metals, though overall there was an upward trend in distribution of total metals along the downstream transects of Svínafellsjökull (Figure 5.5a), Kviarjökull and Fjallsjökull (Figure 5.5b), while no particular trend in downstream distribution was observed at Virkisjökull, Skaftafellsjökull and Skeiðarárjökull. Moreover, even without taking into account soil samples collected from the glacier foreland of Skeiðarárjökull, which had considerably higher metal concentrations, soil samples in all other glaciers had higher values of all measured metals compared to sediments.

5 Soil development at Vatnajökull ice cap, Iceland

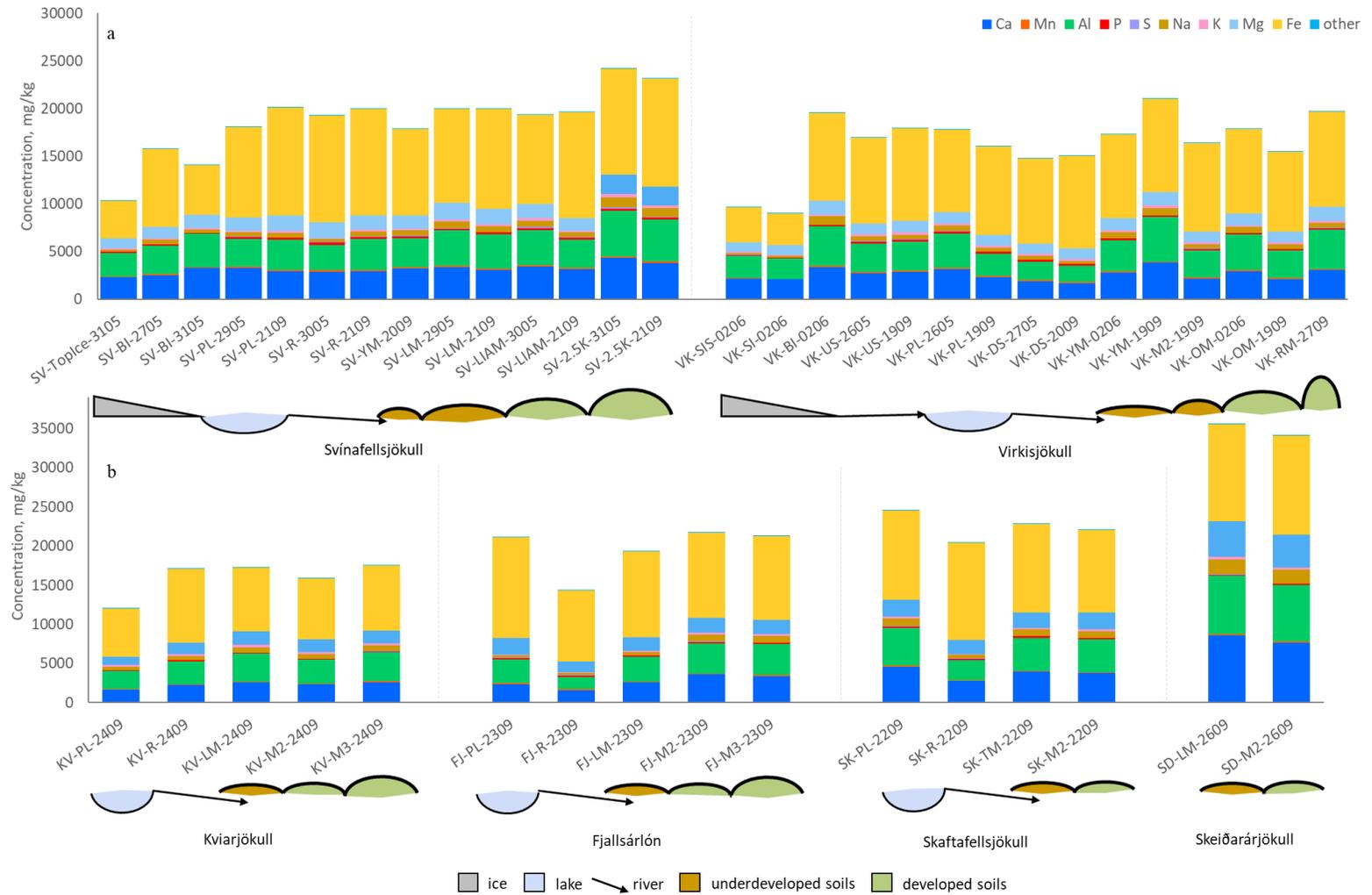


Figure 5.5. Stacked bar chart showing mean concentration of metals (mg/kg) along (a) Svínafellsjökull and Virkisjökull, (b) Kviarjökull, Fjallsjökull, Skaftafellsjökull and Skeiðarárjökull transects in Iceland. Other metals include Cr, Co, Ni, Cu, Zn, Pb and Cd.

## 5.2.2 Biomarkers

Soil samples with high TOC content from four glaciers (Svínafellsjökull, Virkisjökull, Kviarjökull and Fjallsjökull) were further analysed for organic biomarkers (see Appendix 13 for the samples chosen for organic biomarker analysis - TOC values highlighted in red) to see soil development pattern along the downstream transects of glacier forelands (see Figure 5.2 for the location of the selected samples on a map). No samples were chosen from Skaftafellsjökull and Skeiðarárjökull due to the low TOC concentrations in all or some soil samples, i.e., only one sample from Skaftafellsjökull had high enough TOC content, which wouldn't have contributed to the soil development pattern at the glacier foreland. Moreover, due to the low TOC content, as not many signals of organic biomarkers were found in sediment samples from Sweden, no sediment samples were investigated for organic biomarkers in Iceland.

### 5.2.2.1 Difference between transects investigated for organic biomarker

Figure 5.6 shows NMDS plots based on Bray-Curtis similarity indexes of the total BHP compounds and total GDGTs in samples analysed for organic biomarkers across four glaciers in Iceland. The plots did not show clustering of samples by glacier. However, it could be seen that samples from Fjallsjökull occupied the top left part of the plots, samples from Virkisjökull – the top half, samples from Kviarjökull were clustered close to each other in Figure 5.6b, suggesting that there was not much difference in GDGT concentrations, while samples from Svínafellsjökull were scattered around the plots. Even though clear clustering of samples by transect wasn't observed, PERMANOVA showed a significant variance in total BHPs ( $p$ -value = 0.05) and total GDGTs between sites ( $p$ -value < 0.05).

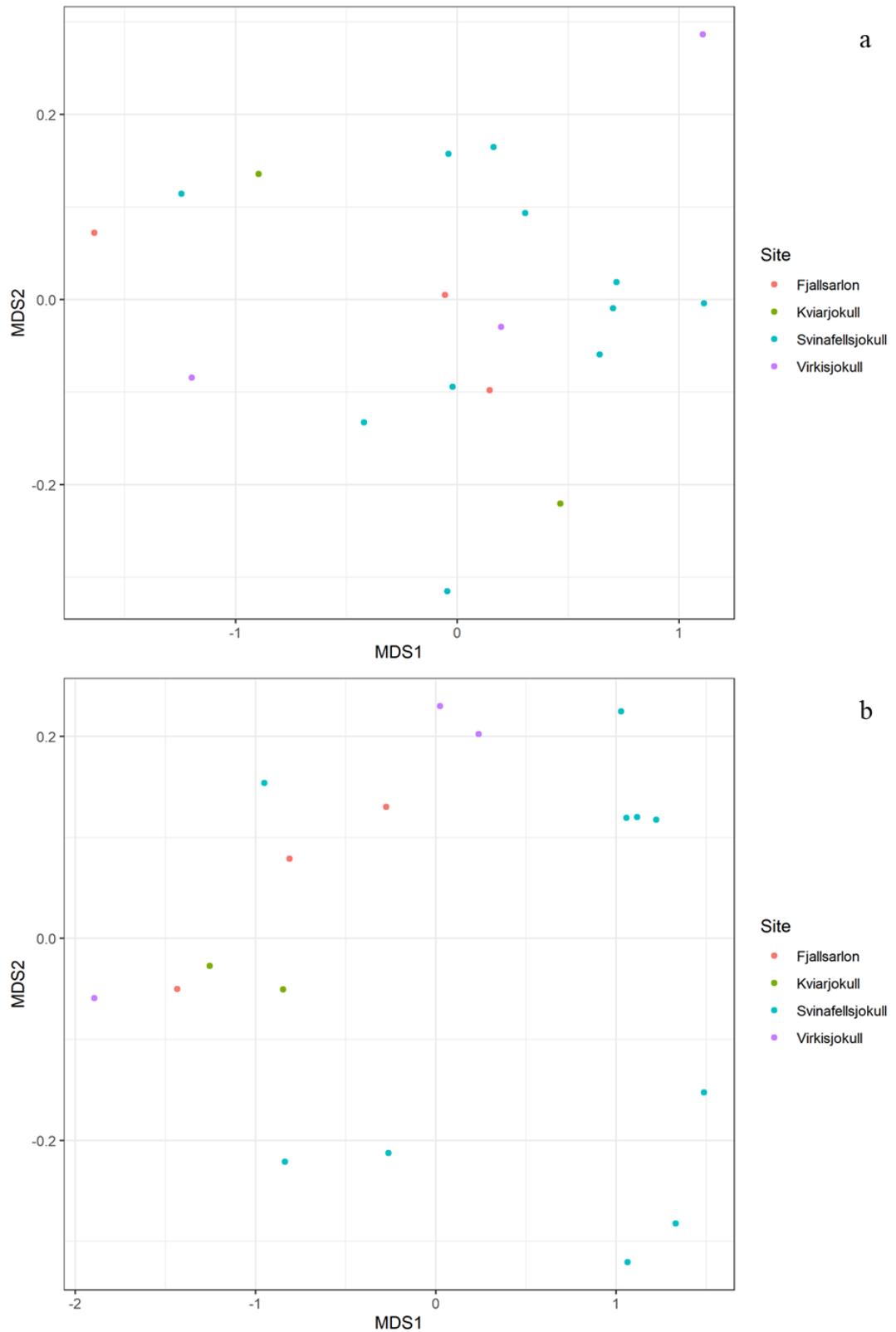


Figure 5.6. NMDS plots showing difference in (a) BHPs and (b) GDGTs between four sites in Iceland. When the data values were larger than common abundance class scales, Wisconsin double standardisation was performed; when values looked very large, sqrt transformation was also performed; and distance matrix based on Bray–Curtis dissimilarity was obtained.

### 5.2.2.2 BHP concentrations and distribution pattern

BHPs were present in all analysed samples from the Vatnajökull ice cap. Up to 28 individual BHPs, grouped in five main categories (BHT, amino, sugar, aminosugar and soil markers) were identified in the samples (Appendix 16). The mean relative concentration of total BHPs was  $2469 \pm 1404$   $\mu\text{g/g}$  TOC ranging from 944 to 4796  $\mu\text{g/g}$  TOC, while mean absolute concentration was  $25 \pm 23$   $\mu\text{g/g}$  sediment. BHTs were the most abundant individual BHP compound (mean relative concentration of  $1307 \pm 795$   $\mu\text{g/g}$  TOC), followed by methylated BHT ( $320 \pm 174$   $\mu\text{g/g}$  TOC), 35-aminotriol ( $99 \pm 95$   $\mu\text{g/g}$  TOC), BHT glucosamine ( $87 \pm 95$   $\mu\text{g/g}$  TOC) and BHT cyclitol ether ( $71 \pm 84$  251  $\mu\text{g/g}$  TOC). Meanwhile, adenosylhopane ( $190 \pm 94$   $\mu\text{g/g}$  TOC) and adenosylhopane type 3 ( $82 \pm 72$   $\mu\text{g/g}$  TOC) were the most abundant soil marker BHPs.

There was no overall trend of increase in relative concentration of total BHPs or soil specific BHPs along the downstream transect of glaciers in Iceland (Figure 5.7). Therefore, it would be the best to look at each glacier foreland separately.

#### ***Svínafellsjökull***

The relative concentration of total BHPs ranged from 944 to 2532  $\mu\text{g/g}$  TOC (Appendix 16). There was no build-up of relative concentration of total BHPs with the age of moraines, as the youngest moraine, SV-PL-2905, had the highest relative concentration, suggesting that BHPs are not increasing as fast as OC. Meanwhile, the  $R'_{\text{soil}}$ , indicative of increasing importance of soil marker BHPs, increased from 0.27 to 0.35, following an upward trend downstream (Figure 5.7). Fractionally, SV-LIAM-3005 and SV-2.5K-3105 had similar distribution of compound groups, with similar soil marker BHPs (28% at SV-LIAM-3005 and 27% at SV-2.5K-3105) (Figure 5.8). SV-PL-2905 had higher fraction of aminosugars (19%), mostly comprised of BHT cyclitol ether (220  $\mu\text{g/g}$  TOC) and BHT glucosamine (123  $\mu\text{g/g}$  TOC).

#### ***Virkisjökull***

The relative concentration of total BHPs decreased downstream from 4459  $\mu\text{g/g}$  TOC (VK-YM-0206) to 2342  $\mu\text{g/g}$  TOC (VK-RM-2709) (Appendix 16). The initial increase in the  $R'_{\text{soil}}$  from the youngest moraine, VK-YM-0206 (0.15), to the next older moraine, VK-OM-0206

(0.24), was followed by a decrease in the most developed moraine, VK-RM-2709 (0.11) (Figure 5.7). Similar to SV-PL-2905 from Svínafellsjökull, VK-YM-0206 had the highest fraction of aminosugars (17%), with high concentrations of BHT cyclitol ether (251  $\mu\text{g/g}$  TOC) and BHT glucosamine (332  $\mu\text{g/g}$  TOC). There was an increase in the fractional abundance of BHTs (82%) at VK-RM-2709 (Figure 5.8).

### ***Kviarjökull***

At the Kviarjökull, the relative concentration of total BHPs increased from 3168  $\mu\text{g/g}$  TOC (KV-M2-2409) to 4796  $\mu\text{g/g}$  TOC (KV-M3-2409), while the  $R'_{\text{soil}}$  decreased from 0.15 to 0.09 (Figure 5.7). Comparably, the fractional abundance of soil specific BHPs decreased from 13% (KV-M2-2409) to 7% (KV-M3-2409). KV-M3-2409 had higher fractional abundance of sugar compounds (8%) compared to KV-M2-2409 (1%) (Figure 5.8).

### ***Fjallsjökull***

The relative concentration of total BHPs in samples collected at Fjallsjökull ranged from 1053  $\mu\text{g/g}$  TOC (FJ-LM-2309) to 1466  $\mu\text{g/g}$  TOC (FJ-M2-2309) (Appendix 16). Both the relative concentration of total BHPs and the  $R'_{\text{soil}}$  followed a trend of initial increase, followed by decrease in the oldest moraine (Figure 5.7). The  $R'_{\text{soil}}$  increased from 0.15 (FJ-LM-2309) to 0.17 (FJ-M2-2309) and subsequently decreased to 0.10 (FJ-M3-2309). Similarly, the fractional abundance of soil specific biomarkers increased from 12% (FJ-LM-2309) to 13% (FJ-M2-2309), followed by decrease down to 8% (FJ-M3-2309) (Figure 5.8).

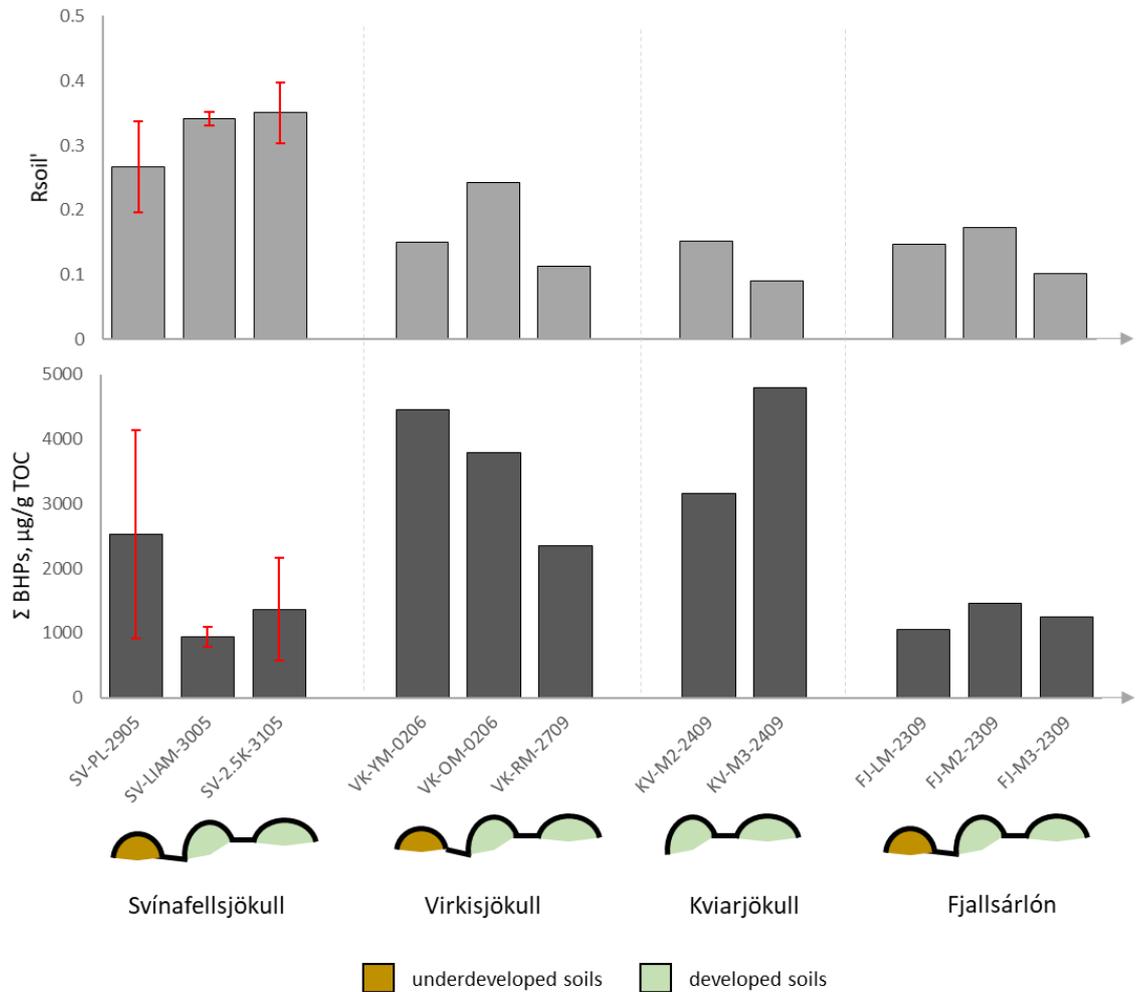


Figure 5.7. Average distribution of total BHPs ( $\mu\text{g/g TOC}$ ) and  $R'_{soil}$  along transects at Svínafellsjökull, Virkisjökull, Kviarjökull and Fjallsjökull glaciers in Iceland. Error bars represent  $\pm$  one standard deviation on field replicate samples (mostly triplicates, see Appendix 16 for more details) collected from individual sampling locations at Svínafellsjökull. Only one field replicate per sampling location from other glaciers was analysed for organic biomarkers. Charts at the bottom of the graph show downstream (left to right) transect profiles.

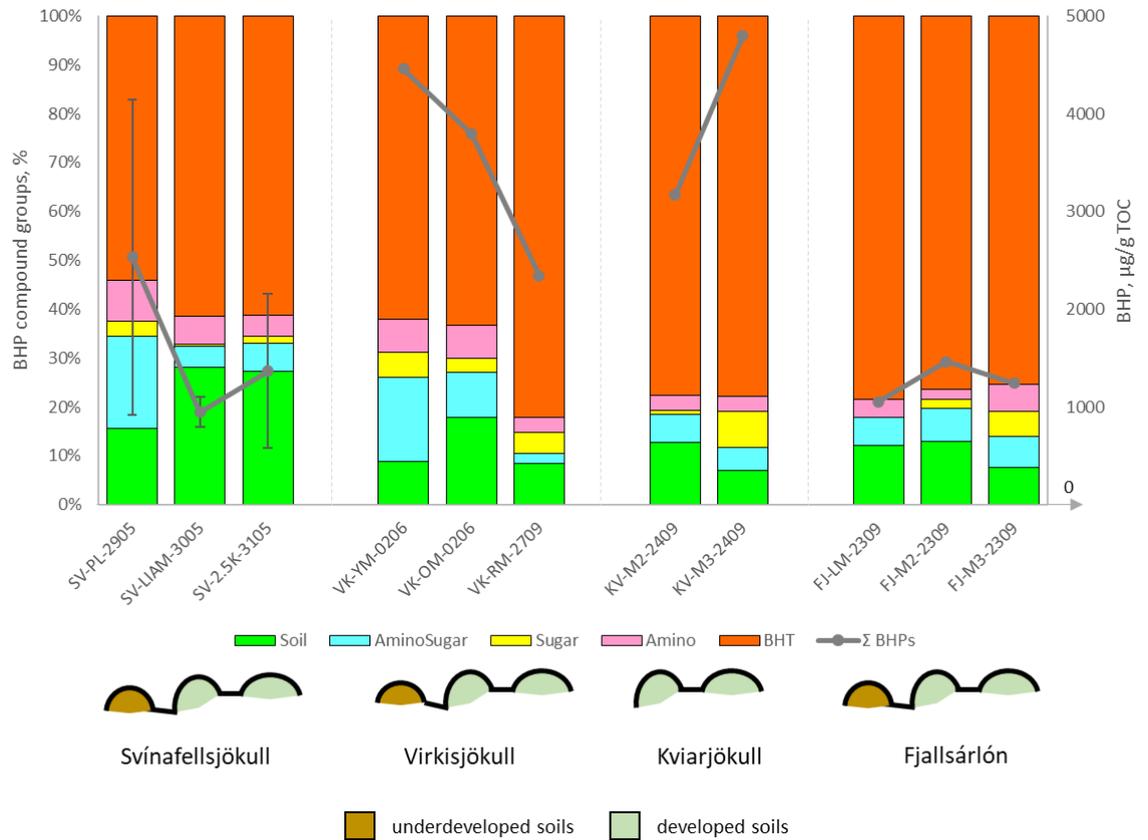


Figure 5.8. Total BHP concentration (grey circles and lines) and fractional abundance of BHP compound groups (BHTs, aminos, sugars, aminosugars and soil BHPs; coloured bars) at Svínafellsjökull, Virkisjökull, Kviarjökull and Fjallsjökull glacier transects in Iceland. Error bars represent  $\pm$  one standard deviation on field replicate samples collected from individual sampling locations at the Svínafellsjökull glacier. Only one field replicate per sampling location from other glaciers was analysed for organic biomarkers. Charts at the bottom of the graph show downstream (left to right) transect profiles.

### 5.2.2.3 GDGT concentrations and distribution along the transects

Samples were also analysed for GDGTs. Branched GDGTs were found in all samples analysed for organic biomarkers at the Vatnajökull icecap (Figure 5.9), the most abundant brGDGTs being Ia, IIa and IIIa compounds. Ib, Ic, IIb and IIc compounds were also present in samples SV-LIAM-3005 and SV-2.5K-3105. SV-LIAM-3005 also contained very small amount of crenarchaeol GDGT (1%). The mean relative concentration of total GDGTs was  $112 \pm 74$  µg/g TOC, varying from 35 to 258 µg/g TOC (Appendix 16), while the mean absolute concentration was  $1.52 \pm 2.14$  µg/g sediment. Svínafellsjökull (229 µg/g TOC, SV-2.5K-3105) and Virkisjökull (258 µg/g TOC, VK-OM-2709) glaciers had the highest concentrations compared to the other sites in Iceland.

There was no overall trend of distribution of relative concentration of total GDGTs with the age of moraines (Figure 5.9). Generally, the mean of relative concentration of total GDGTs was higher at Svínafellsjökull (154±69 µg/g TOC) and Virkisjökull (135±113 µg/g TOC), compared to Kviarjökull (96±53 µg/g TOC) and Fjallsjökull (59±27 µg/g TOC).

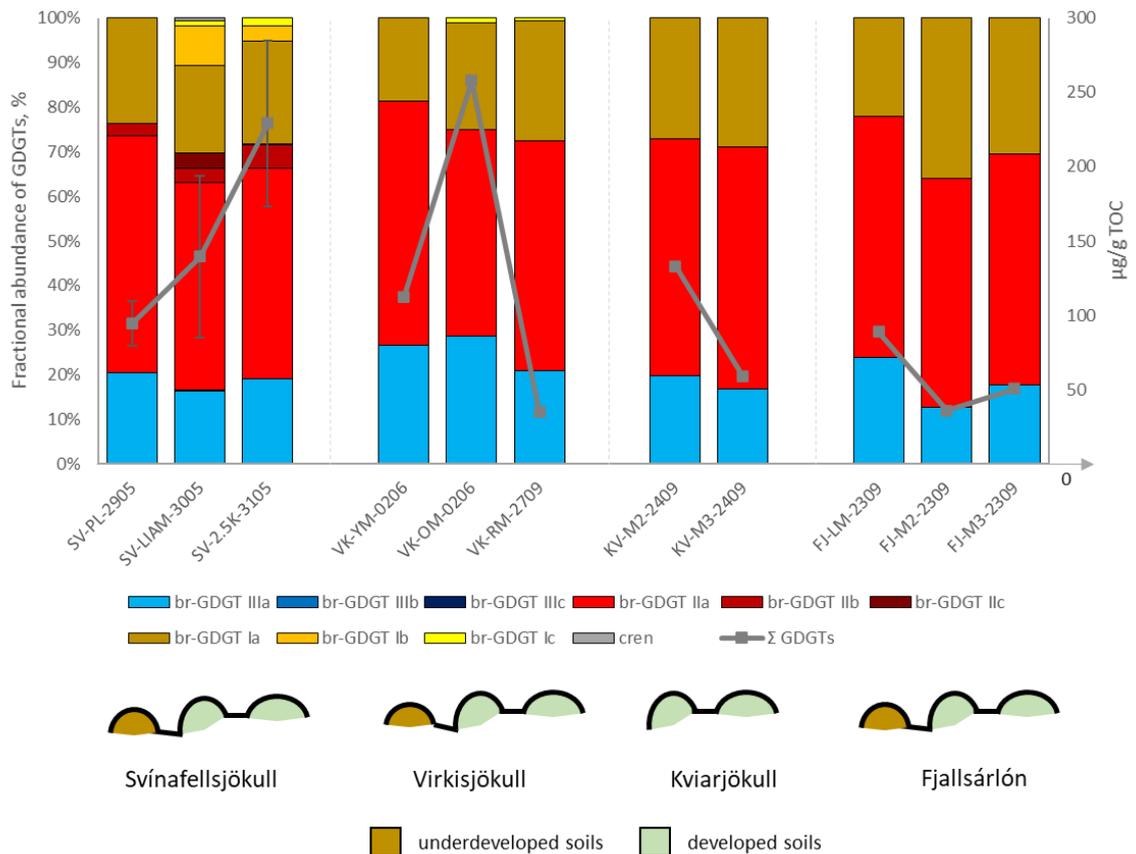


Figure 5.9. Relative concentration of total GDGTs (grey circles and lines) and fractional abundance of branched GDGT compounds and crenarchaeol GDGT (coloured bars) along downstream transects of the Svínafellsjökull, Virkisjökull, Kviarjökull and Fjallsjökull glaciers in Iceland. Error bars represent  $\pm$  one standard deviation on field replicate samples collected from individual sampling locations at the Svínafellsjökull glacier. Only one field replicate per sampling location from other glaciers was analysed for organic biomarkers. Charts at the bottom of the graph show downstream (left to right) transect profiles.

### 5.2.2.3.1 GDGT proxies

Based on GDGTs, some GDGT proxies were also analysed: soil pH varied from 6.1 to 7.3, while annual mean air temperature (MAT) fluctuated between 1.5 and 8.7 °C (Figure 5.10). Generally, soil pH and MAT followed a similar downward trend. At Svínafellsjökull, this trend inversely corresponded with total BHPs, did not correspond with the  $R'_{\text{soil}}$  and brGDGTs; at Virkisjökull, it was similar to the downstream distribution of total BHPs but did

not correspond to the  $R'_{\text{soil}}$  and brGDGTs; at Kviarjökull, soil pH and MAT had the downward trend inversely corresponded to total BHPs and similar to the  $R'_{\text{soil}}$  and brGDGTs; while at Fjallsjökull, MAT had the same trend as total BHPs and the  $R'_{\text{soil}}$  and inversely corresponded to brGDGTs and soil pH had a constant downward trend that did not correspond with other measurements.

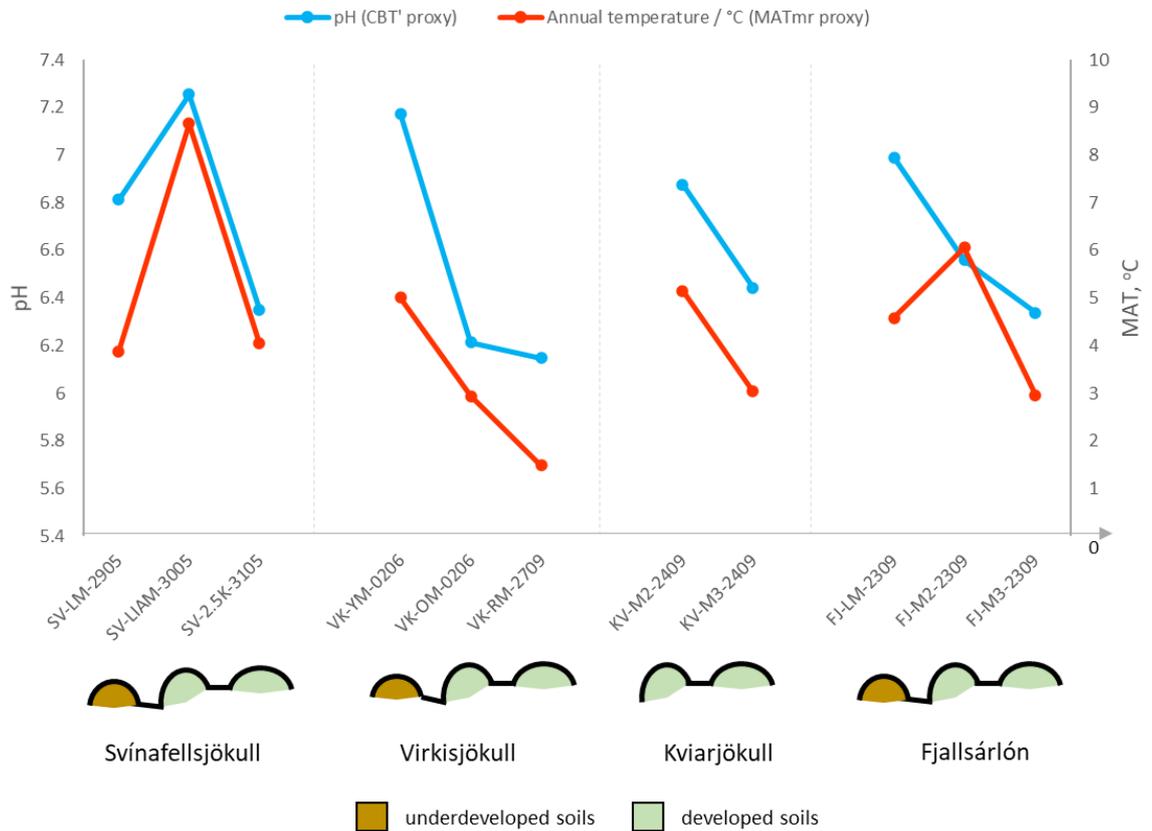


Figure 5.10. Downstream distribution of GDGT proxies, soil pH and annual mean air temperature (MAT, °C) at Svínafellsjökull, Virkisjökull, Kviarjökull and Fjallsjökull glaciers in Iceland.

#### 5.2.2.4 Relationship between TOC, BHPs and GDGTs

In order to determine if organic biomarkers were related to OC concentrations linear regression of TOC concentration (%) versus absolute concentration of biomarkers ( $\mu\text{g/g}$  sediment) were plotted for samples collected at the Vatnajökull ice cap. TOC (%) and absolute concentration of total BHPs ( $\mu\text{g/g}$  sediment) showed a positive relationship ( $R^2=0.61$ , Pearson correlation=0.78) (Figure 5.11a). Similarly, there was a significant correlation between TOC (%) and total GDGTs ( $\mu\text{g/g}$  sediment) with an  $R^2$  value of 0.74 and Pearson correlation of 0.86 (Figure 5.11b). On the contrary, there was no correlation

between total BHPs ( $\mu\text{g/g}$  sediment) and total GDGTs ( $\mu\text{g/g}$  sediment) ( $R^2=0.22$ , Pearson correlation=0.47) (Figure 5.11c).

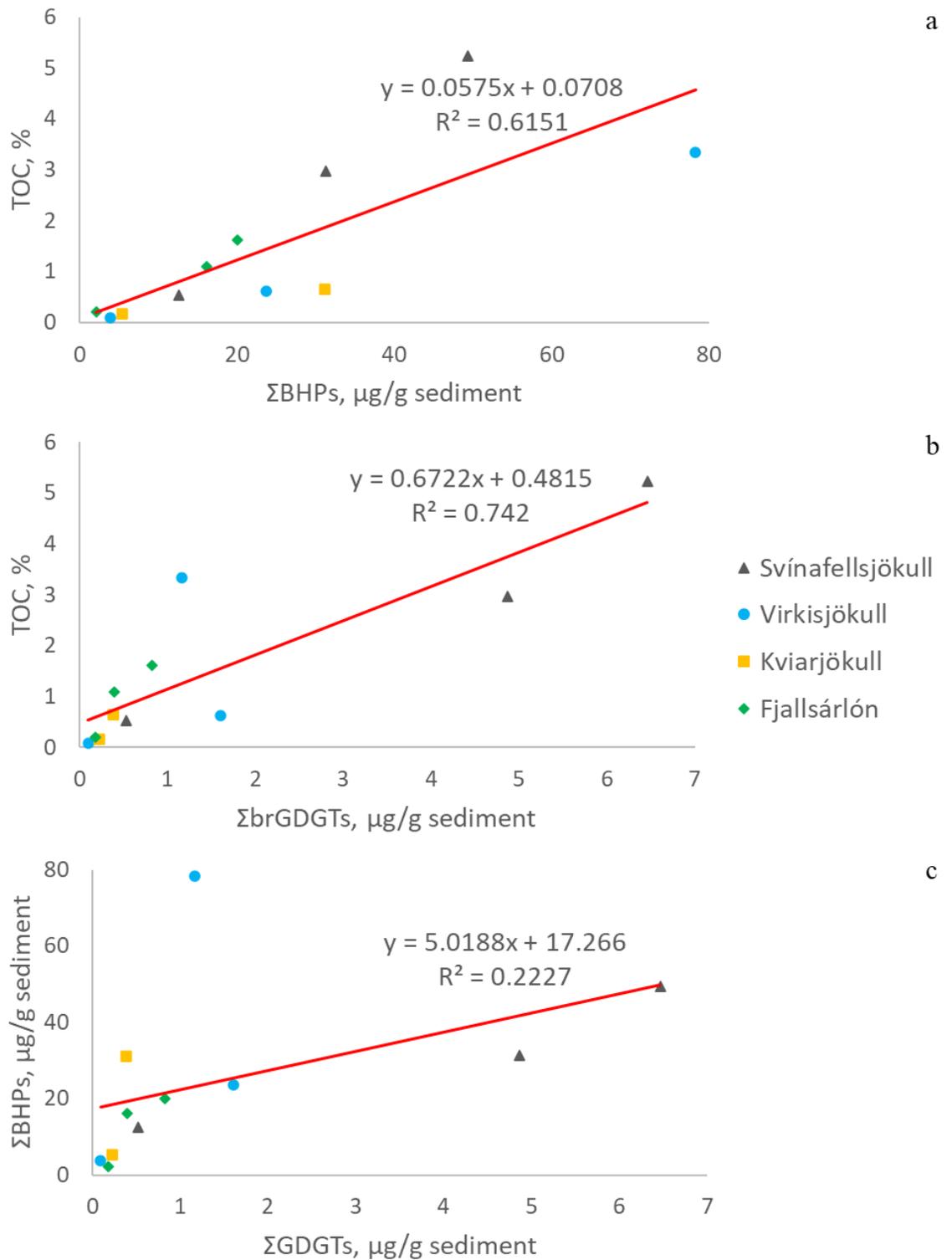


Figure 5.11. Relationship between total organic carbon (TOC) (%) and (a) total BHPs ( $\mu\text{g/g}$  sediment) and (b) total GDGTs ( $\mu\text{g/g}$  sediment), and (c) between total BHPs ( $\mu\text{g/g}$  sediment) and total brGDGTs ( $\mu\text{g/g}$  sediment) across sites at the Vatnajökull ice cap, SE Iceland.

## 5.2.3 Microbial analysis

### 5.2.3.1 Sample selection and DNA concentration

A total of 48 soil samples including some replicates from all six glaciers at the Vatnajökull ice cap in Iceland were analysed for microbial communities, with 16 samples from Svínafellsjökull, 12 samples from Virkisjökull, 8 from Kviarjökull, 5 from Fjallsjökull, 3 from Skaftafellsjökull and 4 from Skeiðarárjökull. Samples were chosen to represent downstream transects away from the glacier front (see Figure 5.2 for the location of the selected samples on a map). Most of these samples had sufficient DNA, and all of them produced visible bands in PCR. Samples collected from Iceland had a wide range of DNA concentration from BDL to 59.2 ng/ml (Appendix 18), samples from Svínafellsjökull having the highest concentration ( $21.87 \pm 15.66$  ng/ml) and samples from Kviarjökull having the lowest values ( $8.14 \pm 3.44$  ng/ml). There was a significant difference in DNA concentrations between samples from each transect (PERMANOVA analysis,  $p$ -value < 0.05).

### 5.2.3.2 Chemical characterisation of soil samples investigated for microbial community structure

In order to see if there was a difference in chemical properties between the glaciers PCA was conducted on the sub-set of soil samples selected for further microbial community analysis (Figure 5.12). PC1 explained 41.0% of total variance and had the strongest negative correlation with Ca, Na, Mg, Cr, Al, Ni and Cu, and less strong negative correlation with Fe and Mn. PC2 (22.5% of total variance) had strong negative correlation with TC, S, TOC and N, and negative correlation with P and Zn (Figure 5.12).

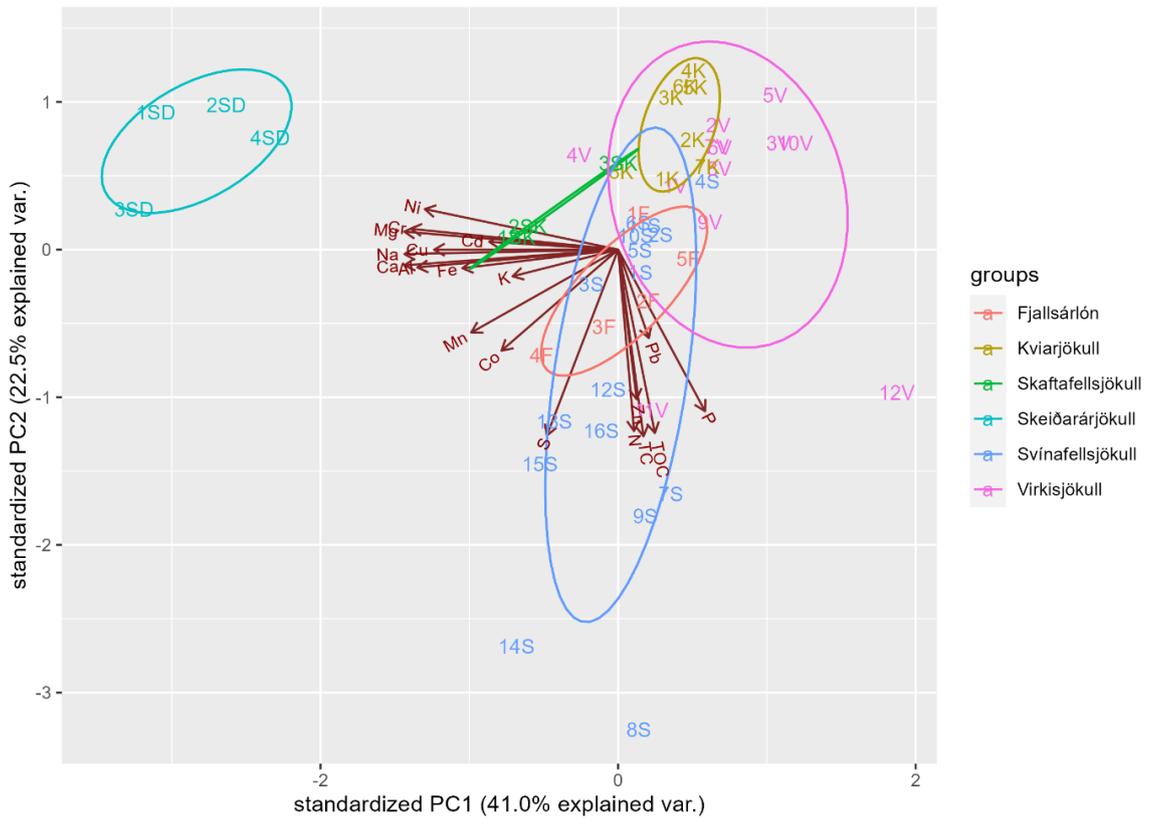


Figure 5.12. PCA ordination plot illustrating the discrimination between samples investigated for microbial community structure from the Vatnajökull ice cap, Iceland according to chemical properties by the downstream transect. The input data was standardised to have zero mean and variance one. The ellipses represent the core area added by confidence interval of 68%. Sample codes are given in Table 5.4.

Table 5.4. Codes of selected soil samples from Vatnajökull ice cap, Iceland shown in Figure 5.12 and Figure 5.13. S stands for Svínafellsjökull, V – Virkisjökull, K – Kviarjökull, F – Fjallsjökull, SK – Skaftafellsjökull, SD – Skeiðarárjökull.

Code	Sample	Code	Sample	Code	Sample
1S	SV-R-2109_1	1V	VK-YM-0206_1	5K	KV-M2-2409_1
2S	SV-R-2109_3	2V	VK-YM-0206_2	6K	KV-M2-2409_2
3S	SV-LM-2905_1	3V	VK-YM-0206_3	7K	KV-M3-2409_1
4S	SV-LM-2905_3	4V	VK-YM-1909_2	8K	KV-M3-2409_2
5S	SV-LM-2109_1	5V	VK-M2-1909_2	1F	FJ-LM-2309_1
6S	SV-LM-2109_2	6V	VK-M2-1909_3	2F	FJ-LM-2309_2
7S	SV-LIAM-3005_1	7V	VK-OM-0206_1	3F	FJ-M2-2309_1
8S	SV-LIAM-3005_2	8V	VK-OM-0206_2	4F	FJ-M3-2309_1
9S	SV-LIAM-3005_3	9V	VK-OM-0206_3	5F	FJ-M3-2309_2
10S	SV-LIAM-2109_1	10V	VK-OM-1909_2	1SK	SK-TM-2209_2
11S	SV-LIAM-2109_3	11V	VK-RM-2709_2	2SK	SK-M2-2209_2
12S	SV-2.5K-3105_2	12V	VK-RM-2709_3	3SK	SK-M2-2209_3
13S	SV-2.5K-3105_3	1K	KV-R-2409_1	1SD	SD-LM-2609_1

14S	SV-2.5K-3105_4	2K	KV-R-2409_3	2SD	SD-LM-2609_2
15S	SV-2.5K-2109_2	3K	KV-LM-2409_1	3SD	SD-M2-2609_1
16S	SV-2.5K-2109_4	4K	KV-LM-2409_2	4SD	SD-M2-2609_2

The PCA plot shows that samples collected from the Skeiðarárjökull (on the left side of the plot, Figure 5.12) have different chemical properties compared to samples from the other sites. Samples from Svínafellsjökull, Virkisjökull, Kviarjökull, Fjallsjökull, and Skaftafellsjökull are located at the centre of the plot closer to each other. Samples from Svínafellsjökull are stretched along PC2, indicative of influence by TC, TOC, and TN. Samples from other sites form tighter clusters, samples from Virkisjökull and Kviarjökull also being grouped along PC2, while samples from Fjallsjökull and Skaftafellsjökull are at an angle, having same influence by PC1 and PC2. Sample 12V (see Table 5.4 for the sample name) is to the right of the plot away from other samples from Virkisjökull, while samples 8S and 14S from Svínafellsjökull are at the bottom of the plot. These three samples have the highest TOC content (see Appendix 13). Moreover, PERMANOVA analysis showed a statistically significant difference between the subset of samples chosen for microbial analysis collected at the Vatnajökull ice cap by transect (p-value < 0.05).

### 5.2.3.3 Microbial community structure

#### 5.2.3.3.1 Clustering of microbial communities by glacier

Further, an NMDS ordination plot (Figure 5.13) did not demonstrate clear clustering of bacterial family-level distribution of samples by transect. Sample 1SK (SK-TM-2209\_2) from Skaftafellsjökull is to the far right (Figure 5.13a). This sample had low TOC content (0.03%). Sample 9V (VK-OM-0206\_3) from Virkisjökull is positioned in the top half of the plot compared to the other samples (Figure 5.13b). On the contrary, PERMANOVA analysis showed that there was a statistically significant difference between family-level bacteria in samples by transect (p-value < 0.05). In addition, when analysed against each other, family-level bacteria in samples from Svínafellsjökull were significantly different from that in samples from other sites (PERMANOVA analysis, p-value < 0.05).

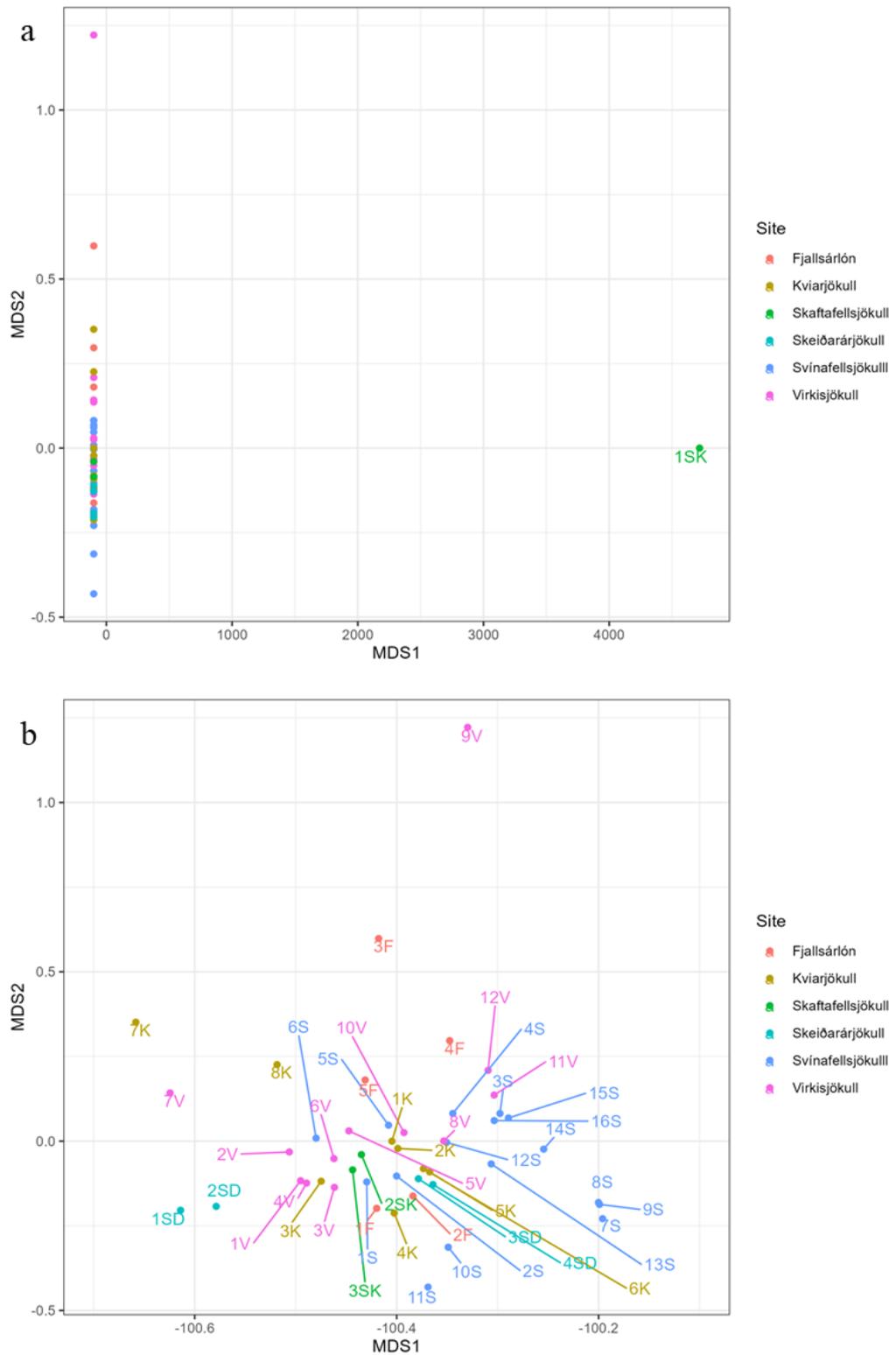


Figure 5.13. (a) Full scale and (b) zoomed-in two-dimensional NMDS plot of family-level bacterial communities in samples from the Vatnajökull ice cap, Iceland by transect. When the data values were larger than common abundance class scales, Wisconsin double standardisation was performed; when values looked very large, sqrt transformation was also performed; and distance matrix based on Bray–Curtis dissimilarity was obtained. Sample codes are given in Table 5.4.

### 5.2.3.3.2 Alpha diversity

Shannon–Weiner diversity index and Chao1 species richness estimate values were plotted to evaluate the microbial alpha diversity in each sample from Iceland analysed for microbial communities (Figure 5.14). Overall, samples from Kviarjökull had highest alpha diversity, followed by samples from Virkisjökull, Fjallsjökull, and Svínafellsjökull. Samples from Skaftafellsjökull and Skeiðarárjökull had lower richness and diversity compared to other sites.

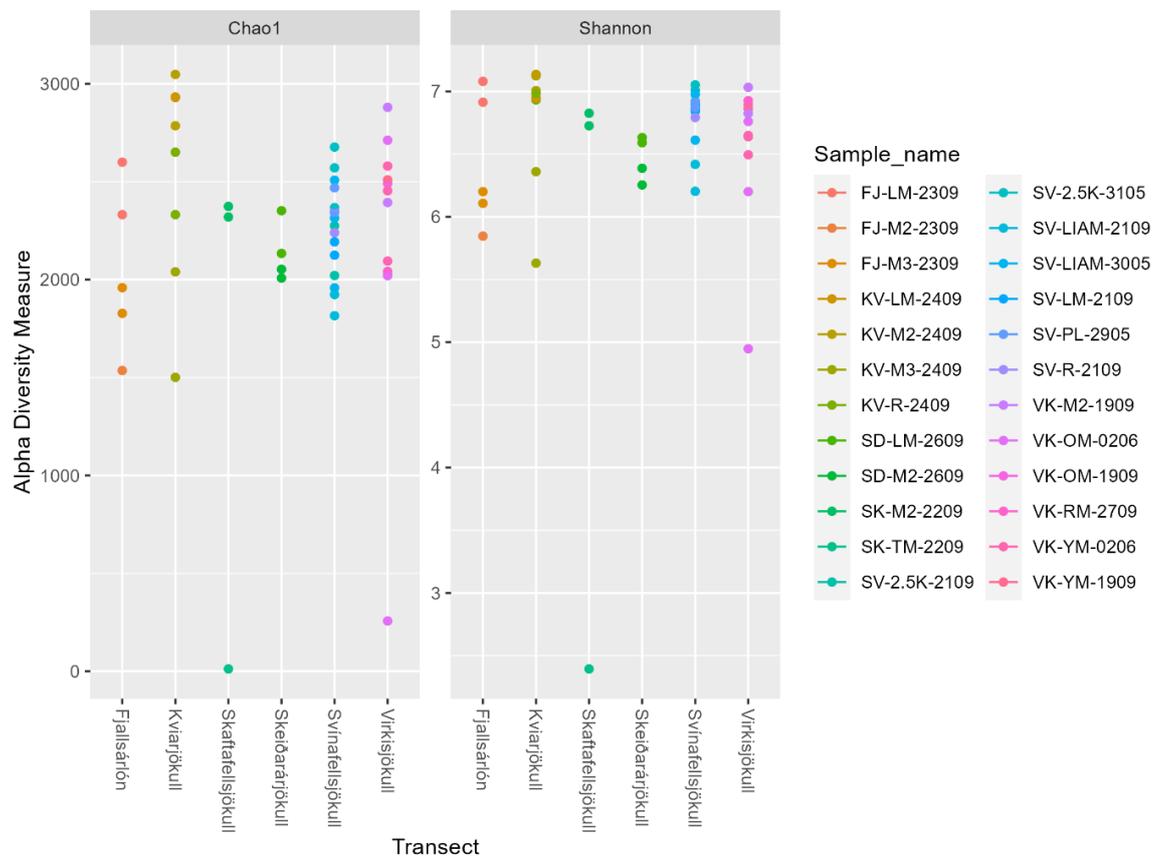


Figure 5.14. Alpha diversity (Shannon) and richness (Chao1) variation in soil samples from Fjallsjökull, Kviarjökull, Skaftafellsjökull, Skeiðarárjökull, Svínafellsjökull and Virkisjökull transects in Iceland.

No trend in alpha diversity with the age of samples was observed in any glacier forelands from Iceland (Figure 5.15). At Svínafellsjökull, sample from the oldest moraine (SV-2.5K-3105\_2) had the highest diversity, while a sample from a younger LIA moraine (SV-LIAM-2109\_1) had the lowest diversity (Figure 5.15a). At Virkisjökull, microbial diversity did not increase with the age of samples – sample from the oldest moraine at this glacier, VK-OM-0206\_3, had the lowest microbial diversity (Figure 5.15b). Similarly, soil samples collected

from older moraines from Kviarjökull (KV-M3-2409\_1) and Fjallsjökull (FJ-M2-2309\_1) had lower richness and diversity compared to some other younger samples (Figure 5.15c and Figure 5.15d). A sample from a younger moraine at Skaftafellsjökull (SK-TM-2209\_2) indicated the lowest diversity compared to other samples from this and other glaciers (Figure 5.15d). Alpha diversity measurements were stable in samples from Skeiðarárjökull (Figure 5.15d).

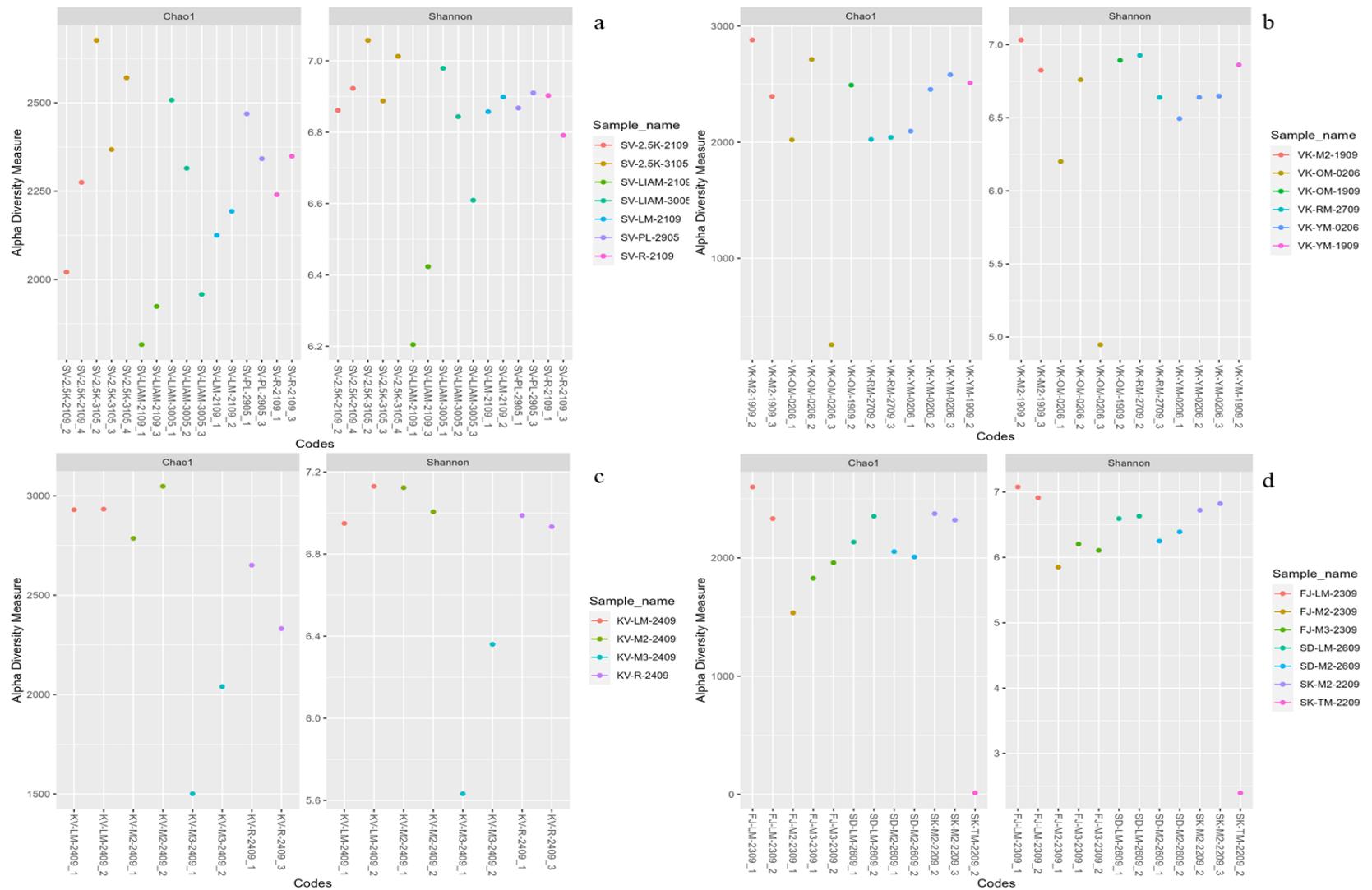


Figure 5.15. Alpha diversity (Shannon) and richness (Chao1) variation in soil samples from (a) Svínafellsjökull, (b) Virkisjökull, (c) Kviarjökull, and (d) Fjallsjökull, Skaftafellsjökull, and Skeiðarárjökull glaciers in Iceland.

### 5.2.3.3.3 Phylum-level community composition

Figures 5.16a and 5.17a show distribution of bacterial 16S rRNA gene derived phyla along downstream transects at the Vatnajökull ice cap in Iceland. Across all samples from six glacier forelands, the dominant phyla were Acidobacteria, Actinobacteria, Chloroflexi, Planctomycetes, Proteobacteria and Verrucomicrobia. The mean relative abundance of these bacteria in samples from each glacier are presented in Table 5.5, which shows that despite having common dominant phyla, concentration of these bacteria was different at each glacier. Bacteroidetes (37.5%) and Nitrospirae (17.5%) were abundant in sample SK-TM-2209\_2 (Skaftafellsjökull), which had low DNA concentration (BDL, see Appendix 18). Moreover, PERMANOVA analysis showed significant difference ( $p$ -value < 0.05) between samples by glacier.

Table 5.5. Mean relative abundance (%) of the dominant phyla in samples from six glaciers at the Vatnajökull ice cap.

	Svínafellsjökull	Virkisjökull	Kviarjökull	Fjallsjökull	Skaftafellsjökull	Skeiðarárjökull
<b>Acidobacteria</b>	21.2±4.7	22.0±5.9	24.4±3.4	24.5±5.6	14.4±12.4	23.8±10.6
<b>Actinobacteria</b>	14.0±5.4	9.2±2.7	7.5±4.0	6.5±1.2	6.4±5.6	14.5±9.0
<b>Chloroflexi</b>	15.2±3.9	17.5±4.1	20.3±3.0	19.6±5.4	21.9±1.8	14.6±5.5
<b>Planctomycetes</b>	10.7±2.7	11.5±2.2	11.6±1.4	11.5±2.3	7.4±6.5	7.8±0.5
<b>Proteobacteria</b>	16.9±4.1	12.9±3.0	10.9±2.7	12.4±3.3	7.7±6.9	10.5±0.8
<b>Verrucomicrobia</b>	10.4±3.0	12.7±2.7	11.4±4.0	9.3±1.3	8.3±8.0	12.1±1.3

### 5.2.3.3.4 Family-level community composition

Figure 5.16b and Figure 5.17b show bacterial family-level distribution along the downstream transects of glacier forelands in Iceland. The “other” category includes family-level bacteria that had less than 0.5% relative abundance in all samples collected in Iceland. Relative abundance of the most abundant family-level bacteria in samples from the Vatnajökull ice cap are presented in Table 5.6. Chthoniobacteraceae, unclassified at family-level RB41, unclassified at family-level iii1-15, unclassified at family-level Ellin6529 and Gemmataceae were the most abundant bacteria in samples from Svínafellsjökull, Virkisjökull, Kviarjökull, Fjallsjökull, and Skeiðarárjökull. Meanwhile, S24-7 (Bacteroidetes)

and unclassified at family-level Nitrospirales (Nitrospirae) were the most abundant bacteria detected in sample SK-TM-2209\_2 from Skaftafellsjökull. The different bacterial distribution in sample SK-TM-2209\_2 was also observed when plotting NMDS data (Figure 5.13a).

Table 5.6. Mean relative abundance (%) of the dominant family-level bacteria in samples from six glaciers at the Vatnajökull ice cap.

	Svinafellsjökull	Virkisjökull	Kviarjökull	Fjallsjökull	Skaftafellsjökull	Skeiðarárjökull
<b>Unclassified iii1-15 (Acidobacteria)</b>	6.4±1.5	5.7±2.6	5.8±2.3	4.8±3.8	3.2±3.0	5.5±3.2
<b>Unclassified RB41 (Acidobacteria)</b>	6.0±4.4	7.7±5.6	9.1±3.4	10.0±4.8	5.7±5.2	9.8±4.9
<b>S24-7 (Bacteroidetes)</b>	0.0±0.0	0.0±0.0	0.0±0.0	0.0±0.0	10.8±18.8	0.0±0.0
<b>Unclassified Ellin6529 (Chloroflexi)</b>	6.4±2.7	4.1±1.7	4.5±1.8	2.6±1.4	9.7±4.6	4.2±1.9
<b>Unclassified Nitrospirales (Nitrospirae)</b>	0.0±0.0	0.0±0.0	0.0±0.0	0.0±0.0	5.8±10.1	0.0±0.0
<b>Gemmataceae (Planctomycetes)</b>	3.4±1.0	4.4±1.0	5.3±1.4	4.2±1.9	3.4±3.0	2.9±0.4
<b>Chthoniobacteraceae (Verrucomicrobia)</b>	7.3±2.6	10.2±3.4	9.5±4.6	7.4±1.7	7.6±7.5	10.6±1.4

Replicates of samples from the Vatnajökull ice cap generally had similar phyla (Figure 5.16a and Figure 5.17a) and family-level bacterial compositions (Figure 5.16b and Figure 5.17b), which was expected based on their close proximity to each other on the NMDS plot (Figure 5.13b). As expected, sample SK-TM-2209\_2 had different microbial composition (Figure 5.17). Moreover, sample VK-OM-0206\_1 also seems to have a slightly different microbial community composition (Figure 5.16) compared to other replicates.

5 Soil development at Vatnajökull ice cap, Iceland

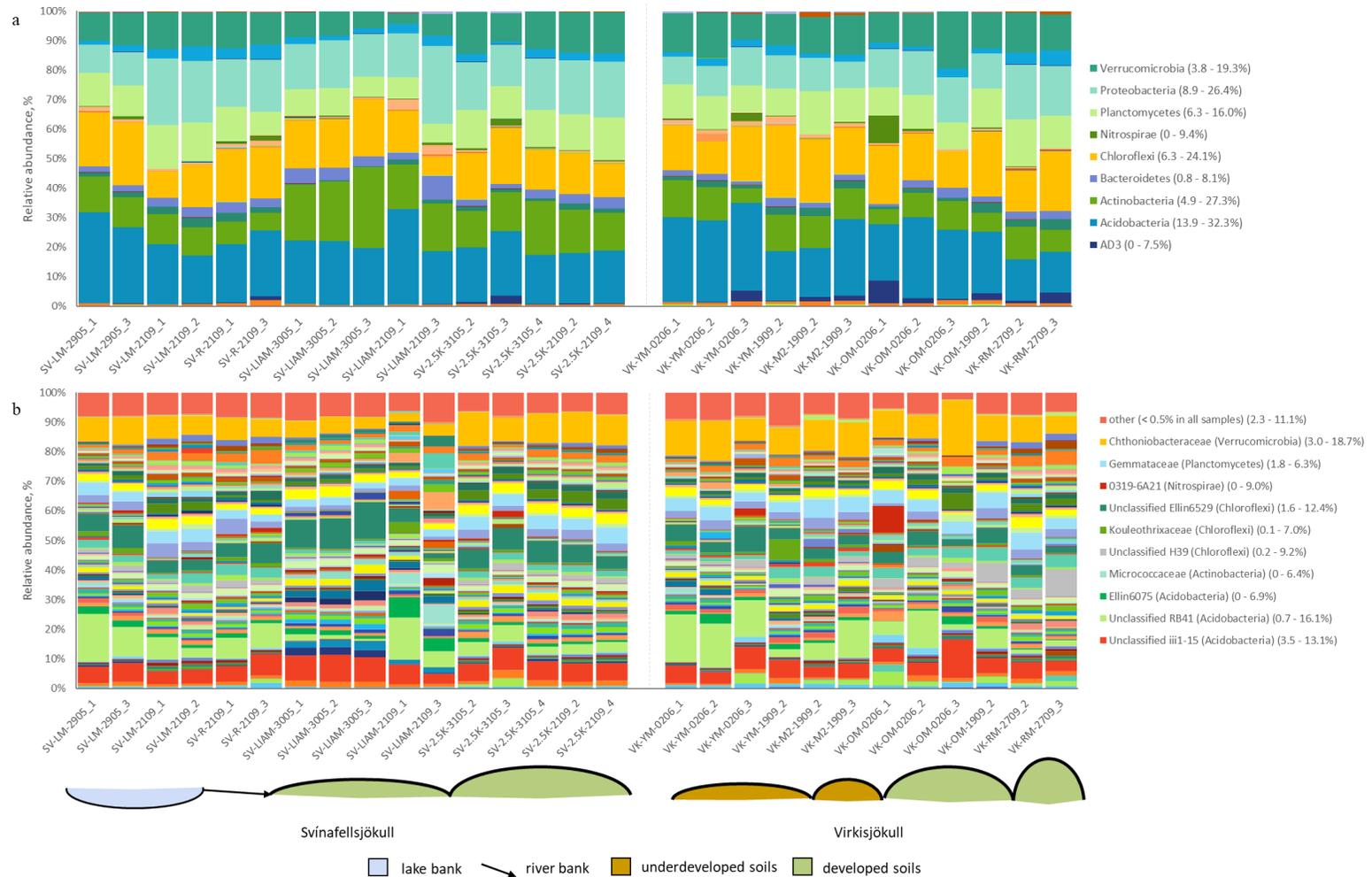


Figure 5.16. Relative abundance of bacterial taxa: (a) all assigned taxa at phylum level where possible and (b) all assigned taxa shown to the highest resolution possible down to the family level where possible (phylum level given in parentheses) for each sample along the Svínafellsjökull and Virkisjökull transects in Iceland. Selected taxa of high abundance given in parentheses. Charts at the bottom of the graph show downstream (left to right) transect profiles.

5 Soil development at Vatnajökull ice cap, Iceland

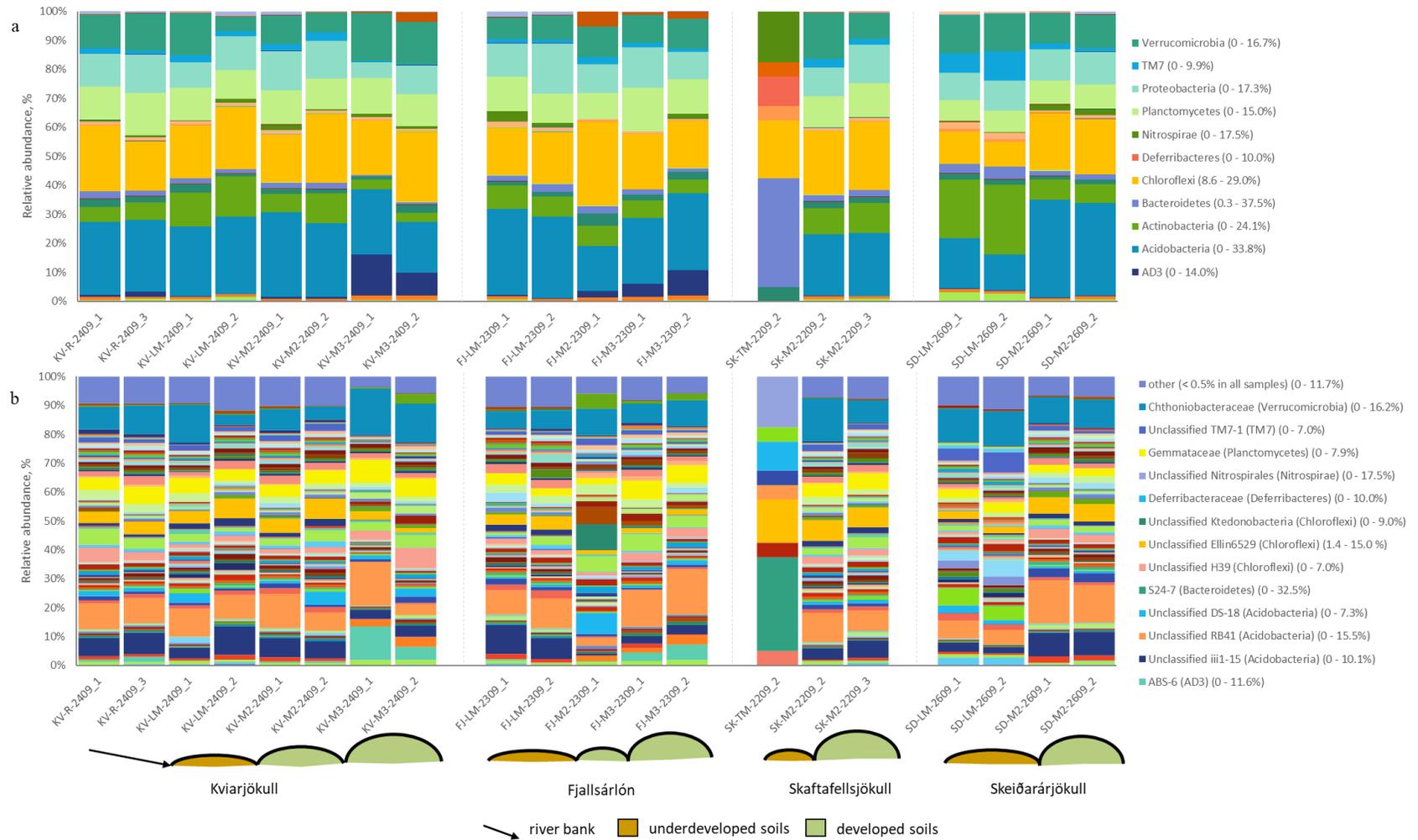


Figure 5.17. Relative abundance of bacterial taxa: (a) all assigned taxa at phylum level where possible and (b) all assigned taxa shown to the highest resolution possible down to the family level where possible (phylum level given in parentheses) for each sample along the Kviarjökull, Fjallsjökull, Skaftafellsjökull and Skeiðarárjökull transects in Iceland. Selected taxa of high abundance given in parentheses. Charts at the bottom of the graph show downstream (left to right) transect profiles.

## 5.3 Discussion

The aim of this chapter was to understand the distribution of OC, N, metals, organic biomarkers, and microbial community along downstream transects of glaciers from the Vatnajökull ice cap. The Vatnajökull ice cap is an older catchment with some samples representing recent deglaciation, while some were as old as 5000-6000-years-old. Within the ice cap six glaciers were studied, soil samples from four glaciers, Svínafellsjökull, Virkisjökull, Kviarjökull, and Fjallsjökull, were further investigated for organic biomarkers, while soil samples from all six glaciers were studied for microbial community structure. Generally, it was expected that older soil samples would have higher concentration of TOC, organic biomarkers and higher bacterial diversity compared to younger soils sampled closer to the glacier front. Moreover, it was hypothesised that TOC and organic biomarker concentrations would increase along the downstream transects, while microbial community diversity would be lower in recently deglaciated soils but would stabilize in samples from LIA moraines and older soils.

### 5.3.1 TOC, TN, and C/N in Iceland compared to other catchments

TOC analysis of samples collected at the Vatnajökull ice cap had a large range varying from less than 0.01% up to  $5.24 \pm 2.68\%$ . These values were similar to some other sites. For instance, Wietrzyk et al. (2018) reported TOC values ranging from 0.69% to 3.81% in samples from a glacier foreland in Svalbard, which has been ice free for 0-96 years. Meanwhile, in Switzerland, these values were slightly higher, ranging between 0.15% and 7.97%, in samples from a glacier foreland that has been retreating for 15-700 years (Prietz et al., 2013). TOC values recorded in Iceland were much higher than values obtained from the samples collected at the Tarfala valley, Sweden ( $0-1.08 \pm 0.09\%$ , this study). These findings show that the catchment studied in Iceland accumulated more OC compared to the bare catchment in Sweden and is comparable to similar sites in Svalbard and Switzerland. Some samples in Iceland were collected from moraines older than the ones in Svalbard and Switzerland, suggesting that geology of the terrain rather than the age of exposure plays an important role in the accumulation of soil derived OC. Prietz et al.

(2013) also suggested that soil-forming moraine material, i.e. geology, affected OC concentrations.

Generally, soils from Iceland had much higher TOC concentrations ( $0.96\pm 1.62\%$ ) compared to sediment samples ( $0.04\pm 0.10\%$ ). Similarly, samples from Sweden also had higher TOC content in soils than sediments, as well as soil samples (0.4-7.6%) from the Mt. Everest region compared to sediments samples (0.2-1.6%) (Guzella et al., 2016). As expected soils were more organic rich compared to sediments, most likely due to the development of the ecosystem, such as plant growth, in soils. Moreover, the highest TOC concentration was observed in soil samples collected from Svínafellsjökull ( $1.76\pm 2.05\%$ ), while soil samples from Skeiðarárjökull had the lowest TOC concentration ( $0.12\pm 0.10\%$ ). TOC concentrations of soil samples collected from Skeiðarárjökull were comparable to those of sediment samples from the other glacier systems studied at the Vatnajökull ice cap. This suggests that soils from the glacier foreland of Skeiðarárjökull are more mineral in nature.

Across all sites, there was a general trend of OC build-up with the age of moraines. Similarly, Wietrzyk et al. (2018) found that TOC concentration increased from 0.69% in recently deglaciated soils to 3.81% in more developed older soils. This observation suggests that OC tends to accumulate over time along glacier forelands in Iceland. The only exception was an initial upward trend followed by lower TOC content in the older moraine at Svínafellsjökull (SV-2.5K-3105/2109). Samples collected from the LIA moraine had a big difference between years 2018 and 2019 (Figure 5.18). This could be explained by the sampling location, which was vegetated by the invasive species, Nootka lupine (*Lupinus nootkatensis*) in 2018 (Figure 5.18a and b). In 2019, it was difficult to access the area and the sample was collected from more bare landscape closer to the glacier (Figure 5.18c and d). Perhaps, the high TOC content is due to the build-up of soil derived OM caused by plant succession. For instance, similarly, Dümig et al. (2011) observed accumulation of organic matter due to plant growth in a glacier foreland in Switzerland. This suggests that the moraine is still mostly made up of sediment with a topsoil accumulating enough OM to show high TOC signals due to the plant material.

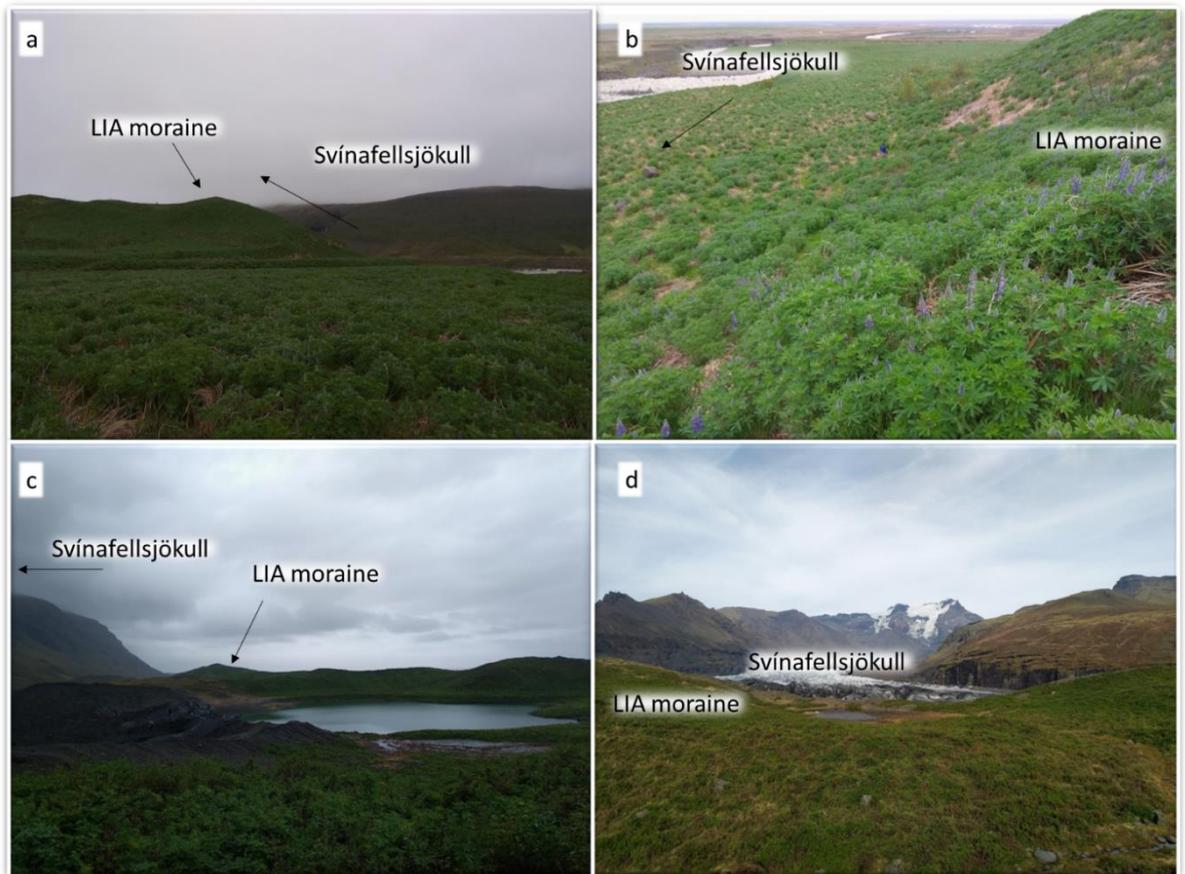


Figure 5.18. LIA moraine at Svínafellsjökull in 2018 (a and b) and in 2019 (c and d).

TN values also varied in samples from Iceland (0-0.66%) and were similar to findings in samples from glacier forelands in Svalbard (0.04-0.15%, Wietrzyk et al., 2018) and Switzerland (0.02-0.6%, Prietzel et al., 2013). Generally, samples with high TOC also had higher TN readings. The relationship between TOC and TN at Svínafellsjökull, Virkisjökull and Fjallsjökull was strong, suggesting that nitrogen at this transects is organic in nature (Stein and Rack, 1995 in Huon et al., 2002) and can be transformed to ammonia ( $\text{NH}_3$ ) and ammonium ( $\text{NH}_4$ ) to become more available to be utilized by plants (Brust, 2019). This is not surprising, as glacier forelands at these three sites contained very old moraines, suggesting that they have organic rich soils. Surprisingly, this relationship was also strong at Skaftafellsjökull, while TOC and TN correlation was weak at Kviarjökull, which had a moraine dating back to 3200 BP (Guðmundsson, 1997; Bennet et al., 2010). This discrepancy shows that other readings, such as C/N ratio and organic biomarkers, should be considered when accessing the build-up of OM at the study site. Relationship between

TOC and TN at Skeiðarárjökull was weak, most likely due to younger age of samples, as well as low TOC content, suggesting mineral nature of samples.

The mean C/N ratios across six sites varied from  $3.78 \pm 5.08$  to  $8.11 \pm 7.54$  (Table 5.7). Compared to other studies, C/N ratios of soil samples were comparable to values reported in Svalbard (C/N = 17-25, Wietrzyk et al., 2018) and Switzerland (C/N = 8-22, Prietzel et al., 2013). Similarly, organic soil in glacier forelands in Svalbard had the ratio of 23, while C/N ratio in mineral soils was either also 23 or as high as 123 (Yoshitake et al., 2007). In contrast, the highest C/N ratio in Iceland was observed at the most developed moraine at Virkisjökull ( $39.44 \pm 7.88$  at VK-RM-2709). According to Yu et al. (2010), C/N ratio is lowest in aquatic sediment samples (from  $7.5 \pm 1.1$  in marine environments to  $12.7 \pm 2.3$  in freshwater environments), higher in soils (from  $8.9 \pm 1.1$  to  $17.9 \pm 3.6$ ) and plants (from  $22.7 \pm 11.6$  to  $24.6 \pm 9.4$ ). Therefore, based on C/N ratios (Table 5.7) it can be suggested that soil samples collected from Svínafellsjökull, Virkisjökull and Fjallsjökull are descriptive of soil environment, while samples from other three sites are more representative of aquatic sediment samples. This is in line with the previous observations of TOC content and relationship between TOC and TN, suggesting that soil samples collected from Svínafellsjökull, Virkisjökull and Fjallsjökull are more developed compared to samples from Kviarjökull, Skaftafellsjökull and Skeiðarárjökull.

Table 5.7. Mean and standard deviation (SD) of C/N ratios of all samples, sediment and soil samples collected at each glacier at the Vatnajökull ice cap.

Glacier	Sediments		Soils		All	
	Mean	SD	Mean	SD	Mean	SD
Svínafellsjökull	1.4	2.58	12.15	6.56	7.29	7.44
Virkisjökull	1.08	1.65	11.99	13.62	5.79	10.44
Kviarjökull	1.11	0.46	6.78	4.76	4.89	4.72
Fjallsjökull	1.53	1.55	12.5	6.61	8.11	7.54
Skaftafellsjökull	0.83	0.26	6.73	5.98	3.78	5.08
Skeiðarárjökull	-	-	5.94	5.69	5.94	5.69

### 5.3.2 Some variation in metal concentration

Principal component analysis on chemical composition of samples from the Vatnajökull ice cap, as well as PERMANOVA indicated that there was not much difference between the samples from different glaciers (Figure 5.1b). Samples from Kviarjökull, Fjallsjökull,

Skaftafellsjökull, Svínafellsjökull, and Virkisjökull were clustered close to each other, suggesting that they had little difference in chemistry by site. On the other hand, samples from Skeiðarárjökull were located away from other samples in the top right corner of the plot, suggesting its difference compared to other sampling sites at the ice cap. This was expected as soil samples from Skeiðarárjökull had lower TOC and higher metal concentrations compared to samples from other glacier forelands in Iceland. This glacier frequently experiences jökulhlaups, which deposit sediments, 1996 jökulhlaup being one of the high-magnitude ones (Robinson et al., 2008). Further, PCA plots and PERMANOVA showed that there was more variation in chemistry by sample type. Similarly, proglacial soil and sediment samples in the Italian Alps were different (Colombo et al., 2020). This suggests that soils and sediments have different chemical properties. In this study, soils rather than sediments were investigated to observe accumulation of soil derived OC along transects of glacier forelands from the Vatnajökull ice cap.

Analysis of metal concentrations indicated that, similar to samples from Sweden, Fe ( $9423 \pm 2209$  mg/kg), Al ( $3419 \pm 1158$  mg/kg), Ca ( $3120 \pm 1290$  mg/kg) and Mg ( $1609 \pm 683$  mg/kg) were the most abundant metals in soil and sediment samples from the Vatnajökull ice cap. In line with this finding, Toubes-Rodrigo (2017) also reported that Fe, Al, Ca and Mg were the main metals in sediments trapped in basal ice collected from Svínafellsjökull. Fe, Al, Ca and Mg also were the most abundant metals in soils from a glacier foreland at the Tibetan Plateau (Bing et al., 2016). Moreover, Fe, Al, and Ca were dominant metals in samples from a glacier foreland in Svalbard (Venkatachalam et al., 2021). Overall, concentrations of metals recorded in samples from Iceland were slightly higher than that at the Tarfala valley in Sweden but were considerably lower than elsewhere. For instance, according to Bing et al. (2016), concentrations of Al ( $15400 \pm 4800$  -  $50400 \pm 7600$  mg/kg), Ca ( $19100 \pm 3600$  -  $33000 \pm 2200$  mg/kg) and Mg ( $4000 \pm 600$  -  $14600 \pm 3100$  mg/kg) were much higher, while concentration of Fe in Iceland was at the lower end of those recorded at the Tibetan plateau ( $8800 \pm 2400$  -  $28400 \pm 6300$  mg/kg). Similarly, concentrations of Fe ( $25547$ - $53774$  mg/kg) and Al ( $12561$ - $39008$  mg/kg) were much higher in glacier forelands in Svalbard, while Ca ( $537$ - $2353$  mg/kg) (Venkatachalam et al., 2021) had lower values than that recorded in Iceland. Comparison with other glacier forelands shows that sediments and soils at the Vatnajökull ice cap are low in metals, which suggests that TOC is more important

factor in the development of the catchment. Overall, metal concentrations were much higher in samples collected from Skeiðarárjökull compared to other glacier forelands from this study. This suggests that even though the glaciers are from the same ice cap they have different chemical properties, potentially affecting bacterial diversity (Venkatachalam et al., 2021).

Moreover, similar to TOC, metal concentration increased downstream along the Svínafellsjökull (Figure 5.5a), Kviarjökull and Fjallsjökull (Figure 5.5b) transects. On the contrary, at the Tibetan plateau, there was a downward trend in metal concentrations in older soils along the glacier foreland transects with samples collected from sites that were deglaciated in 2012-1890 (Bing et al., 2016). Meanwhile, Venkatachalam et al. (2021) reported no trend in distribution of metals along the transect that has been deglaciated for up to 1900 years, which is similar to what was observed in Sweden and at Virkisjökull and Skaftafellsjökull. This discrepancy between different sites suggests that geology of the catchment rather than time of exposure affects the distribution of metals along the transects and since metals shape the microbial community diversity of glacier forelands (Venkatachalam et al., 2021), it is expected to see some difference in microbial community structure and diversity of different glaciers as well.

### 5.3.3 Organic biomarkers

As TOC affects composition of organic biomarkers (Höfle et al., 2015), similar to findings in Sweden, it was expected that concentrations of BHPs and GDGTs would be highly influenced by TOC content. Moreover, linear regression analysis showed that there was a strong positive relationship between TOC and organic biomarkers (Figure 5.11).

Relative concentration of total BHPs recorded in Iceland ( $2469 \pm 1404$   $\mu\text{g/g}$  TOC) was lower compared to that in Sweden ( $4301 \pm 3427$   $\mu\text{g/g}$  TOC), most likely due to the higher OC content as absolute concentration in Iceland was higher ( $25 \pm 23$   $\mu\text{g/g}$  sediment) compared to Sweden ( $12 \pm 15$   $\mu\text{g/g}$  sediment). Relative concentration in Sweden was much higher compared to the values recorded in other studies. For instance, relative concentration of total BHPs ranged from 72 to 489  $\mu\text{g/g}$  TOC in estuary and open bay sediment samples from Northern Sweden (Doğrul Selver et al., 2012), from 138 to 281  $\mu\text{g/g}$  TOC in river mouth sediments from Siberia (Doğrul Selver et al., 2015), from 53 to 1454  $\mu\text{g/g}$  TOC in soil

samples from Northern England (Cooke et al., 2008), from 7 to 660 µg/g TOC in sediment and soil samples from the Bayelva River catchment (Rethemeyer et al., 2010). Values recorded in this study were higher even compared to values recorded in Siberian permafrost soils, which ranged from 84 to 1111 µg/g TOC (Höfle et al., 2015). This could be due to the low OC content in Sweden compared to other studies. For instance, OC concentration was 0.01% up to 5.24±2.68% in Iceland, the values were 8-13.6% in river mouth samples from Siberia (Vonk et al., 2010 in Doğrul Selver et al., 2015), 1.7-10.7% in soil samples from Northern England (Cooke et al., 2008), 0.1-28.4% in sediment and soil samples from the Bayelva River catchment (Rethemeyer et al., 2010) and 1.5-28.4% in Siberian permafrost soils (Höfle et al., 2015) compared to 0.01 - 5.24±2.68% in Iceland. Moreover, absolute concentration of BHPs was 4-121 µg/g dry soil in soils from Northern England (Cooke et al., 2008) compared to 25±23 µg/g sediment in Iceland.

### **5.3.3.1 Soil specific organic biomarkers in Iceland compared to other catchments and their distribution**

#### **5.3.3.1.1 Soil marker BHPs**

Soil marker BHPs are abundant in soils (Cooke et al., 2008; Doğrul Selver et al., 2012; Höfle et al., 2015), which allows us to use them as indicators of accumulation of soil derived OC and soil development. BHP analysis of soil samples from the Vatnajökull ice cap revealed presence of BHPs and specifically soil marker BHPs in all samples. Compared to Sweden, where soil marker BHPs were present only in some samples with relatively high TOC content, glacier forelands in Iceland studied for organic biomarkers were older. Moreover, soil marker BHPs in Iceland (16-28 % of total BHPs at Svínafellsjökull; 8-18% – Virkisjökull; 7-13% – Kviarjökull and Fjallsjökull) were higher than that at Storglaciären (0-12%) but comparable to Tarfalajaure (8-21%) in Sweden, as well as some estuary sediment samples in the Arctic. For instance, fractional abundance of soil marker BHPs increased from 15% at the Mackenzie River to 37% at the Indigirka River systems (Cooke et al., 2009). However, the values were lower than that in soils in Northern England (Cooke et al., 2008), which ranged from 20% to 48% and from permafrost regions in the Arctic, which ranged from 20% to 73% at the Bayelva River catchment (Rethemeyer et al., 2010) and went up to 82% in Siberia (Doğrul Selver et al., 2015). These observations suggest that soil marker BHPs in

Iceland are comparable to that at Tarfalajaure, some estuary sediment samples in the Arctic, and some soils in Northern England, however, have much lower relative abundance than organic rich permafrost (total of 1672 Pg of OC in the northern circumpolar permafrost region, Tarnocai et al., 2009), indicating that OC plays an important role in presence of BHPs.

Since the  $R'_{\text{soil}}$  index is a function of soil marker BHPs, their fractional abundance is comparable to the  $R'_{\text{soil}}$  index across the four glaciers, which was 0.27-0.35 at Svínafellsjökull, 0.11-0.24 at Virkisjökull, 0.09-0.15 at Kviarjökull and 0.10-0.17 at Fjallsjökull. According to Zhu et al. (2011), the  $R_{\text{soil}}$  (similar to the  $R'_{\text{soil}}$  index but takes into account methylated soil marker BHPs as well) in soils has an average value of  $0.7 \pm 0.1$ . However, values identified in samples from Iceland were similar to the  $R'_{\text{soil}}$  identified in samples from Tarfala valley (0-0.34) in the present study and aquatic sediments in Northern Sweden in a different study (0.08-0.30, Doğrul Selver et al., 2012). As expected, compared to permafrost catchments across the Arctic, the  $R'_{\text{soil}}$  index was much lower in the study sites in Iceland. For instance, the  $R'_{\text{soil}}$  index was 0.57 in Kolyma River in Northeast Siberia (Doğrul Selver et al., 2015) and ranged from 0.16 to 0.62 in river sediment samples from Yenisei River in Siberia (De Jonge et al., 2016). Record of soil marker BHPs and the  $R'_{\text{soil}}$  index in soil samples from Iceland suggests that soils in this deglaciated catchment are still accumulating soil derived OC.

#### **5.3.3.1.2 BrGDGTs**

BrGDGTs, produced in soils and indicative of terrestrial OC (Hopmans et al., 2004; Weijers et al., 2006; Doğrul Selver et al., 2012), were present in all samples from the Vatnajökull ice cap, while one sample from Svínafellsjökull (SV-LIAM-3005) also had some crenarchaeol (1%). Relative concentration of total brGDGTs was highest in soil samples from Virkisjökull and Svínafellsjökull, having much lower values at Kviarjökull and Fjallsjökull (Appendix 16), which is comparable to how soil specific BHPs contributed to the total BHPs at each site, suggesting that the two biomarkers were correlated. The mean relative concentration of total GDGTs at the Vatnajökull ice cap ( $112 \pm 74 \mu\text{g/g TOC}$ ) was lower than that in Sweden ( $403 \pm 163 \mu\text{g/g TOC}$ ), while the mean absolute concentration was higher:  $1.52 \pm 2.14 \mu\text{g/g}$  sediment in Iceland compared to  $0.77 \pm 1.19 \mu\text{g/g}$  sediment in Sweden. Relative

concentration of GDGTs in Iceland was higher compared to other sites in the Arctic. For instance, relative concentration of total GDGTs was 0.26-0.91  $\mu\text{g/g}$  TOC in Northern Sweden (Doğrul Selver et al., 2012), 3.2-4.5  $\mu\text{g/g}$  TOC at the Kolyma River (Doğrul Selver et al., 2015) and 0.1-35  $\mu\text{g/g}$  POC at the Yenisei River (De Jonge et al., 2015). This discrepancy is most likely due to the relatively low TOC concentration in samples from Iceland (0.01 - 5.24 $\pm$ 2.68%) compared to other sites (8-13.6% in river mouth samples from Siberia, Vonk et al., 2010 in Doğrul Selver et al., 2015).

Concentrations of brGDGTs can be used as a proxy to study past temperatures (Weijers et al., 2007) and soil pH (Peterse et al., 2012). GDGT proxy values in this chapter were compared to the existing literature. GDGT proxy pH ranged from 6.1 to 7.3, while MAT ranged from 1.5 to 8.7  $^{\circ}\text{C}$  (Figure 5.10). Both proxies followed a downward trend along the transects, however, since the transects were relatively short, the trend is probably an indication that there was not enough GDGTs to measure MAT accurately. In literature, soil  $\text{pH}_{\text{H}_2\text{O}}$  of topsoil ranged from 5.7 $\pm$ 0.2 to 7.4 $\pm$ 0.2 along the downstream transect at Skaftafellsjökull, with younger moraines having alkaline environment, while older moraines were acidic (Vilmundardóttir, 2014). It seems that, similar to what has been observed in Sweden, GDGT proxy pH is in sync with what is reported in literature, suggesting that this proxy is reliable. Meanwhile, Vilmundardóttir et al. (2014) reported mean annual temperature of 5.1  $^{\circ}\text{C}$  at Skaftafellsjökull, while MacDonald et al. (2016) reported mean annual temperature of 5.5  $^{\circ}\text{C}$  at Virkisjökull. It seems GDGT proxy MAT is similar to what has been reported in previous literature, though quite large fluctuation suggests that it should be considered with care, in accordance with what has been observed in Sweden.

### **5.3.3.2 Soil development in Iceland**

As discussed earlier, there is a clear pattern of accumulation of soil derived OM based on TOC concentrations, with soils from more established moraines having higher TOC values compared to that from younger moraines. Based on this observation and similar findings in another study by Wietrzyk et al. (2018), it was concluded that OC accumulates over time. Therefore, it was expected to see similar patterns in the distribution of organic biomarkers along the transects. However, there was no clear trend of relative concentration of total BHPs or soil marker BHPs build-up with the age of moraines in Iceland. The  $R'_{\text{soil}}$  index used

to observe changes in the distribution of soil marker BHPs along transects (Doğrul Selver et al., 2012) also did not follow an upward trend. Since previously soil marker BHPs and their proxies were only used to observe transport of terrestrial OC from land to sea (e.g., Bischoff et al., 2016; Cooke et al., 2008; Doğrul Selver et al., 2012; Doğrul Selver et al., 2015; Spencer-Jones et al., 2015; Zhu et al., 2011) and not accumulation of soil derived OC in deglaciated forelands, it was interesting to compare findings in Iceland to the ones observed in Sweden. Generally, in Iceland, the  $R'_{\text{soil}}$  increased initially followed by a subsequent decrease in the most established moraines. This might be due to the catchment in Iceland being older than the Tarfala valley in Sweden. This suggests that even though soils accumulate OM over time after being exposed, build-up of soil marker BHPs (1) plateaus out after reaching a certain stage and/or (2) other BHP compounds, such as BHT, methylated BHT, aminotriol, BHT glucosamine and BHT cyclitol ether, overtake and mask soil marker BHPs.

Similarly, relative concentration of total brGDGTs ( $\mu\text{g/g TOC}$ ) did not increase along the transect from younger to older moraines. Along soil profiles in France, Kim et al. (2011) observed that concentration of brGDGTs increased in deeper soil profiles, therefore it was expected that the amount of brGDGTs ( $\mu\text{g/g TOC}$ ) would also be higher in older soils, indicating accumulation of this organic biomarker. However, since no such or any other pattern was observed in glacier foreland transects in Iceland, this suggests that the age of samples does not play any role in the production and preservation of brGDGTs.

Moreover, there was no relationship between the absolute concentrations of BHPs and GDGTs, as well as no similar pattern of downstream distribution of these organic biomarkers. This is in line with findings in Kim et al. (2011) reporting that BHPs and brGDGTs have different trends along soil profiles, suggesting that organic biomarkers are produced by different organisms and therefore, behave differently. This shows that looking at both soil marker BHPs and brGDGTs could give the best representation of soil development in deglaciated soils, similar to findings in Doğrul Selver et al. (2012) and Doğrul Selver et al. (2015), which concluded that the use of proxies based on both biomarkers is important to trace terrestrial OC.

### 5.3.3.3 Source and nature of organic biomarkers

Soil marker BHPs and brGDGTs are used as markers of terrestrial OC signal. GDGTs were represented by brGDGTs that are potentially produced in soils by Acidobacteria (Weijers et al., 2009) (Table 4.4). Most of the samples from Iceland had no crenarchaeol, which has an aquatic origin (Zell et al., 2013). This was expected as only soil samples were analysed for organic biomarkers.

Similar to the catchment in Sweden, adenosylhopane ( $190\pm 94$   $\mu\text{g/g}$  TOC) and adenosylhopane type 3 ( $82\pm 72$   $\mu\text{g/g}$  TOC) were the most abundant soil specific BHPs in Iceland and made up 16-28% of total BHPs at Svínafellsjökull; 8-18% – Virkisjökull; 7-13% – Kviarjökull and Fjallsjökull. Adenosylhopane and related structures are produced by purple non-sulfur bacteria, *Nitrosomonas europaea*, and *Bradyrhizobium japonicum* (as cited in table 2.2a, Cooke, 2011) (Table 4.4).

The main BHP compounds in samples from Iceland were similar to that identified in Sweden. BHTs, being the most abundant BHP, contributed to 41-63% of total BHPs, followed by methylated BHT (9-21%), 35-aminotriol (1-8%), BHT glucosamine (1-7%) and BHT cyclitol ether (0.4-9%). BHT is a common compound and used to describe both aquatic (Doğrul Selver et al., 2012, and references therein) and terrestrial environments (Doğrul Selver et al., 2012, and references therein). In soil samples from Iceland, BHT most likely has a terrestrial origin as the samples were collected from glacier foreland. BHT, aminotriol and BHT cyclitol ether are produced by various organisms (see Table 4.4 for more details), Me-BHT is produced by Cyanobacteria, *Rhodopseudomonas palustris* and *Methylobacterium organophilum* and descriptive of different environments, while Methylophilic bacteria, Thermoacidophilic bacteria, and *Zymomonas mobilis* produce BHT glucosamine which is a common structure in sediments (as cited in table 2.2a, 2.2b, Cooke, 2011) but was also present in soil samples from Iceland, providing new knowledge about the environment this BHP compound can be found in. Some bacteria, such as Cyanobacteria, *Methylobacterium organophilum*, Methylophilic bacteria, *Zymomonas mobilis*, produce several BHP compounds, suggesting that if detected, these organisms could be a potential source of different biomarkers.

### **5.3.4 Microbial community diversity in samples from the Vatnajökull ice cap**

In order to understand soil OC accumulation and development in glacier forelands, it is important to study not only what has been preserved in the form of organic biomarkers but also what organisms are participating in the production of these biomarkers. Therefore, microbial community structure of soils along the glacier foreland transects in Iceland was analysed.

#### **5.3.4.1 Chemical composition of samples, and their effect on microbial diversity**

Microbial analyses were carried out on some soil samples from the glacier forelands of all six glaciers at the Vatnajökull ice cap. Generally, similar to Sweden sediment samples had very little DNA compared to soils and were not amplified with PCR; hence the microbial analysis was carried out only on soil samples.

In order to understand the differences between the transects, and because microbial community diversity is highly affected by metals (Venkatachalam et al., 2021), PCA was performed on chemical composition of samples chosen for microbial community studies (Figure 5.12). A PCA plot showed that samples collected from the Skeiðarárjökull had different chemical composition compared to samples from the other sites, which formed clusters closer to each other. It seems that samples from Svínafellsjökull are mostly influenced by TC, TOC, and TN, while samples from Virkisjökull, Kviarjökull, Fjallsjökull and Skaftafellsjökull are influenced by OM content, as well as metal concentrations. This pattern of clustering was expected as soil samples from Skeiðarárjökull were much more mineral in nature compared to soils from other glaciers. This suggests that soil samples collected from Skeiðarárjökull could be inhabited by different microorganisms compared to other five glacier forelands.

#### **5.3.4.2 Bacterial diversity**

The alpha diversity measurements showed some difference in microbial community composition between the glaciers. The alpha diversity measurements showed that samples

collected from the glacier forelands of Kviarjökull, Virkisjökull, Fjallsjökull, and Svínafellsjökull had higher richness and diversity compared to samples from Skaftafellsjökull and Skeiðarárjökull, which is most likely due to the low TOC concentration in samples from these two transects. Moreover, samples from Iceland had overall higher diversity than samples from Sweden. This could be because soils from Iceland are older and foster more established microbial communities, as well as having different bedrock geology. This suggests that OC might play an important role in the diversity of microbial community structure in glacier forelands. In line with this, Kaštovská et al. (2007) also observed that sediments and soils with low organic matter and low OC content from several glaciers in Svalbard had lower bacterial abundance and diversity. However, despite the expectations, bacterial diversity did not increase with the age of samples along the glacier foreland transects in samples from Iceland. Moreover, some samples from older moraines, such as SV-LIAM-2109\_1 from Svínafellsjökull, VK-OM-0206\_3 from Virkisjökull, KV-M3-2409\_1 from Kviarjökull and sample FJ-M2-2309\_1 from Fjallsjökull, had lower diversity compared to younger samples from these glaciers. This is contrary to the findings in other studies where time of exposure after deglaciation affects microbial diversity of soils. For instance, Mapelli et al. (2018) reported that bacterial communities increased along the chronosequence of a glacier foreland in Svalbard, while Venkatachalam et al. (2021) found that recently deglaciated samples had lower alpha diversity compared to more mature soils. As Schütte et al. (2009) suggested community composition stabilised in older soils, which could be true for even in younger soils from Iceland, as well as the fact that microbial diversity and their behaviour may vary by glacier foreland.

#### **5.3.4.3 Bacterial taxa**

Dominant phyla identified in samples from the Vatnajökull ice cap were Acidobacteria, Actinobacteria, Chloroflexi, Planctomycetes, Proteobacteria and Verrucomicrobia (Table 5.5, Figure 5.16a and Figure 5.17a). Generally, there was no major difference in phylum distribution between the glaciers, except for a sample from a poorly developed moraine at Skaftafellsjökull (SK-TM-2209\_2), which had high relative abundance of phyla Bacteroidetes and Nitrospirae. Overall, phyla identified in Iceland is comparable to the findings of other similar studies. For instance, Acidobacteria, Actinobacteria, Bacteroidetes, and Proteobacteria were the major phyla identified in soils collected across the Arctic

tundra (Chu et al., 2010). Similarly, Schütte et al. (2010) reported that Acidobacteria and Actinobacteria were the dominant phyla identified in samples from a glacier foreland in Spitsbergen, while in Svalbard, Actinobacteria and Proteobacteria made up the majority of bacterial community and Planctomycetes, Acidobacteria, and Bacteroidetes were also present in smaller quantities (Seok et al., 2016). Further, Acidobacteria, Actinobacteria, and Proteobacteria, as well as Chloroflexi and Verrucomicrobia were major phyla in soil samples from a glacier foreland in Svalbard (Venkatachalam et al., 2021). Nitrospirae was also present in small quantities in one of the soil samples from a glacier in the Himalayas (Shivaji et al., 2011). These observations suggest that soil samples from the Vatnajökull ice cap are generally populated by microbial communities similar to other cold catchments. Compared to the findings reported from Sweden, where candidate phylum AD3 was one the dominant phyla, samples in Iceland indicated low relative abundance of this phylum. On the other hand, one of the dominant phyla in Iceland, that was not abundant in samples from Sweden, was Verrucomicrobia. Interestingly, Bacteroidetes was one of the major phyla in samples with low TOC, both in Sweden and Iceland. Similarly, Venkatachalam et al. (2021) reported that Bacteroidetes was mainly identified in younger samples. Therefore, it seems that Bacteroidetes is a dominant phylum in low carbon, young Arctic soils, and its relative abundance drastically decreases in more developed carbon rich samples.

Similar to the findings in Sweden, no particular pattern was seen in the microbial community composition along the glacier foreland transects at the Vatnajökull ice cap. The only observation concerning the change in bacterial community along the transect was observed at Kviarjökull and Fjallsjökull. Candidate phylum AD3 had higher concentration in older soils from glacier forelands of these glaciers. This is in line with findings in Sweden and in Mateos-Rivera et al. (2016), which reported higher relative abundance of candidate phylum AD3 in mature soils (LIA) from the Styggedalsbreen glacier foreland in Norway. Otherwise, the lack of observable pattern of change in microbial community in Iceland is contrary to other studies, where microbial succession along the chronosequences was observed. For instance, in Spitsbergen, microbial succession was observed along the chronosequence of a glacier foreland (Schütte et al., 2009). Similarly, Venkatachalam et al. (2021) observed microbial succession in soils along a chronosequence in Svalbard. Schütte et al. (2010) suggested that bacterial communities stabilize in older soils (150-year-old soils

in Spitsbergen). It seems that in Iceland, microbial communities in soils adapted to the new environment and stabilised quickly even in more recent soils (since the LIA), in line with the previous observations of alpha diversity.

Further, in order to understand microbial community composition in soils from glacier forelands in Iceland each bacterial phylum and family was investigated in more detail. Acidobacteria was present in all samples from Iceland, except SK-TM-2209\_2. The relative abundance of this phylum is in line with studies stating that Acidobacteria was one of the dominant phyla in soil samples (Janssen, 2006) and findings from Sweden. Relative abundance of Acidobacteria was lower in older soils from Svínafellsjökull, Virkisjökull, Kviarjökull, and Fjallsjökull, suggesting that its relative abundance decreases with increase in carbon content. This observation is in agreement with other studies suggesting that the phylum is more abundant in low carbon environments and addition of organic matter decreases the relative abundance of Acidobacteria (Fierer et al., 2005; Delgado-Balbuena et al., 2016). On the other hand, a site with lower TOC content behaved similar to the samples from Sweden: the relative abundance decreased downstream in samples from Skeiðarárjökull. This suggests that in low carbon samples the microbial community, including Acidobacteria, is still being established.

According to Kielak et al. (2016), there is an ecological relationship between Acidobacteria and Proteobacteria. In agreement with that, similar to Acidobacteria, Proteobacteria was identified in all samples except SK-TM-2209\_2. Compared to other studies (Mateos-Rivera et al., 2016; Nemergut et al., 2007) or the findings in Sweden, there was no particular pattern in variation of Proteobacteria with the age of moraines, suggesting that the phylum homogenizes in more developed catchments. Sphingomonadaceae, Xanthomonadaceae and Bradyrhizobiaceae were the dominant family members of Proteobacteria. Sphingomonadaceae are usually chemoorganotrophic and can be found in different environments, including soils and cryoconites (Glaeser and Kämpfer, 2014). Moreover, species of this family, *Zymomonas mobilis*, was reported to produce BHT, BHT cyclitol ether and BHT glucosamine (Table 4.4), which were abundant BHP compounds in samples from Iceland. Bradyrhizobiaceae is a nitrogen-fixing bacterium found in different environments, including soils (Marcondes de Souza et al., 2014). *Bradyrhizobium japonicum* that produces soil marker BHPs and *Rhodopseudomonas palustris* that produces Me-BHT, and

*Rhodopseudomonas acidophila*, which produces BHT are species of this family-level bacteria (Table 4.4). This suggests that members of Proteobacteria were responsible for producing the abovementioned BHP compounds, allowing to link the current microbes with the biomarkers. Xanthomonadaceae was one of the dominant family-level bacteria from soils at a glacier foreland in East Antarctica (Yan et al., 2017) and cryoconites from King George Island, Antarctica (Zdanowski et al., 2017), suggesting that study sites in Iceland have similar microbial metabolisms as some other sites in polar regions.

Actinobacteria had high relative abundance in all samples from Iceland, but similar to the previously discussed Proteobacteria and Acidobacteria, was not detected in sample SK-TM-2209\_2. Most Actinobacteria are aerobic and can be either heterotrophic or chemoautotrophic (Barka et al., 2016). Relative abundance of Actinobacteria decreased along the downstream transects at the Virkisjökull, Kviarjökull, Fjallsjökull, and Skeiðarárjökull glacier forelands. This is in line with findings in Sweden, where relative abundance of Actinobacteria was higher in younger sediments and decreased in older soils. Different glaciers had different members of this phylum, with the only exception of family-level bacteria, Nocardioideae, which had similar relative abundance in samples from all six glaciers. Members of this family are chemoorganotrophs found in different environments, including soils (Pershina et al., 2018). On the other hand, Micrococcaceae was abundant in samples from Svínafellsjökull but not other glaciers. Members of Micrococcaceae were present in samples from other studies, including soil samples from South Shetland Islands, Antarctica (Lamilla et al., 2017). Orders Acidimicrobiales and Solirubrobacterales had high relative abundance in soils from Skeiðarárjökull but were also found in samples collected from western Antarctic Peninsula (Chong et al., 2012). This suggests that even though abundant only at some glaciers in Iceland, these bacteria can be found in other polar habitats as well.

Chloroflexi also had high relative abundance in all samples from the Vatnajökull ice cap. This phylum can be found in different environments, including cold habitats (Costello and Schmidt, 2006) such as Iceland. Moreover, it is thought that Chloroflexi can perform photosynthesis in soils (Ji et al., 2016). Relative abundance of Chloroflexi had some variance, decreasing along the transect at the Svínafellsjökull glacier foreland, and increasing along the Kviarjökull, Skaftafellsjökull, and Skeiðarárjökull transects. Mateos-

Rivera et al. (2016) reported that abundance of this phylum increased with increasing soil age along a glacier chronosequence in Norway. However, different patterns along different transects in Iceland suggest that this phylum behaves differently regardless of the soil age. Classified at class-level Ellin6529, H39, JG30-KF-CM45, oc28 and Ktedonobacteria, as well as family-level Kouleothrixaceae and Ktedonobacteraceae were main members of the phylum, however, literature indicates that members of Chloroflexi were poorly studied. Members of class Ktedonobacteria have been reported to inhabit different environments, including extreme and cold environments (Yabe et al., 2017), which is applicable to this study. Meanwhile, previously, members of family Kouleothrixaceae were detected in rhizosphere (Astorga-Eló et al., 2020; Lopez et al., 2017), and Ellin6529 fixes N<sub>2</sub>, which contributes to the enrichment of deglaciated soils in labile N (Lopes et al., 2015), suggesting that their presence in samples from Iceland could be due to the development of soil and establishment of vegetation.

Similar to Acidobacteria, Actinobacteria, and Proteobacteria, Planctomycetes were present in all samples from Iceland, except SK-TM-2209\_2. Planctomycetes were found in different environments, including freshwater, soil (Fuerst and Sagulenko, 2011) and polar and alpine environments (Costello and Schmidt, 2006; Ji et al., 2016; Mateos-Rivera et al., 2016). Gemmataceae, Pirellulaceae, and class-level WD2101 were the most abundant members of Planctomycetes. Gemmataceae is a strictly aerobic chemoorganotroph (Kulichevskaya et al., 2020), explaining its presence in soil samples. Meanwhile, order-level WD2101 was mostly detected in peats (Dedysh et al., 2021; Ivanova et al., 2016), which is not true for the study site in Iceland, but its presence could indicate presence of organic matter. Pirellulaceae had high abundance in water and ice samples from a lake in Subarctic Russia (Zakharova et al., 2021), suggesting that this bacterium can be found in other cold habitats as well.

According to Schlesner et al. (2006), Verrucomicrobia has a moderate degree relationship with Planctomycetes. Verrucomicrobia was detected in all samples except SK-TM-2209\_2. Similar to Planctomycetes, relative abundance of Verrucomicrobia did not follow a pattern along the glacier foreland transects. Verrucomicrobia is found in various aquatic and terrestrial habitats (Schlesner et al., 2006). For instance, it was one of the abundant phyla in a glacier foreland in Norway (Mateos-Rivera et al., 2016) and Svalbard (Venkatachalam

et al., 2021). On family level, Chthoniobacteraceae, which is a free-living aerobic and saccharolytic organism (Janssen and Hedlund, 2015), was the most abundant member of Verrucomicrobia. Similarly, Chthoniobacteraceae was also one of the dominant family-level bacteria from soils at a glacier foreland in East Antarctica (Yan et al., 2017). Therefore, presence of members of Verrucomicrobia in Icelandic samples highlights that this bacterium is likely common in soils in glacier forelands.

Bacteroidetes and Nitrospirae were detected in smaller quantities in all samples from Iceland, while their relative abundance in sample SK-TM-2209\_2 was as high as 37.5% and 17.5%, respectively. Bacteroidetes have been reported to grow in anoxic environments, fermenting inorganic material of subglacial origin (Sheik et al., 2015), which could explain its relative abundance in the sample collected from a poorly developed lake moraine, but it doesn't explain why it is not abundant in other samples with low TOC content. Most members of Nitrospirae are aerobic chemolithotrophs (Garrity and Holt, 2001), which again explains why it was detected in a sample with low TOC but doesn't explain why it wasn't abundant in other low carbon samples.

Generally, bacteria detected in Iceland are similar to that in other polar and alpine habitats, as well as soils in general (e.g., Chu et al., 2010; Costello and Schmidt, 2006; Mateos-Rivera et al., 2016; Schütte et al., 2010; Seok et al., 2016; Venkatachalam et al., 2021). However, unlike microbial succession along glacier foreland chronosequences elsewhere (e.g., Mateos-Rivera et al., 2016; Schütte et al., 2009; Venkatachalam et al., 2021), relative abundance of microbial communities in soils from the Vatnajökull ice cap did not increase with the age of soils. Relative abundance of Acidobacteria and Actinobacteria decreased along the some glacier foreland transects in Iceland, while other dominant phyla did not vary with the age of soils. Therefore, it seems that with the exception of Acidobacteria and Actinobacteria microbial communities in soils from the Vatnajökull ice cap adapted to the new environment and homogenized quickly along the glacier foreland transects as suggested by Schütte et al. (2009).

#### **5.3.4.4 Relationship between microbial community and organic biomarkers**

Specific biomarkers are associated with particular environments and source organisms. Table 4.4 shows source organisms of specific organic biomarkers, abundant in Icelandic

samples. An attempt was made to link specific members of the microbial community detected in Iceland to production of these organic biomarkers. BHPs, including soil marker BHPs, were detected in all analysed samples from the Vatnajökull ice cap. Adenosylhopane and adenosylhopane type 3 were the most abundant soil marker BHPs. Soil marker BHPs are generally abundant in soils (Cooke et al., 2008; Doğrul Selver et al., 2012; Höfle et al., 2015) and in this study, were used as indicators of soil development in glacier forelands in Iceland and therefore, this compound and microorganisms producing it are of particular interest.

Adenosylhopane and adenosylhopane type 3 were detected in all samples from Iceland. Some bacteria responsible for production of soil marker BHPs were also detected in these samples. For instance, family-level bacterium Bradyrhizobiaceae (Proteobacteria) was one of the dominant bacteria in Icelandic samples, except sample SK-TM-2209. A member of this family, *Bradyrhizobium japonicum*, was reported to produce soil marker BHPs (as cited in table 2.2a, Cooke, 2011). Relative abundance of this bacteria did not follow a particular trend, but also it did not follow the same pattern as downstream distribution of soil marker BHPs. There was also a reasonable amount of family-level bacterium Hyphomicrobiaceae (Proteobacteria) in all samples except for SK-TM-2209. A member of this family, a purple non-sulfur bacterium *Rhodomicrobium vannielii* (Neunlist et al., 1985), produces soil marker BHPs. Relative abundance of Hyphomicrobiaceae also did not follow a particular trend or a trend of soil marker BHPs distribution along the downstream transects. Further, adenosylhopane producing Rhodospirillaceae, also known as purple non-sulfur bacteria (as cited in table 2.2a, Cooke, 2011) was present in small quantities (< 2%) in all samples from Iceland. Similarly, adenosylhopane producing family Nitrosomonadaceae (species *Nitrosomonas europaea* (as cited in table 2.2a; Seemann et al., 1999)) was detected in LIA moraine samples from Svínafellsjökull. This suggests that the moraine is inhabited by bacteria that is not common in other sites in Iceland, which could be due to presence of invasive species Nootka lupine (*Lupinus nootkatensis*).

Other abundant BHP compounds in samples from Iceland were BHTs, Me-BHT, aminotriol, BHT glucosamine and BHT cyclitol ether. Relative abundance of these biomarkers did not follow a particular trend along the transects. As mentioned earlier, family Bradyrhizobiaceae (Proteobacteria) was one of the dominant bacteria in Icelandic samples.

Species belonging to this family are responsible for producing BHP compounds other than soil marker BHPs. For instance, *Bradyrhizobium japonicum* also produces aminotriol (Bravo et al., 2001), *Rhodopseudomonas palustris* produces Me-BHT (as cited in table 2.2a, Cooke, 2011), and *Rhodopseudomonas acidophila* produces BHT (Flesch and Rohmer, 1988). Another abundant family-level bacterium in samples from Iceland was Sphingomonadaceae. A species of this family, *Zymomonas mobilis*, was reported to produce BHT, BHT cyclitol ether and BHT glucosamine (Flesch and Rohmer, 1988; Flesch and Rohmer, 1989; Moreau 1997; Neunlist et al., 1988; Talbot et al., 2001; as cited in table 2.2a, Cooke, 2011). Xanthomonadaceae was also abundant in Icelandic samples, species of this family, *Frateuria aurantia*, was reported to produce BHT and BHT cyclitol ether (Joyeux et al., 2004). There was also a reasonable amount of Hyphomicrobiaceae (Purple non-sulfur bacterium *Rhodomicrobium vannielii* (Neunlist et al., 1985)), which produces BHT as well as soil marker BHPs (discussed earlier).

Some other bacteria from Table 4.4 were detected in samples from Iceland, however, their relative abundance was low and did not follow a particular pattern of downstream distribution. For instance, Rhodospirillaceae, also known as purple non-sulfur bacteria (as cited in table 2.2a, Cooke, 2011), which was present in small quantities (< 2%) in all samples from Iceland produces BHT as well as adenosylhopane. Similarly, members of family-level bacterium Nitrosomonadaceae (species *Nitrosomonas europaea* (as cited in table 2.2a; Seemann et al., 1999)) detected in LIA moraine samples from Svínafellsjökull also produces aminotriol (as well as soil marker BHPs). Some family-level bacteria, members of which produce BHT, were detected in the samples in small quantities: Nostocaceae (*Anabaena cylindrica* (Talbot et al, 2008), *Nostoc muscorum* (Bisseret et al., 1985; Talbot et al, 2008)), and Frankiaceae (*Frankia* (Berry et al., 1991; Rosa-Putra et al., 2001)). A small amount of family-level bacterium Methylococcaceae, a member of which (*Methylococcium album* (Talbot et al., 2001)) produce aminotriol, was detected in samples from Kviarjökull, suggesting that this glacier foreland has developed different microbial community compared to other glaciers studied in this work.

Generally, most of the bacteria detected in Icelandic samples produce more than one BHP compound. For instance, on top of the already discussed bacteria, members of Acetobacteraceae produce both BHT (genus *Acetobacter* (Peiseler and Rohmer, 1992;

Talbot et al., 2007a), genus *Gluconacetobacter* (Talbot et al., 2007a), specie *Acetobacter xylinum* (Ourisson and Rohmer 1992; Herrmann et al., 1996)) and BHT cyclitol ether (specie *Acetobacter xylinum* (Ourisson and Rohmer 1992; Herrmann et al., 1996)). Pseudomonadaceae (specie *Azotobacter vinelandii* (Vilcheze et al., 1994)) also produce both BHT and BHT cyclitol ether. Members of Beijerinckiaceae (*Beijerinckia indica* and *Beijerinckia mobilis* (Vilcheze et al., 1994)) and Methylocystaceae (*Methylocystis parvus* (Talbot et al., 2001), *Methylosinus trichosporium* (Neunlist and Rohmer, 1985a)) produce BHT and aminotriol. Cyanobacteria (Talbot et al., 2003a; Talbot et al, 2008; as cited in table 2.2a, Cooke, 2011) produce BHT and Me-BHT. BHT, BHT cyclitol ether and aminotriol are produced by a member of family-level bacterium Desulfovibrionaceae (*Desulfovibrio* (Blumenberg et al., 2006)); BHT, BHT cyclitol ether, aminotriol, and Me-BHT are produced by a member of Methylobacteriaceae (*Methylobacterium organophilum* (as cited in table 2.2a, Cooke, 2011; Bisseret et al., 1985; Flesch and Rohmer, 1988; Knani et al., 1994; Neunlist et al., 1988; Renoux and Rohmer, 1985; Talbot et al, 2007b)). The fact that bacteria detected in Icelandic samples produce more than one BHP compound and that their distribution pattern along glacier foreland transects in Iceland did not match that of specific biomarkers makes it difficult to pinpoint the original source of the most abundant biomarkers in samples from Iceland.

BrGDGTs is another biomarker specific to terrestrial OC (Hopmans et al., 2004; Weijers et al., 2006; Doğrul Selver et al., 2012) and was detected in all samples from Iceland. Acidobacteria, which is potentially responsible for producing brGDGTs (Weijers et al., 2009), was one of the dominant phyla in samples from Iceland. Distribution pattern of these biomarker and bacterium did not follow the same trend along downstream transects of glacier forelands, suggesting that perhaps not all members of this phyla produce brGDGTs.

To summarise, based on the findings of this study, it seems that members of Proteobacteria (Sphingomonadaceae, Xanthomonadaceae and Bradyrhizobiaceae) have highly contributed to the abundance of BHPs, and as suggested by Weijers et al. (2009), Acidobacteria is a potential source of brGDGTs, while other bacteria had some influence in the accumulation of BHP compounds. Most of the bacteria in samples from Iceland linked to organic biomarker production had low relative abundance. Nevertheless, bacteria with low relative abundance may have produced organic biomarkers detected in Icelandic

samples either by adding to the accumulation of organic biomarkers over a long period of time or the current microbial community might not be representative of the biomarkers produced by different bacteria in the past.

## **5.4 Conclusion**

The Vatnajökull ice cap in Iceland is an older catchment compared to the Tarfala valley in Sweden with higher OC content and may be comparable to similar sites elsewhere (e.g., Svalbard (Wietrzyk et al., 2018) and Switzerland (Prietz et al., 2013)). There was a clear pattern of increase in TOC (1) from sediments to soils and (2) from younger to older soils. This strengthens the hypothesis that soils accumulate OC along glacier foreland transects. Four glaciers (Svínafellsjökull, Virkisjökull, Kviarjökull and Fjallsjökull) were further investigated for organic biomarkers and had detectable amounts of BHPs and brGDGTs. Abundance of soil marker BHPs in Iceland was comparable to that in Tarfala, some estuary sediment samples in the Arctic (Cooke et al., 2009), and some soils in Northern England (Cooke et al., 2008), however, was much lower than organic rich permafrost (Doğrul Selver et al., 2015; Rethemeyer et al., 2010). Moreover, distribution of soil marker BHPs and the  $R'_{\text{soil}}$  index in soil samples from Iceland suggest that soils in this deglaciated catchment have been accumulating soil derived OC and developing. Despite the expectations, concentration and distribution of soil BHPs along the downstream transects of different glaciers had varying patterns. Therefore, observations of BHP distribution at the Vatnajökull ice cap suggest that the initial accumulation of soil derived OC in deglaciated catchments (1) plateaus out after reaching a certain stage and (2) other BHP compounds produced by various microorganisms overtake and mask soil marker BHPs. Observations also suggest that the age of moraines does not play any role in the production and preservation of brGDGTs. The analysis showed that both metals and OC content played an important role in the establishment of microbial communities. Major phyla identified in samples from the Vatnajökull ice cap were Acidobacteria, Actinobacteria, Chloroflexi, Planctomycetes, Proteobacteria and Verrucomicrobia, which is similar to that in other polar and alpine habitats, as well as soils in general (e.g., Chu et al., 2010; Costello and Schmidt, 2006; Ji et al., 2016; Mateos-Rivera et al., 2016; Schütte et al., 2010; Seok et al., 2016; Venkatachalam et al., 2021). There was not much variation in the relative abundance of

most bacteria (except Acidobacteria and Actinobacteria) along the transects, suggesting that they could have adapted to the new environments quickly. Additionally, Acidobacteria, which are a potential source of brGDGTs (Weijers et al., 2009), and members of Proteobacteria (Sphingomonadaceae, Xanthomonadaceae and Bradyrhizobiaceae) have highly contributed to the abundance of biomarkers, while others produced small amounts of BHP compounds. Overall, this study of glacier forelands in Iceland showed that though observed patterns were not clear it is important to look at both soil marker BHPs and brGDGTs, as well as to investigate microbial communities to understand accumulation of soil derived OC along glacier foreland transects. Even though time of exposure after deglaciation influenced initial accumulation of soil marker BHPs, it is not a dominant factor as it did not influence the downstream trends observed in GDGTs and microbial communities .

## 6 Accumulation of soil derived OC at the Zackenberg valley, Greenland

### 6.1 Introduction

This chapter investigates accumulation of soil OC along the Zackenberg valley in NE Greenland. Zackenberg valley has been deglaciated for the longest period out of the three study sites (Tarfala valley in Sweden and Vatnajökull ice cap in Iceland studied in previous chapters), being ice free since the Early Holocene (ca. 10.5 ka) (Garcia-Oteyza et al., 2022). Therefore, it was expected that this catchment was more complex and had a higher rate of soil OC accumulation. Soil and sediment samples were collected along one 50 km long transect at the Zackenberg valley (Figure 3.22) to investigate changes in environmental chemistry, organic biomarkers, and microbial community structure.

The aim of this chapter was to understand accumulation of soil derived OC by looking at catchment chemistry, organic biomarkers and microbial community along the downstream transect of the Zackenberg valley. This was achieved by carrying out the following objectives:

1. To determine TOC, C/N and major metals along the transect;
2. To determine concentration and distribution of BHPs and GDGTs and their proxies ( $R'_{\text{soil}}$ , MAT and soil pH) in selected soil samples based on the results of objective 1 along the transect;
3. To determine microbial community structure in soil and sediment samples selected based on the results of objective 1;
4. To establish a relationship between organic biomarker distribution and microbial community composition in samples from the Zackenberg valley.

Based on these objectives following outcomes were hypothesised:

1. Samples from downstream locations along the transect have higher TOC content due to the accumulation of OM over time, while upstream samples closer to the glacier front are more mineral.
2. Similar to the previous sites, there are no changes in metal concentrations along the transect.
3. Since the catchment has been deglaciated for such a long time, total BHP and GDGT signals have stabilised over time.
4. Similarly, the  $R'_{\text{soil}}$  does not vary along the transect, indicating stabilisation of soil marker BHPs.
5. The bacterial community has also homogenized over time and there wouldn't be much variation along the downstream transect.
6. Soils are more abundant in OC and have higher bacterial diversity compared to sediments.

## 6.2 Results

### 6.2.1 Environmental chemistry

23 samples (10 sediment, 13 soil samples) from the Zackenberg valley in NE Greenland were analysed for TC, TOC, TN, C/N (see Appendix 13 for the sample names, raw data, mean concentration and standard deviation (SD)) and metal concentrations (see Appendix 14 for the mean and SD values). Samples were collected in triplicates.

#### 6.2.1.1 Variation of chemical properties in different sample types and transects

A PCA was conducted on the mean values of triplicates of all soil and sediment samples from the Zackenberg valley (Figure 6.1). PC1 explained 63.5% of total variance, while PC2 explained 16.2%. PC1 had strong positive correlation with Al, Co, Cr, Ni, Mn, Pb, K, Fe, as well as Cu, Cd, and Zn. PC2 had strong positive correlation with P and Ca, and strong negative correlation with As and S. Interestingly, TC, TOC and N had low correlation with PC1 and PC2.

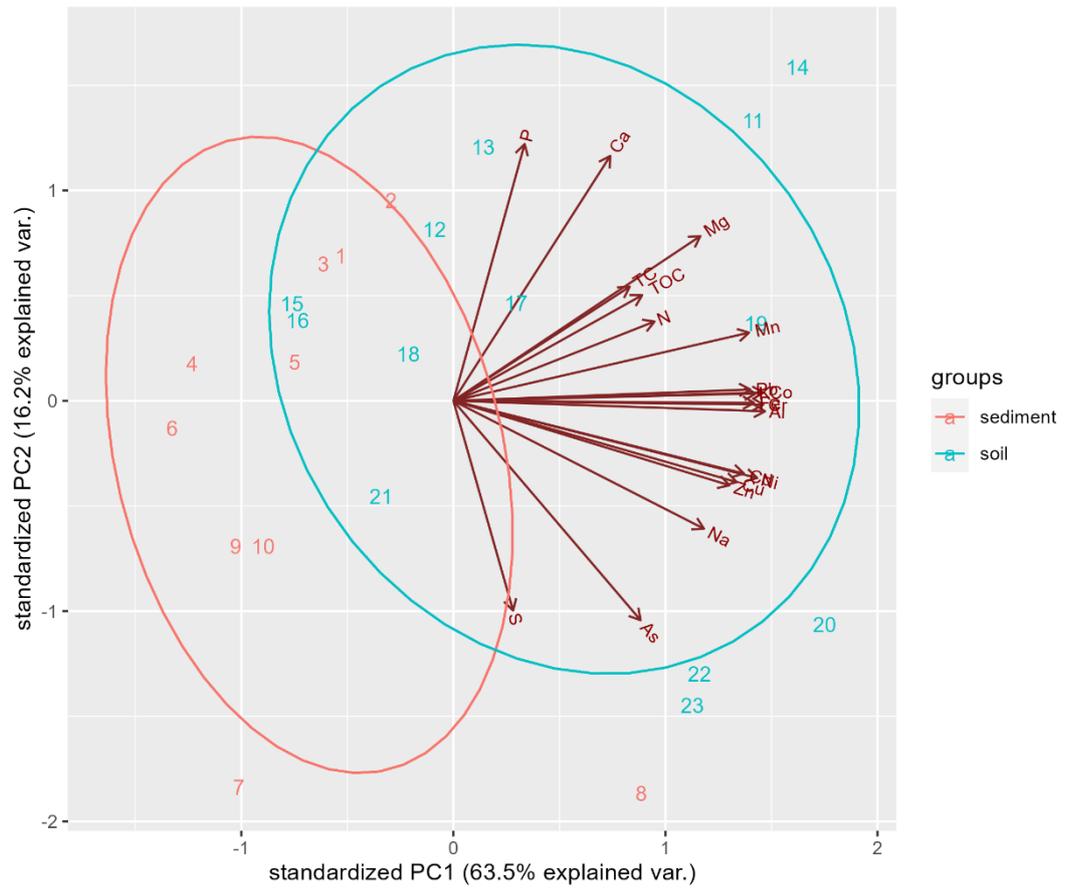


Figure 6.1. PCA ordination plot illustrating the discrimination between mean of triplicates of all samples from the Zackenberg valley, Greenland according to chemical properties by the sample type. The input data was standardised to have zero mean and variance one. The ellipses represent the core area added by confidence interval of 68%. Sample codes are given in Table 6.1.

Table 6.1. Codes of sample names from the Zackenberg valley, Greenland shown on the PCA plots above (Figure 6.1).

Symbol	Sample	Type	Symbol	Sample	Type
1	G-1408	sediment	13	SS-D-0508	soil
2	SS-D-0508	sediment	14	SS-LI-1408	soil
3	SS-L-1008	sediment	15	SS-L-1008	soil
4	SS-L-1308	sediment	16	SS-L-1308	soil
5	ZR-SS-1508	sediment	17	SS-O-1008	soil
6	ZR-BL-0108	sediment	18	ZR-SS-1508	soil
7	ZR-LM-1608	sediment	19	ZR-BL-0108	soil
8	ZR-AC-1608	sediment	20	ZR-LM-1608	soil
9	ZR-B-0808	sediment	21	ZR-AC-1608	soil
10	ZR-S-0208	sediment	22	ZR-B-0808	soil
11	G-1408	soil	23	ZR-S-0208	soil
12	AHR-0508	soil			

PERMANOVA analysis showed that there was statistically significant difference between soil and sediment samples ( $p$ -value < 0.05). A PCA plot indicated that sediment samples were clustered to the left of soil samples (Figure 6.1), away from all vectors. Sediment samples were distributed along PC2 at a  $90^\circ$  to TOC and TN, indicative of variation in chemistry by some metals rather than C. There was some overlap between soil (15, 16, 18, 21) and sediment samples (1, 2, 3, 5) (see Table 6.1 for the sample names). Soil samples 15 and 18 and sediment samples 3 and 5 collected at the same location along the transect, indicating little difference in chemical composition between soils and sediment samples.

### 6.2.1.2 Total organic carbon concentration

Figure 6.2 shows sampling locations and total organic carbon (%) distribution along the downstream transect of the Zackenberg valley. Soil samples had higher TOC content compared to sediment samples: mean TOC concentration of sediment samples was  $0.25 \pm 0.50\%$  ( $n=10$ ), ranging from  $0.01 \pm 0.01$  ( $n=3$ ) to  $1.33 \pm 1.38\%$  ( $n=3$ ) across field triplicates and  $1.62 \pm 1.54\%$  ( $n=13$ ) ranging from  $0.30 \pm 0.15$  ( $n=3$ ) to  $4.88 \pm 3.56\%$  ( $n=3$ ) in soil samples.



Figure 6.2. Sampling locations and total organic carbon (%) distribution along the Zackenberg River Drainage Basin, Greenland (Modified from Google Earth, 2012).

Figure 6.3 shows distribution of mean TOC concentration (%) in relation to the Zackenberg valley downstream transect. There was no apparent trend in sediment (Figure 6.3a) or soil (Figure 6.3b) distribution along the transect. TOC concentrations in sediment samples were highest in sample SS-L-1008 ( $1.33 \pm 1.38\%$ ,  $n=3$ ), while samples ZR-BL-0108 ( $0.01 \pm 0.01\%$ ,  $n=3$ ) and G-1408 ( $0.02 \pm 0.02\%$ ,  $n=3$ ) had the lowest values (Appendix 13). TOC concentrations in soil samples collected along the Store Sø Lake were generally stable. It is worth mentioning that the signal was diluted as tributaries Lindemanselven and Palnatokeelv rivers and Aucellaelv river joined the Zackenberg river.

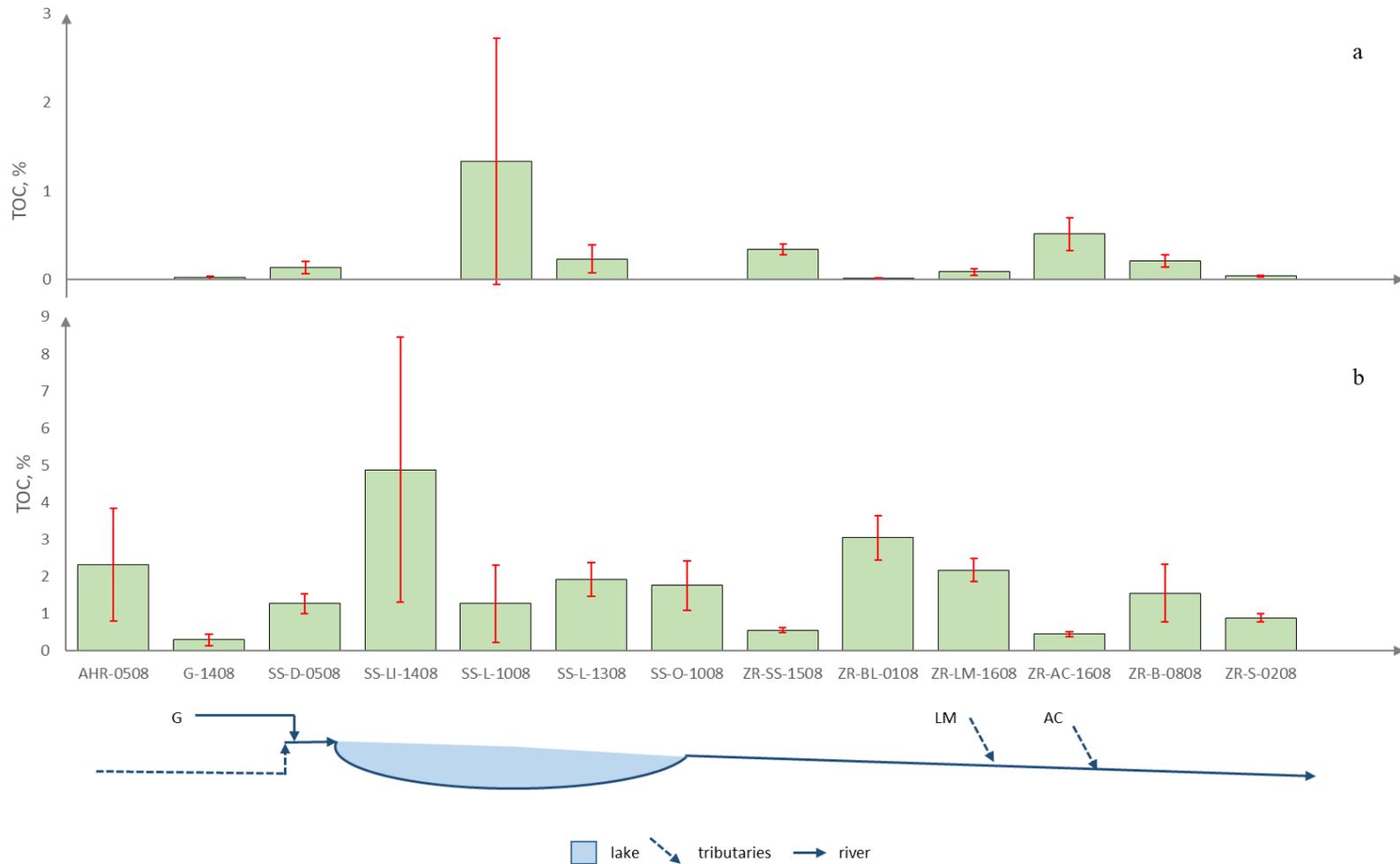


Figure 6.3. Mean distribution of TOC (%) in (a) sediment and (b) soil samples along the Zackenberg valley downstream transect in NE Greenland. Error bars represent  $\pm$  one standard deviation on field triplicate samples collected from individual sampling locations at the Zackenberg valley. Charts at the bottom of the graph show downstream (left to right) transect profile. G stands for glacier meltwater, LM – Lindemanselv and Palnatokeelv rivers, AC – Aucellaelv river. Note: no sediment samples were collected from locations AHR-0508, SS-LI-1408, and SS-O-1008.

### 6.2.1.3 Total nitrogen and C/N ratio

Mean TN concentrations in sediment samples ranged from  $0.01\pm 0.01\%$  ( $n=3$ ) to  $0.08\pm 0.07\%$  ( $n=3$ ) and from  $0.04\pm 0.02\%$  ( $n=3$ ) to  $0.23\pm 0.15\%$  ( $n=3$ ) in soil samples (Appendix 13). Mean C/N ratio was also lower in sediment samples ( $6.77\pm 5.82$ ,  $n=10$ ) compared to soil samples ( $14.13\pm 4.66$ ,  $n=13$ ) (Appendix 13). There was a strong positive correlation between TOC (%) and TN (%) across the samples collected along the Zackenberg valley ( $R^2=0.92$ , Pearson correlation=0.96) (Figure 6.4), suggesting that TN is organic in nature.

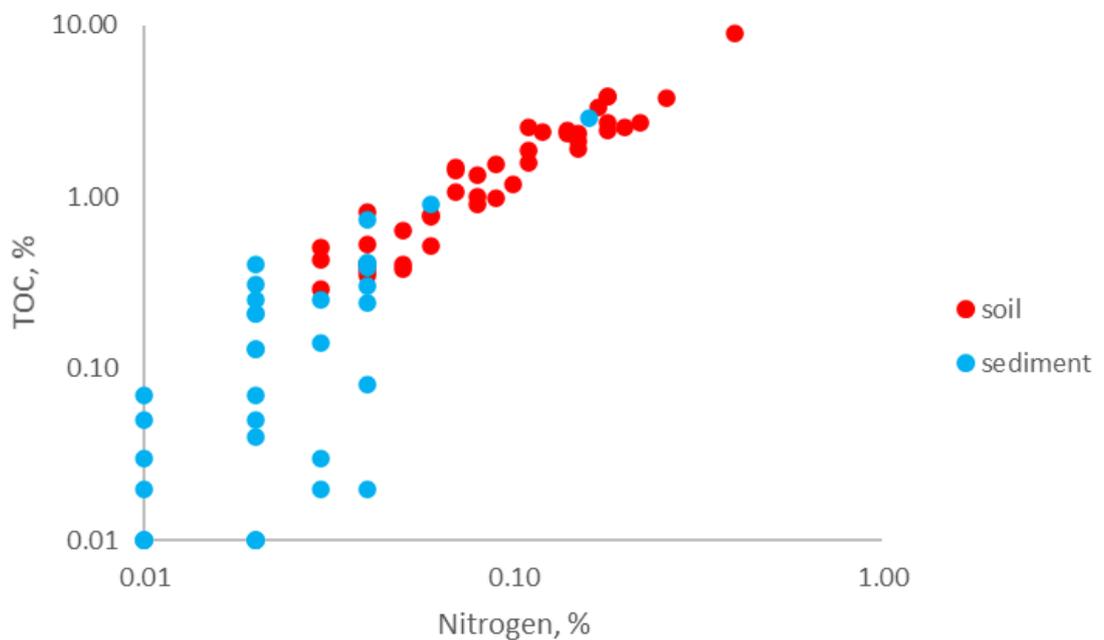


Figure 6.4. Relationship between TOC (%) and total nitrogen (%) for the downstream transect at the Zackenberg valley (logarithmic axes).

### 6.2.1.4 Metal concentration

Distribution of the mean metal concentration in sediment and soils samples is demonstrated in Figure 6.5. Total metal concentration in Greenland was  $10741\pm 4027$  mg/kg ( $n=23$ ) and was higher in soil samples ( $12701\pm 3810$  mg/kg,  $n=13$ ) compared to sediment samples ( $8,192\pm 2,741$  mg/kg,  $n=10$ ). Mean and SD of individual metal concentrations in soil and sediment samples are presented in Table 6.2 (see Appendix 14 for more details). Fe, Al, Ca and Mg had the highest concentrations, followed by K, P, Na, S and Mn, while Zn, Cr, Cu, Ni, Co, Pb, As and Cd had lower concentrations (Table 6.2).

The PCA plot in Figure 6.1 indicates that sediment samples were influenced by PC2, which had strong correlation with some metals (P, Ca, As and S). Concentration of S was higher in sediments compared to soils, while As was not detected in samples from Sweden and Iceland.

There was no particular pattern of distribution of metals in sediments (Figure 6.5a) and soils (Figure 6.5b) along the downstream transect in Zackenberg. Soil sample G-1408 had the highest total metal concentration (19745 mg/kg), while sediment sample SS-L-1308 had the lowest concentration (5975 mg/kg).

6 Soil development at Zackenberg valley, Greenland

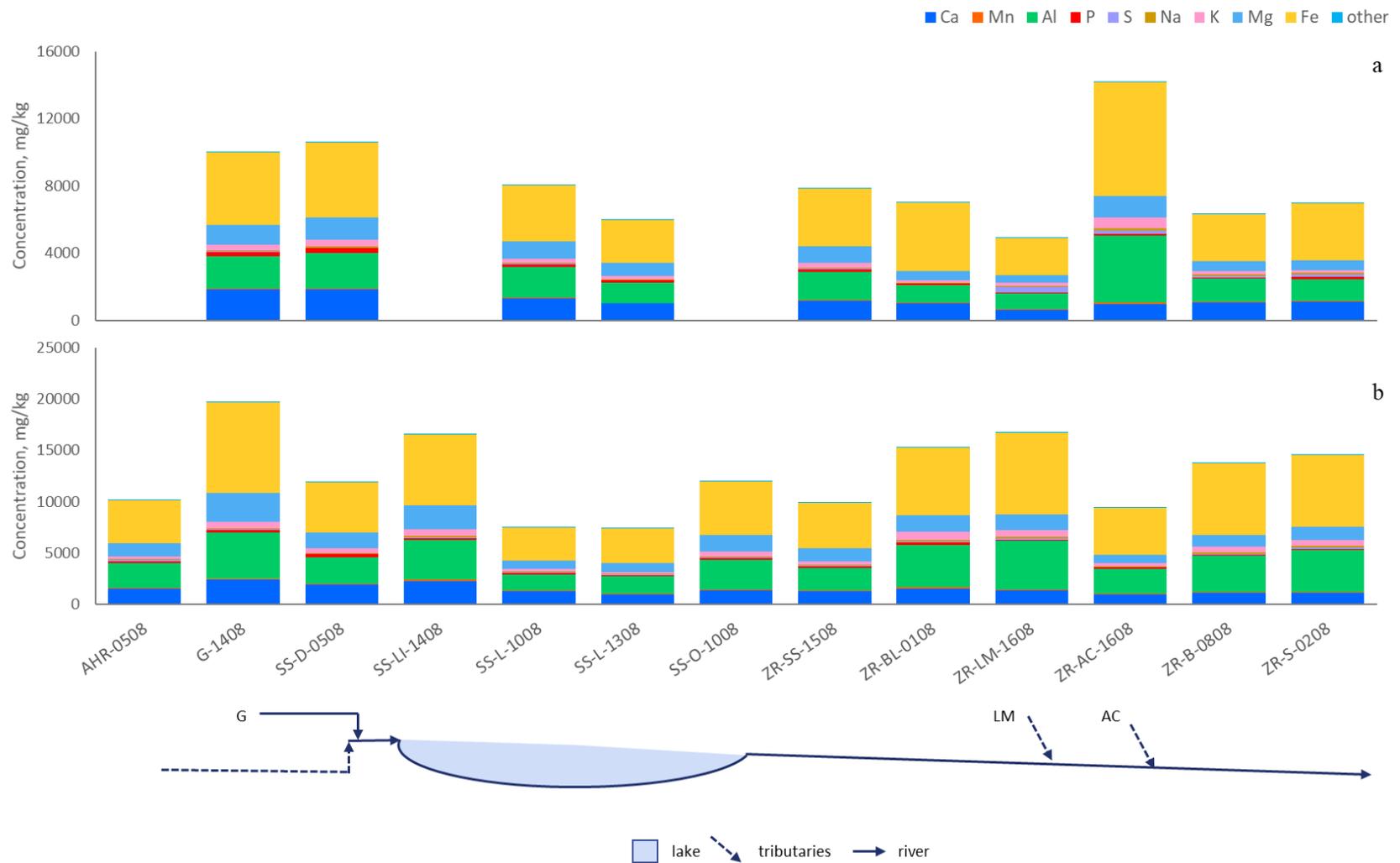


Figure 6.5. Mean concentration of metals (mg/kg) in (a) sediment and (b) soil samples along the Zackenberg valley downstream transect in NE Greenland. Other metals include Cr, Co, Ni, Cu, Zn, As, Pb and Cd. Charts at the bottom of the graph show downstream (left to right) transect profile. G stands for glacier meltwater, LM – Lindemanselv and Palnatokeelv rivers, AC – Aucellaelv river.

Table 6.2. Mean and standard deviation (mg/kg) of metal concentrations in soil and sediment samples from the Zackenberg valley, Greenland.

Metal	Soil		Sediment	
	Mean	SD	Mean	SD
Fe	5718	±1799	3739	±1305
Al	3119	±1070	1735	±876
Ca	1470	±457	1191	±384
Mg	1454	±577	867	±319
K	484	±204	284	±161
P	179	±59	149	±75
Na	102	±32	73	±24
S	74	±38	93	±112
Mn	72	±24	44	±14
Zn	9.80	±3.67	6.57	±2.90
Cr	6.30	±2.11	3.30	±1.66
Cu	5.62	±2.49	3.55	±1.70
Ni	2.96	±1.02	1.82	±0.87
Co	2.53	±0.74	1.61	±0.54
Pb	0.97	±0.21	0.67	±0.19
As	0.48	±0.45	0.33	±0.33
Cd	0.43	±0.13	0.31	±0.10

## 6.2.2 Biomarkers

In order to understand accumulation of soil derived OC along the Zackenberg valley transect, only soil samples were investigated further for organic biomarkers because soil samples had higher TOC concentration compared to sediments and because sediment samples from Sweden did not show many signals of organic biomarker. At least one replicate of each soil sample presented in Figure 6.2 was analysed for organic biomarkers (see Appendix 13 to see the samples chosen for organic biomarker analysis - TOC values highlighted in red).

### 6.2.2.1 BHP concentrations and distribution pattern

BHPs were present in all analysed soil samples from the Zackenberg valley. Overall, up to 31 individual BHPs were identified and grouped into five main categories (BHT, amino, sugar, aminosugar and soil markers) (Appendix 17). The relative concentration of total BHPs ranged from 2499 to 9428  $\mu\text{g/g}$  TOC (mean=5230 $\pm$ 1876  $\mu\text{g/g}$  TOC), while mean absolute concentration was 90 $\pm$ 76  $\mu\text{g/g}$  sediment. BHT was the most abundant individual BHP (2933 $\pm$ 1144  $\mu\text{g/g}$  TOC), followed by methylated BHT (634 $\pm$ 323  $\mu\text{g/g}$  TOC), soil marker BHPs,

adenosylhopane ( $355\pm 157$   $\mu\text{g/g}$  TOC) and adenosylhopane type 3 ( $248\pm 107$   $\mu\text{g/g}$  TOC), aminotriol ( $215\pm 122$   $\mu\text{g/g}$  TOC), BHT isomer ( $142\pm 62$   $\mu\text{g/g}$  TOC), and BHT glucosamine ( $103\pm 55$   $\mu\text{g/g}$  TOC).

There was no apparent trend along the downstream transect of the Zackenberg valley (Figure 6.6). The sample collected closest to the glacier (G-1408) had the lowest relative concentration of total BHPs (2499  $\mu\text{g/g}$  TOC). Relative concentrations of total BHPs increased along the Store Sø Lake (from 4909  $\mu\text{g/g}$  TOC in SS-D-0508 to 9428  $\mu\text{g/g}$  TOC in SS-O-1008), and then decreased after Zackenberg river outflows from the lake (6630  $\mu\text{g/g}$  TOC in ZR-SS-1508 and 3721  $\mu\text{g/g}$  TOC in ZR-BL-0108) and tributaries Lindemanselven and Palnatokeelv (3649  $\mu\text{g/g}$  TOC in ZR-LM-1608) and Aucellaelv (3157  $\mu\text{g/g}$  TOC in ZR-AC-1608) join the river. After that, the relative concentration of total BHPs increased (5317  $\mu\text{g/g}$  TOC in ZR-B-0808) and slightly decreased (3994  $\mu\text{g/g}$  TOC in ZR-S-0208) again before inflowing into the fjord.

Sample G-1408 had a relatively high  $R'_{\text{soil}}$  index (0.28), which is used to trace soil marker BHPs (Figure 6.6). The  $R'_{\text{soil}}$  had a downward trend along the Store Sø Lake (from 0.23, SS-D-0508 to 0.13, SS-O-1008) and after the Zackenberg river outflows from the lake (0.11, ZR-SS-1508). Sample collected along the Zackenberg river after the river outflows from the lake but before the tributaries join (ZR-BL-0108) had the highest  $R'_{\text{soil}}$  index (0.37).  $R'_{\text{soil}}$  decreased after tributary rivers Lindemanselven and Palnatokeelv (0.12, ZR-LM-1608) and Aucellaelv (0.12, ZR-AC-1608) join the Zackenberg river. These values increased downstream after the mixing (0.22 and 0.20, ZR-B-0808 and ZR-S-0208).

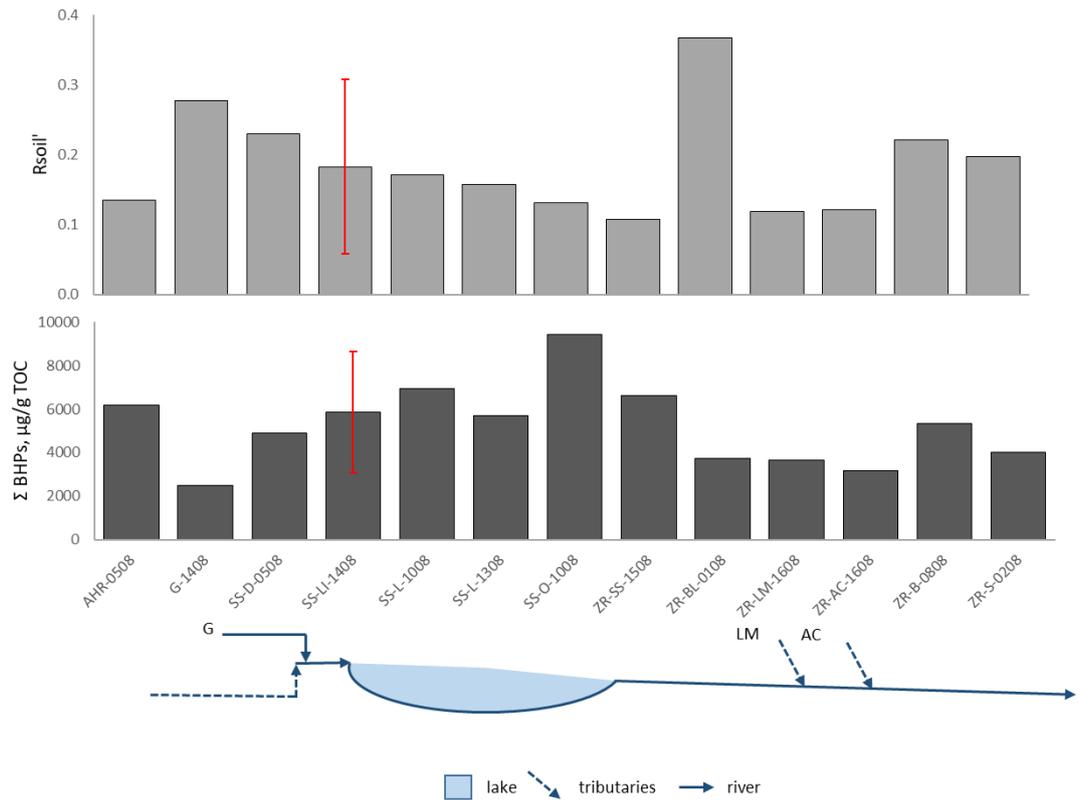


Figure 6.6. Average distribution of total BHPs ( $\mu\text{g/g TOC}$ ) and  $R'_{\text{soil}}$  along the Zackenberg valley in NE Greenland. The error bar represents  $\pm$  one standard deviation on field duplicate samples collected from the sampling location SS-LI-1408. Only one field replicate was analysed for organic biomarkers from other sampling locations. Charts at the bottom of the graph show downstream (left to right) transect profiles. G stands for glacier runoff, LM - Lindemanselven and Palnatokeelv rivers and AC - Aucellaelv River.

Fractionally, soil marker BHPs ranged from 8 to 30% of total BHPs (Figure 6.7). Soil samples collected along the meltwater stream (G-1408) and at the Zackenberg river before tributaries Lindemanselven and Palnatokeelv join the river (ZR-BL-0108) had the highest soil marker BHP fractions (0.22 and 0.30, respectively). Along the transect there was a downward trend in soil BHPs from meltwater stream (G-1408) to after the Zackenberg river outflows from the Store Sø lake (ZR-SS-1508) and an upward trend downstream of the Zackenberg river after joining of the Lindemanselven and Palnatokeelv rivers (ZR-LM-1608). Overall, BHTs made up the majority of total BHPs, ranging from 63 to 79%, while amino (3-11% of total BHPs), aminosugar (2-10% of total BHPs) and sugar (1-7% of total BHPs) compound groups had smaller fractions.

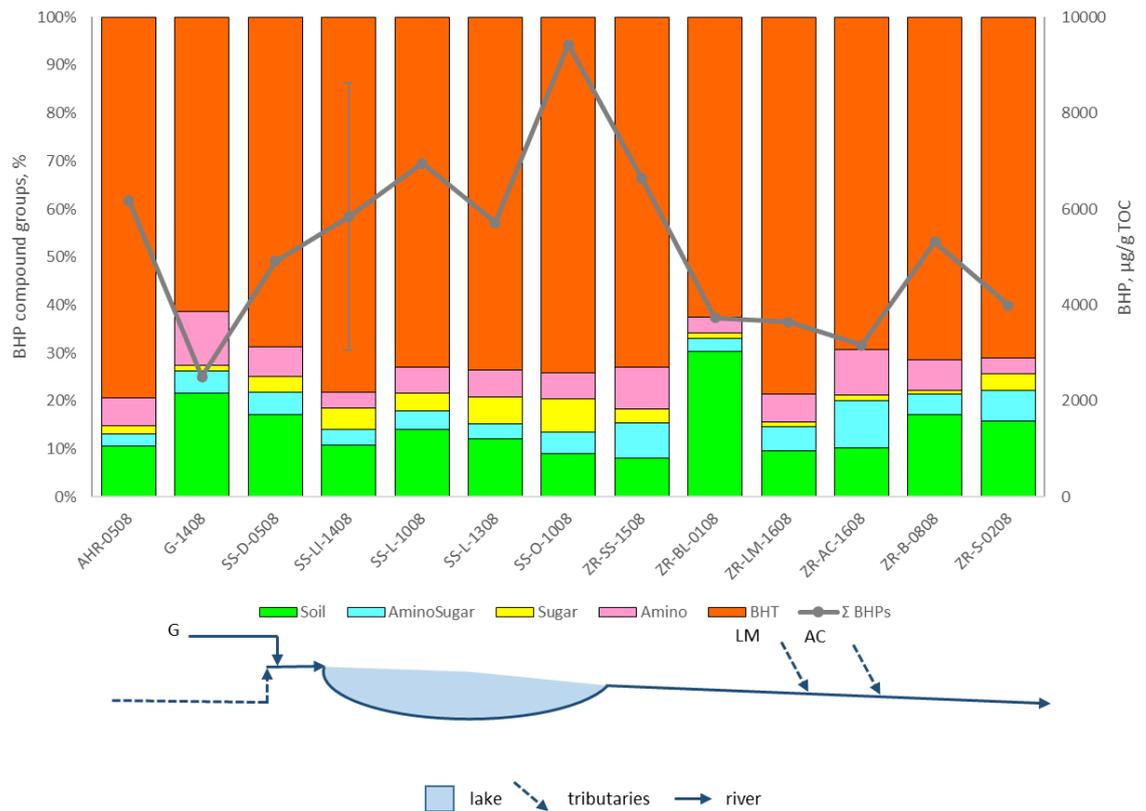


Figure 6.7. Total BHP concentration (grey circles and lines) and fractional abundance of BHP compound groups (BHTs, aminos, sugars, aminosugars and soil BHPs; coloured bars) along the Zackenberg valley in NE Greenland. The error bar represents  $\pm$  one standard deviation on field triplicate samples collected from the sampling location SS-LI-1408. Only one field replicate was analysed for organic biomarkers from other sampling locations. Charts at the bottom of the graph show downstream (left to right) transect profiles. G stands for glacier runoff, LM - Lindemanselv and Palnatokeelv rivers and AC - Aucellaelv River.

### 6.2.2.2 GDGT concentrations and distribution along the transects

Branched GDGTs were detected in all soil samples collected at the Zackenberg valley (Figure 6.8), the most abundant brGDGTs being Ia, IIa and IIIa compounds. Crenarchaeol was also detected in samples G-1408, ZR-BL-0108 and ZR-B-0808. The mean relative concentration of total GDGTs was  $144 \pm 73$   $\mu\text{g/g TOC}$  ranging from  $53$   $\mu\text{g/g TOC}$  (SS-LI-1408) to  $325$   $\mu\text{g/g TOC}$  (SS-L-1008) (Appendix 17), while the mean absolute concentration was  $2.19 \pm 1.25$   $\mu\text{g/g sediment}$ . There was no obvious pattern of total GDGTs distribution along the downstream transect, it varied along the Store Sø Lake ( $53$ - $325$   $\mu\text{g/g TOC}$ ) and along the Zackenberg river ( $54$ - $199$   $\mu\text{g/g TOC}$ ) (Figure 6.8).

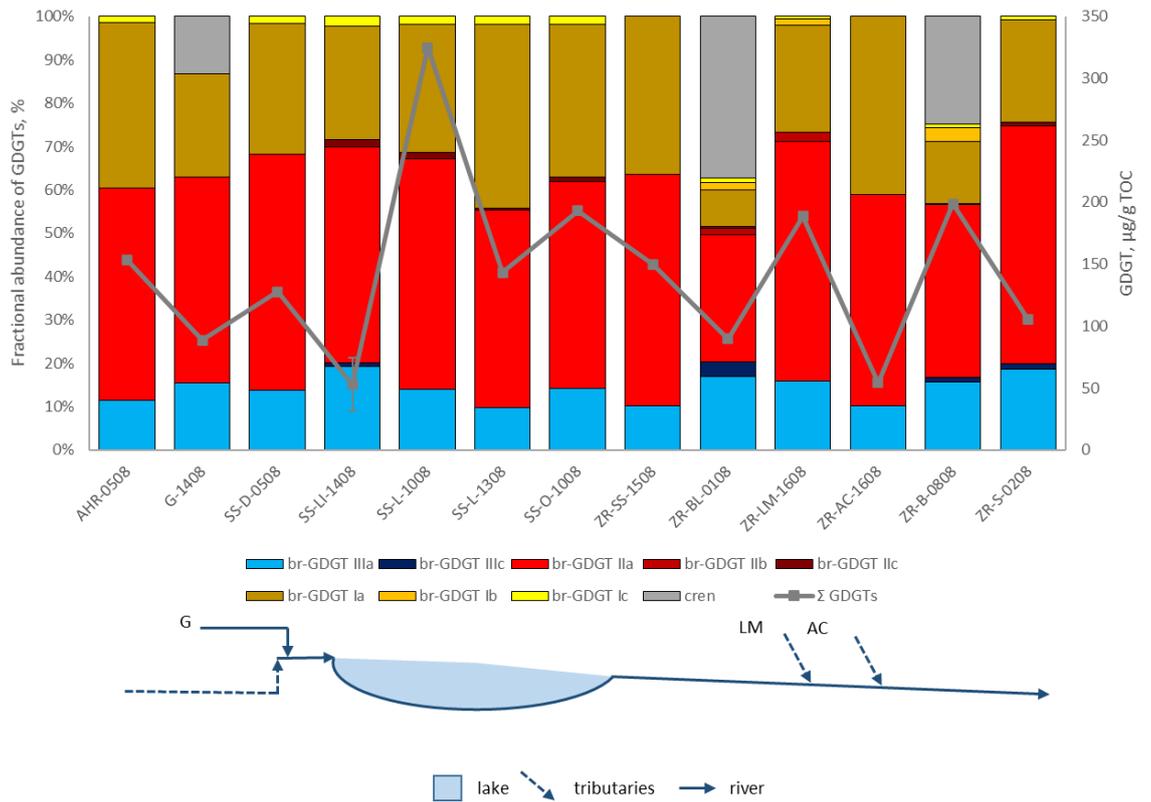


Figure 6.8. Relative concentration of total GDGTs (grey circles and lines) and fractional abundance of branched GDGT compounds and crenarchaeol GDGT (coloured bars) along the Zackenberg valley in NE Greenland. The error bar represents  $\pm$  one standard deviation on field triplicate samples collected from the sampling location SS-LI-1408. Only one field replicate was analysed for organic biomarkers from other sampling locations. Charts at the bottom of the graph show downstream (left to right) transect profiles. G stands for glacier runoff, LM - Lindemanselven and Palnatokeelv rivers and AC - Aucellaelv River.

### 6.2.2.2.1 GDGT proxies

GDGTs can be used as a proxy of past environmental changes, allowing to analyse GDGT-based soil pH and mean annual air temperature (MAT). GDGT proxy soil pH varied from 4.6 to 7, while MAT fluctuated between -2.8 and 5.8 °C (Figure 6.9). There was no particular pattern of distribution along the downstream transect of either of the proxies.

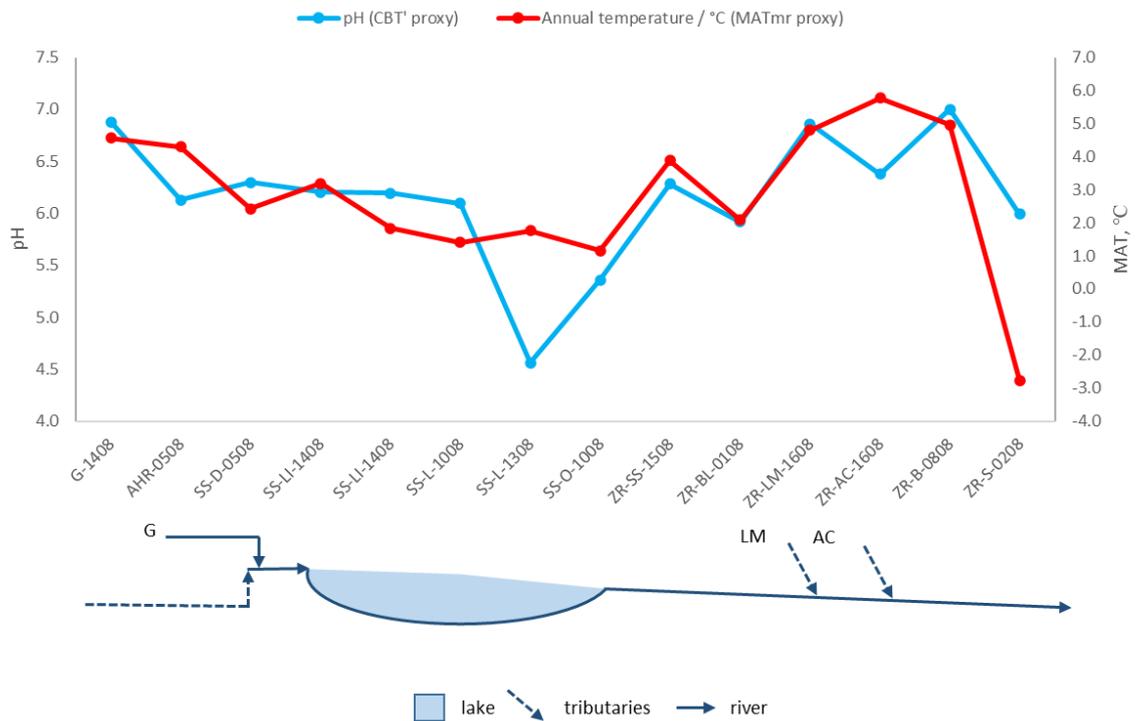


Figure 6.9. Downstream distribution of GDGT proxies, soil pH and annual mean air temperature (MAT, °C) along the Zackenberg valley transect in Greenland. Charts at the bottom of the graph show downstream (left to right) transect profiles. G stands for glacier runoff, LM - Lindemanselv and Palnatookeelv rivers and AC - Aucellaelv River.

### 6.2.2.3 Relationship between TOC, BHPs and GDGTs

The relationship between TOC (%) and absolute concentration of organic biomarkers ( $\mu\text{g/g}$  sediment) was plotted in order to see if organic biomarkers were related to OC content (Figure 6.10). There was a positive relationship between TOC (%) and total BHPs ( $\mu\text{g/g}$  sediment) (Figure 6.10a) with an  $R^2$  value of 0.87 and Pearson correlation coefficient of 0.93. Meanwhile, the relationship between TOC (%) and total brGDGTs ( $\mu\text{g/g}$  sediment) was weak (Figure 6.10b) with an  $R^2$  value of 0.28 and a moderate Pearson correlation coefficient of 0.53. Similarly, there was a weak correlation between total BHPs ( $\mu\text{g/g}$  sediment) and total brGDGTs ( $\mu\text{g/g}$  sediment) (Figure 6.10c) ( $R^2=0.32$ , Pearson correlation=0.56).

6 Soil development at Zackenberg valley, Greenland

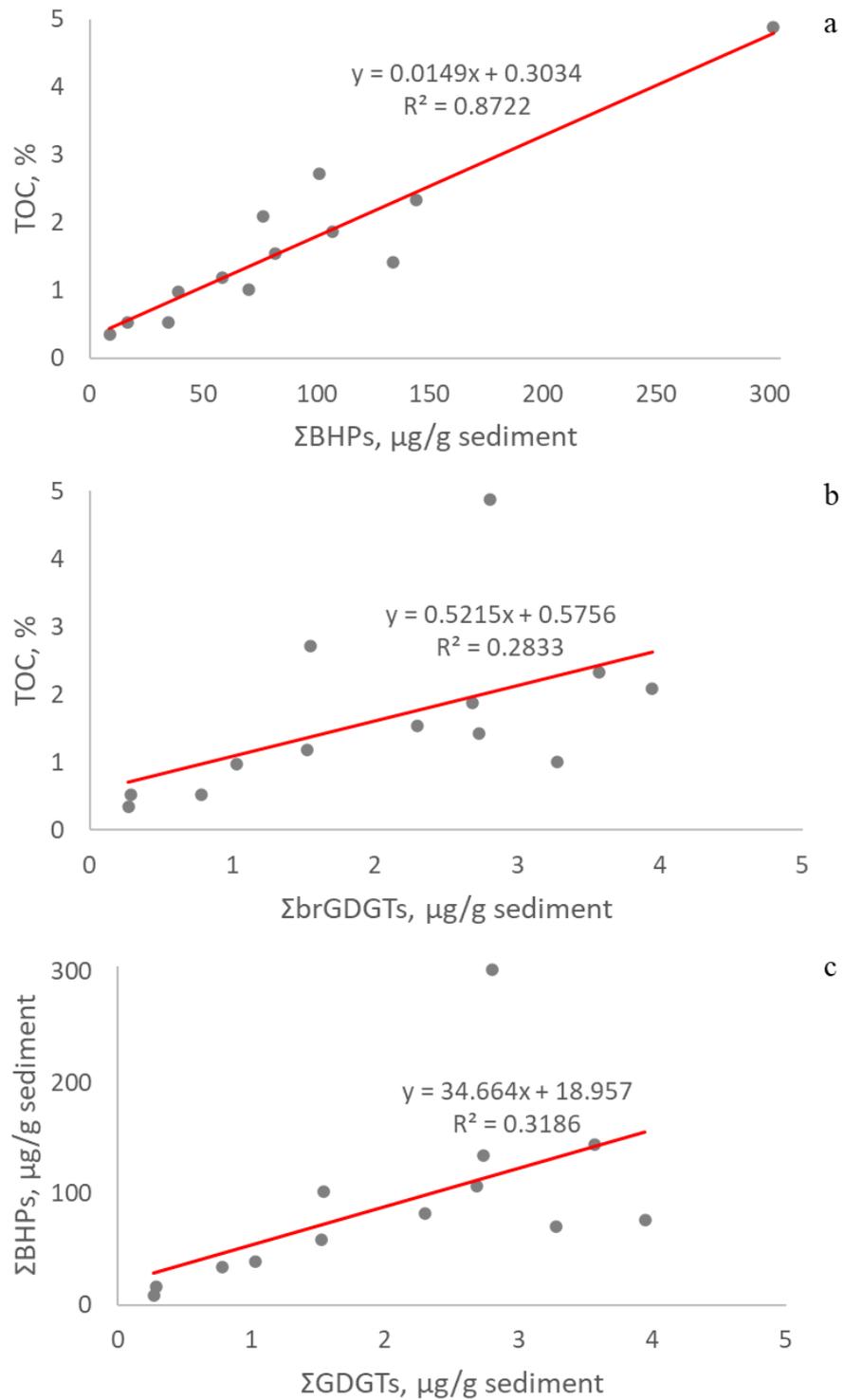


Figure 6.10. Relationship between total organic carbon (TOC) (%) and (a) total BHPs ( $\mu\text{g/g}$  sediment); (b) total brGDGTs ( $\mu\text{g/g}$  sediment), and (c) between total BHPs and total brGDGTs ( $\mu\text{g/g}$  sediment) across all samples collected along the Zackenberg valley, Greenland.

## 6.2.3 Microbial analysis

### 6.2.3.1 Sample selection and DNA concentration

A total of 35 soil and sediment samples from the Zackenberg valley were also investigated for microbial community structure. The samples consisted of 11 sediment and 24 soils, with at least one replicate of each sample demonstrated on a map in Figure 6.2. DNA concentrations of selected samples along the valley ranged from BDL to 61.7 ng/ml (Appendix 18). Generally, samples selected for analysis of microbial community had sufficient DNA and produced a visible band in PCR. Overall, soil samples had higher DNA concentration ( $29.4 \pm 13.1$  ng/ml) compared to sediment samples ( $5.2 \pm 9.9$  ng/ml) and had statistically significant difference by the sample type (Welch's t-test, p-value < 0.05).

### 6.2.3.2 Chemical characterisation of soils and sediments investigated for microbial community structure

In order to see the differences in chemical composition between soil and sediment samples, PCA was conducted on samples selected for further microbial community analysis (Figure 6.11). PC1 explained 58.9% of total variance, while PC2 explained 16.1% of the variance. PC1 had the strongest positive correlation with Co, Al, Cd, Fe, Cr, Zn, Ni, K and Mn, and less strong positive correlations with Pb, Na, Cu and Mg. PC2 had the strongest positive correlations with P, Ca and Mg, and strong negative correlations with S and As (Figure 6.11). Similar to Figure 6.1, which displays PCA plot constructed using mean values of chemical elements of all samples from the Zackenberg valley, TC, TOC and N had low correlation with PC1 and PC2.

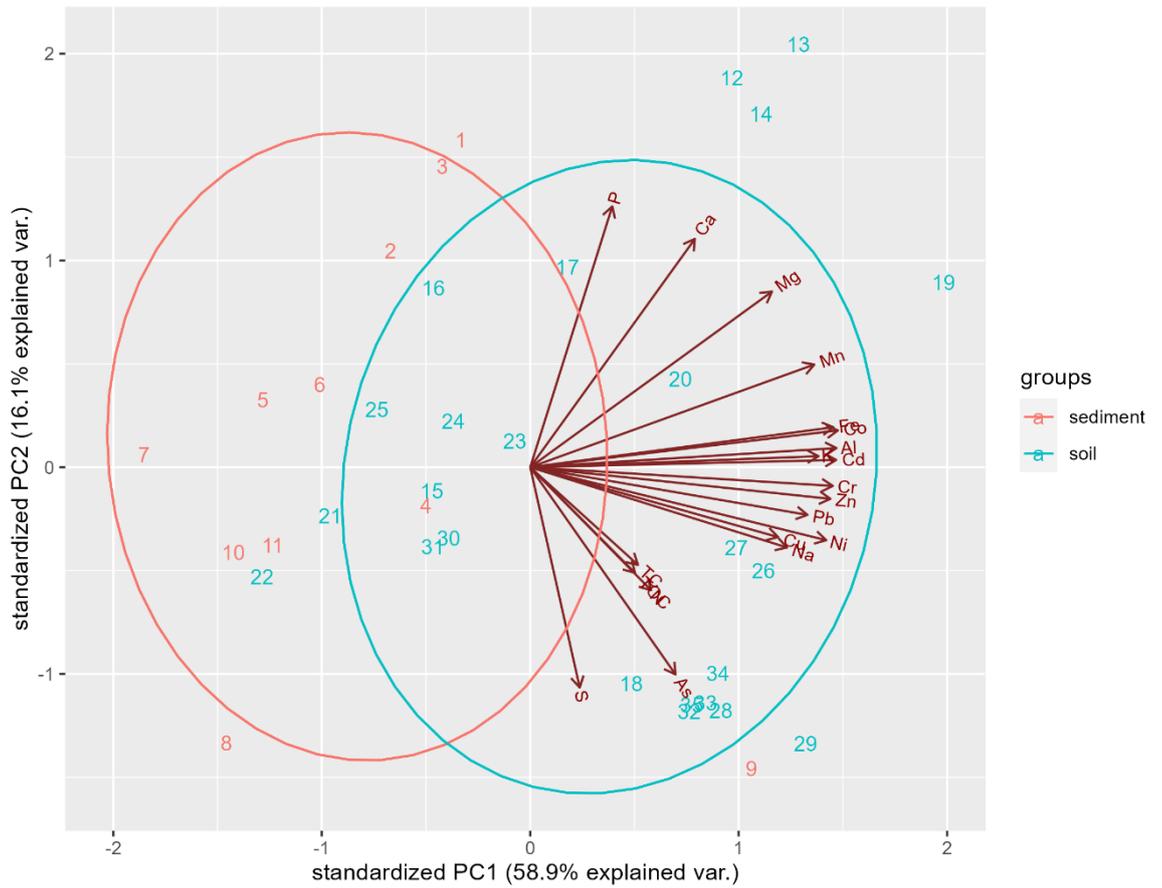


Figure 6.11. PCA ordination plot illustrating the discrimination between samples investigated for microbial community structure from the Zackenberg valley, Greenland according to chemical properties by the sample type. The input data was standardised to have zero mean and variance one. The ellipses represent the core area added by confidence interval of 68%. Sample codes are given in Table 6.3.

Table 6.3. Codes of selected sample names from Zackenberg valley, Greenland shown in Figure 6.11 and Figure 6.12.

Code	Sample	Type	Code	Sample	Type
1	G-1408_3	sediment	19	SS-LI-1408_2	soil
2	SS-D-0508_1	sediment	20	SS-LI-1408_3	soil
3	SS-D-0508_2	sediment	21	SS-L-1008_3	soil
4	SS-L-1008_2	sediment	22	SS-L-1308_1	soil
5	SS-L-1308_3	sediment	23	SS-O-1008_1	soil
6	ZR-SS-1508_2	sediment	24	SS-O-1008_3	soil
7	ZR-BL-0108_1	sediment	25	ZR-SS-1508_2	soil
8	ZR-LM-1608_1	sediment	26	ZR-BL-0108_1	soil
9	ZR-AC-1608_1	sediment	27	ZR-BL-0108_3	soil
10	ZR-B-0808_2	sediment	28	ZR-LM-1608_2	soil
11	ZR-S-0208_3	sediment	29	ZR-LM-1608_3	soil
12	G-1408_1	soil	30	ZR-AC-1608_1	soil
13	G-1408_2	soil	31	ZR-AC-1608_3	soil

14	G-1408_3	soil	32	ZR-B-0808_1	soil
15	AHR-0508_1	soil	33	ZR-B-0808_2	soil
16	SS-D-0508_1	soil	34	ZR-S-0208_1	soil
17	SS-D-0508_2	soil	35	ZR-S-0208_2	soil
18	SS-LI-1408_1	soil			

PERMANOVA analysis showed a statistically significant difference between the subset of sediment and soil samples ( $p$ -value < 0.05). PCA indicated that sediment samples were mostly grouped in the left side of the plot, while soil samples were clustered to the right side. Sediment sample 9 (see Table 6.3 for the sample names) was located in the bottom right part of the plot, close to the soil cluster, indicating similarity in chemical composition with soils. Soil samples 21 and 22 (see Table 6.3 for the sample names) were located to the left of soil samples, indicative of chemical properties similar to soil samples.

### 6.2.3.3 Microbial community structure

#### 6.2.3.3.1 NMDS

Further, an NMDS ordination plot of family-level bacterial communities (Figure 6.12a) demonstrated that soil samples were clustered to the right side of the plot, while sediment samples 1, 7, 8, 9, 10 and 11 (see Table 6.3 for sample names) were scattered, extending to the left. These sediment samples were collected along either the glacier meltwater stream or Zackenberg river, while sediment samples clustered with soil samples (Figure 6.12b) were collected along the Store Sø Lake. PERMANOVA analysis also showed that there was a statistically significant difference between family-level bacterial communities of soil and sediment samples ( $p$ -value < 0.05).

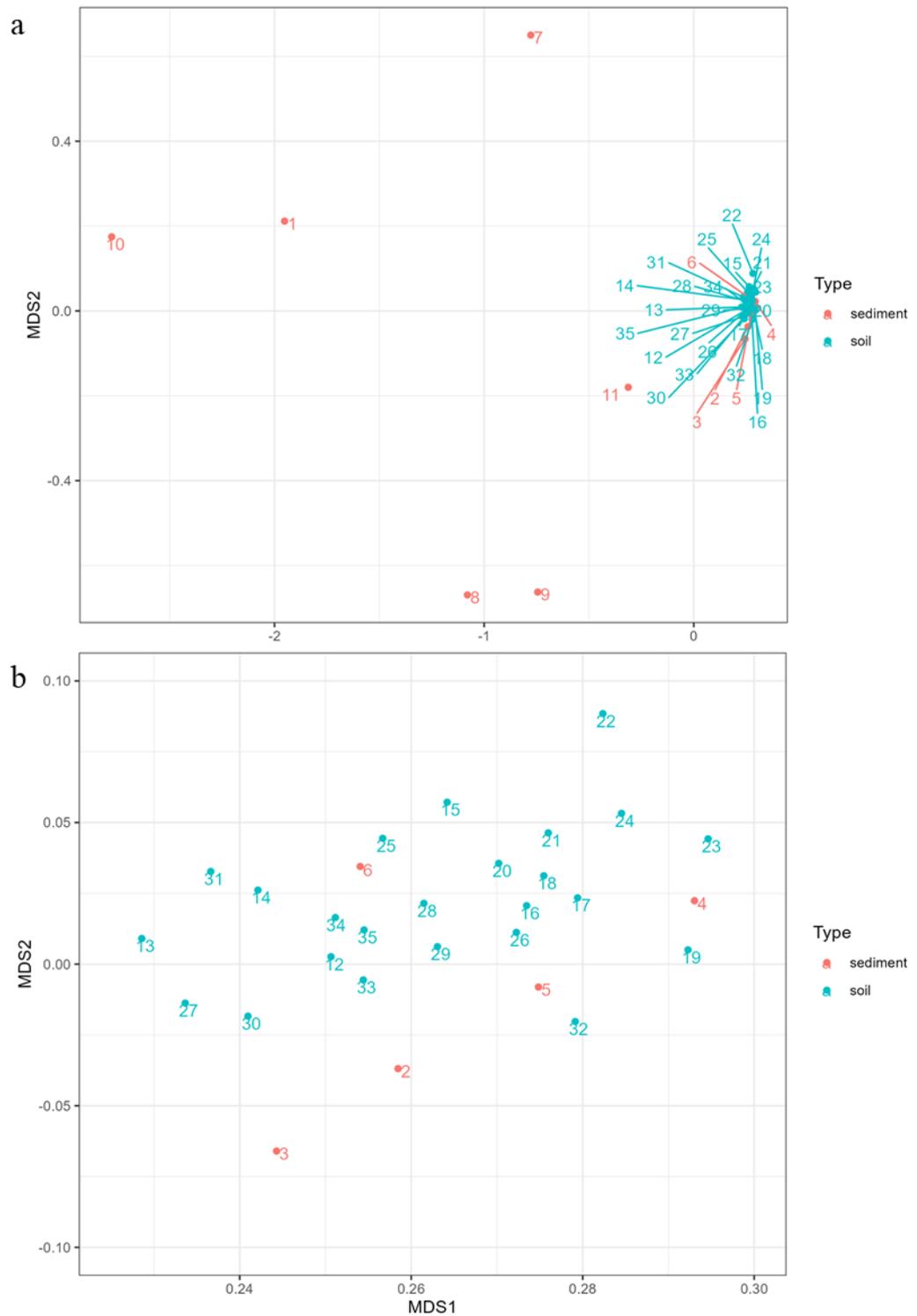


Figure 6.12. (a) Full scale and (b) zoomed-in two-dimensional NMDS plots of bacterial communities in samples from the Zackenberg valley, Greenland by sample type. When the data values were larger than common abundance class scales, Wisconsin double standardisation was performed; when values looked very large, sqrt transformation was also performed; and distance matrix based on Bray–Curtis dissimilarity was obtained. Sample codes are given in Table 6.3.

### 6.2.3.3.2 Alpha diversity

Shannon–Weiner diversity index and Chao1 species richness estimate values were plotted to evaluate the microbial alpha diversity in subset of samples from the Zackenberg valley (Figure 6.13). Both the Shannon–Weiner diversity index and Chao1 species richness estimate indicated that sediment samples collected along the meltwater stream and the Zackenberg river had lower bacterial diversity (G-1408, ZR-BL-0108, ZR-LM-1608, ZR-AC-1608, ZR-B-0808, ZR-S-0208), while sediment samples collected along the lake and soil samples generally had higher bacterial diversity. Interestingly, sediment samples SS-D-0508 and SS-L-1308 had higher diversity compared to soil samples.

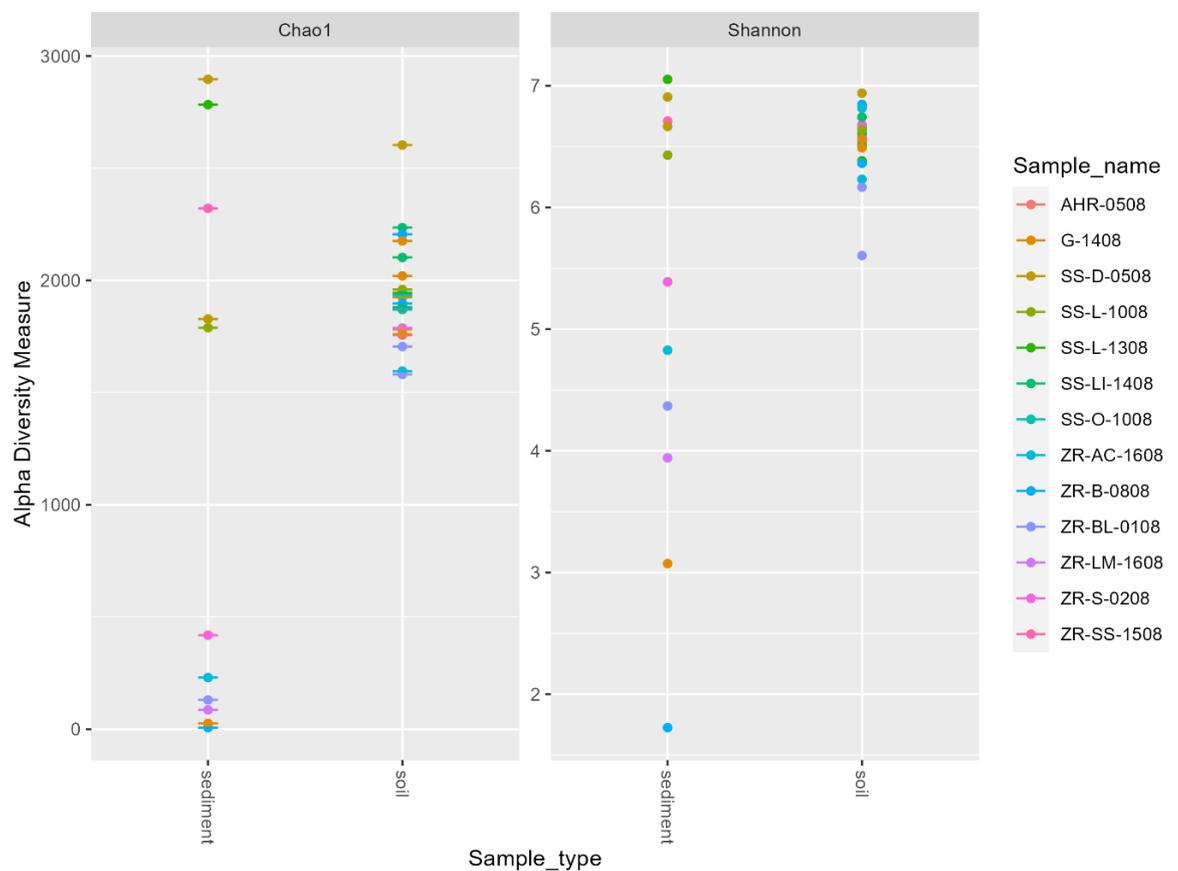


Figure 6.13. Alpha diversity (Shannon) and richness (Chao1) variation in soil and sediment samples from Zackenberg valley in Greenland.

### 6.2.3.3.3 Phylum-level community composition

Bacterial 16S rRNA gene derived phyla distribution in sediment and soil samples along the Zackenberg valley are presented in Figure 6.14a and Figure 6.15a, respectively. Acidobacteria ( $19.6 \pm 8.0\%$ ), Chloroflexi ( $16.3 \pm 6.2\%$ ), Proteobacteria ( $15.4 \pm 11.0\%$ ),

Verrucomicrobia ( $14.2\pm 5.5\%$ ) and Actinobacteria ( $11.8\pm 4.4\%$ ) were the most abundant phyla in all samples collected in Greenland. As expected from the PCA and NMDS plots above, there was some difference in phylum distribution in sediment and soil samples in Greenland. Proteobacteria ( $24.8\pm 15.7\%$ ), Chloroflexi ( $14.7\pm 9.6\%$ ), Actinobacteria ( $12.2\pm 6.5\%$ ) and Acidobacteria ( $12.0\pm 6.8\%$ ) were the most abundant phyla in sediment samples, while Acidobacteria ( $23.2\pm 5.7\%$ ), Chloroflexi ( $17.1\pm 3.8\%$ ), Verrucomicrobia ( $16.0\pm 3.0\%$ ) and Actinobacteria ( $11.7\pm 3.2\%$ ) were the most abundant in soil samples collected from the Zackenberg valley. Moreover, PERMANOVA analysis also showed a statistically significant difference in phylum distribution ( $p$ -value  $< 0.05$ ) between soil and sediment samples.

Similar to sediments from Sweden, sediment samples from Greenland also had Bacteroidetes ( $7.0\pm 8.0\%$ ), however, unlike samples from Sweden, they also had Firmicutes ( $3.8\pm 7.4\%$ ). Compared to other sediments, samples G-1408 and ZR-B-0808 had the highest relative abundance of these phyla. As expected, sediment samples along the lake had similar relative abundances (Figure 6.14a). Meanwhile, there was not much phylum-level bacterial variation in soil samples from Greenland (Figure 6.15a).

#### **6.2.3.3.4 Family-level community composition**

Bacterial family-level distribution of sediment and soil samples along the Zackenberg downstream transect are demonstrated in Figure 6.14b and Figure 6.15b. The dominant family-level bacteria at the Zackenberg valley were Comamonadaceae (Proteobacteria) ( $8.3\pm 8.8\%$ ), Chthoniobacteraceae (Verrucomicrobia) ( $8.2\pm 7.5\%$ ), unclassified at family-level Ellin6529 (Chloroflexi) ( $4.4\pm 3.3\%$ ) and unclassified at family-level RB41 (Acidobacteria) ( $3.8\pm 2.2\%$ ) in sediment samples and Chthoniobacteraceae (Verrucomicrobia) ( $14.4\pm 3.2\%$ ), unclassified at family-level RB41 (Acidobacteria) ( $9.3\pm 5.5\%$ ), unclassified at family-level iii1-15 (Acidobacteria) ( $4.7\pm 2.4\%$ ) and unclassified at family-level Ellin6529 (Chloroflexi) ( $4.1\pm 2.3\%$ ) in soil samples. The “other” category includes family-level bacteria that had less than 0.5% relative abundance in all samples collected in Greenland.

Similar to the phylum-level bacteria, family-level bacteria in sediment samples collected along the Store Sø Lake had similar microbial community composition (Figure 6.14b). Sediments from locations along the meltwater stream and river had different bacterial

composition, with samples G-1408 and ZR-B-0808 having especially distinctive differences from the other sediment samples. Meanwhile, soil samples had less variation in family-level microbial community composition (Figure 6.15b).

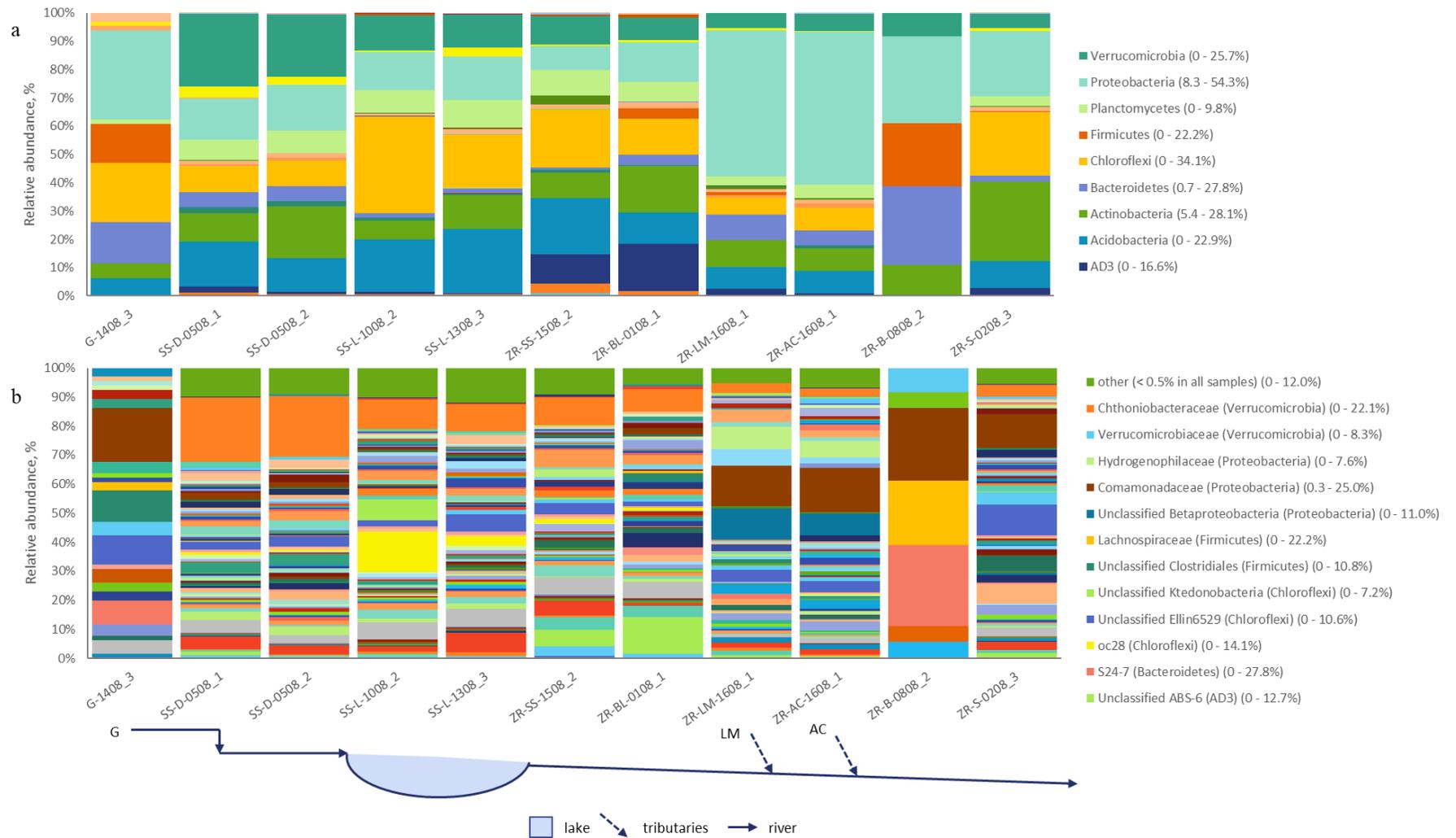


Figure 6.14. Relative abundance of bacterial taxa: (a) all assigned taxa at phylum level and (b) all assigned taxa shown to the highest resolution possible down to the family level where possible (phylum level given in parentheses) for each sediment sample along the Zackenberg valley downstream transect in Greenland. Selected taxa of high abundance given in parentheses. Charts at the bottom of the graph show downstream (left to right) transect profiles.

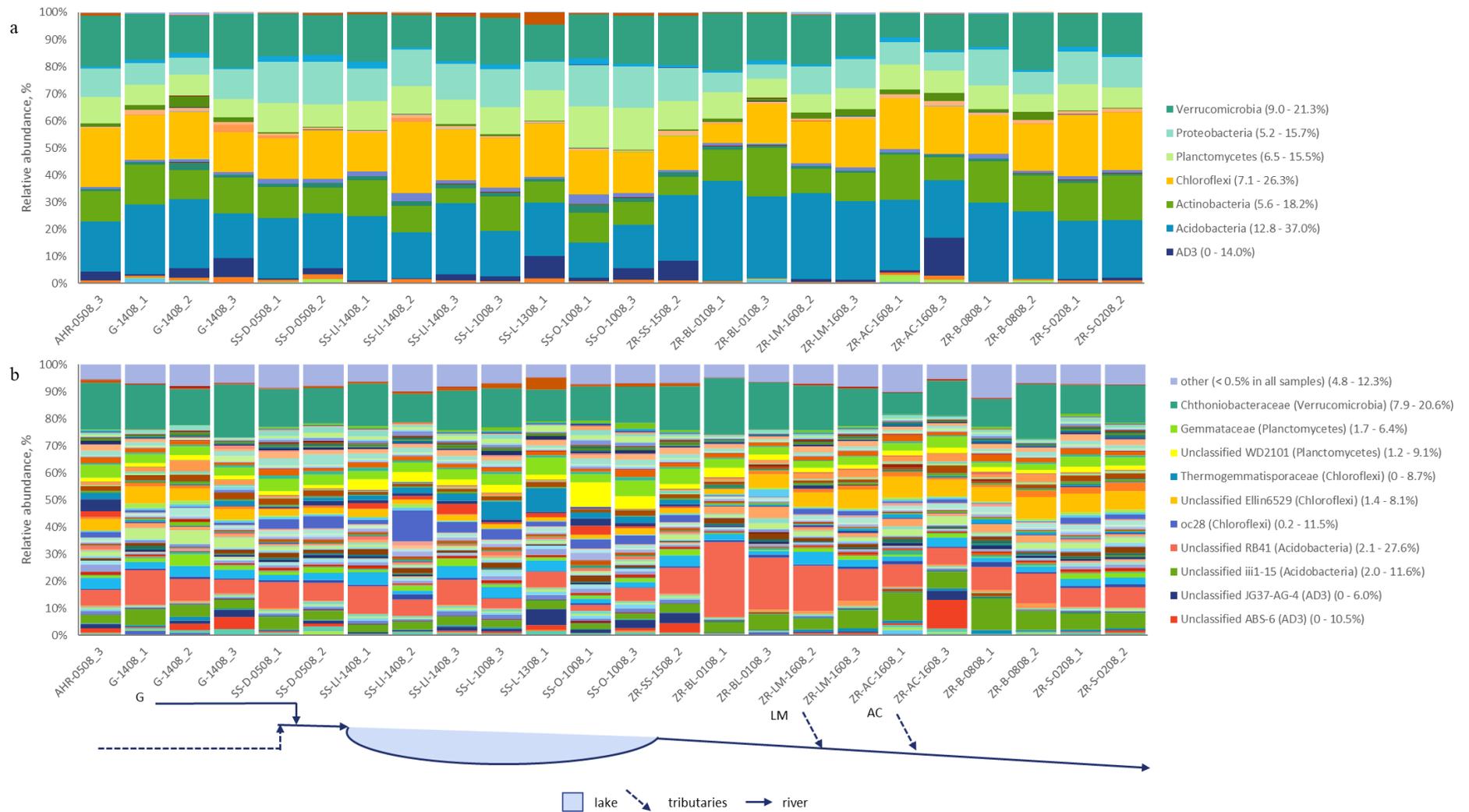


Figure 6.15. Relative abundance of bacterial taxa: (a) all assigned taxa at phylum level and (b) all assigned taxa shown to the highest resolution possible down to the family level where possible (phylum level given in parentheses) for each soil sample along the Zackenberg valley downstream transect in Greenland. Selected taxa of high abundance given in parentheses. Charts at the bottom of the graph show downstream (left to right) transect profiles.

## 6.3 Discussion

The aim of this chapter was to understand catchment development along the Zackenberg valley transect by investigating environmental chemistry (TOC, TN, and metals), organic biomarkers (BHPs and GDGTs), and microbial community composition. A subset of soil samples was investigated for organic biomarkers, and a subset of both soil and sediment samples were analysed for bacterial community structure. It was expected that OC would accumulate along the transect, while organic biomarkers and microbial communities would be homogeneous in this older catchment that has been ice free since the Early Holocene (ca. 10.5 ka) (Garcia-Oteyza et al., 2022). Moreover, it was expected that sediment samples would have lower TOC content, and that there would be a difference between microbial communities of soils and sediments.

### 6.3.1 TOC, TN, and C/N in Greenland compared to other catchments

The mean TOC concentration of replicate samples at a given location collected from the Zackenberg valley ranged from  $0.01 \pm 0.01$  to  $4.88 \pm 3.56\%$ . This value is higher than that recorded in Sweden ( $0-1.08 \pm 0.09\%$ ), suggesting that it is accumulated more soil derived OM than the Tarfala valley catchment. The TOC values in Greenland are comparable to the findings in Iceland ( $0.01-5.24 \pm 2.68\%$ ). Earlier studies also found similar values (Svalbard, 0-96 years after the glacier retreat,  $0.69-3.81\%$  (Wietrzyk et al., 2018); Switzerland, 15-700 years of glacier retreat,  $0.15-7.97\%$  (Prietzl et al., 2013)). This suggests that OC content may reach a certain value and stabilize in developing and developed deglaciated catchments. In line with this, Wietrzyk et al. (2018) suggested that TOC concentration is a function of time of exposure after deglaciation.

Similar to samples from Sweden and Iceland, soil samples from Greenland had higher TOC content ( $1.62 \pm 1.54\%$ ) compared to sediment samples ( $0.25 \pm 0.50\%$ ). This finding is also in line with that in Guzella et al. (2016), reporting higher OC values in soils ( $0.4-7.6\%$ ) compared to sediments ( $0.2-1.6\%$ ) from the Mt. Everest region. This strengthens the observation of proglacial soils being more carbon rich compared to sediments.

There was no overall trend of OC build-up along the downstream Zackenberg valley downstream transect (Figure 6.3). On the contrary, OC accumulation over time along glacier forelands in Iceland was observed. Similarly, Wietrzyk et al. (2018) found that TOC concentration increased in older soils. In both cases, soils were younger than the ones from Greenland: soils in Iceland varied from post LIA, LIA and up to 5000-6000 years (Virikisjökull, Guðmundsson, 1998), and soils in Svalbard ranged from newly exposed soils to 100-year-old soils (Wietrzyk et al., 2018). Meanwhile, Zackenberg valley has been ice free since the Early Holocene (ca. 10.5 ka) (Garcia-Oteyza et al., 2022). Therefore, this suggests that no change in TOC signals was seen due to the stabilisation of soils in Greenland over time.

TN values in samples from Greenland ranged from 0 to 0.40% and were similar to findings in glacier forelands from Iceland (0-0.66%), Svalbard (0.04-0.15%, Wietrzyk et al., 2018) and Switzerland (0.02-0.6%, Prietzel et al., 2013). TOC and TN values in Greenlandic samples were highly correlated ( $R^2=0.92$ , Pearson correlation=0.96), samples with high TOC concentration also having higher TN values. This suggests that nitrogen in samples from Greenland has an organic nature (Stein and Rack, 1995 in Huon et al., 2002) and can be transformed, increasing its availability to vegetation (Brust, 2019). This is in line with the expectations that soils at the Zackenberg valley are rich in organic matter.

As for the C/N ratio, it was lower in sediment samples ( $6.77\pm 5.82$ ) than in soils ( $14.13\pm 4.66$ ), which is higher than the ratios detected in Sweden ( $3.19\pm 3.80$  for sediments and  $7.25\pm 7.82$  for soils) and Iceland ( $1.20\pm 1.82$  for sediments and  $10.36\pm 8.54$  for soils). Moreover, C/N ratios in Greenland are similar to the findings in Wietrzyk et al. (2018), reporting values of 17-25 in glacier forelands in Svalbard and Prietzel et al. (2013), who reported C/N ratios of 8 to 22 in soils from a glacier foreland in Switzerland. C/N has been used to elucidate the nature of organic matter. For instance, Yu et al. (2010) reported that C/N of  $12.7\pm 2.3$  was typical of freshwater environments, while the ratio can vary from  $8.9\pm 1.1$  to  $17.9\pm 3.6$  in soils. Based on this, it seems that C/N of sediment samples from the Zackenberg valley is closest to characterization of freshwater environments, while C/N detected in soil samples is generally descriptive of soils.

### 6.3.2 No variation in metal concentration

Principal component analysis on chemical composition of samples from the Zackenberg valley, as well as PERMANOVA analysis showed that there was a difference between soil and sediment samples. This is in line with findings in Colombo et al. (2020), reporting that OC and elements concentrations were different in proglacial soil and sediment samples in the Italian Alps. This strengthens the hypothesis that soils and sediments have different chemical properties.

The most abundant major metals in Greenland were Fe ( $4857 \pm 1862$  mg/kg), Al ( $2517 \pm 1196$  mg/kg), Ca ( $1348 \pm 441$  mg/kg), and Mg ( $1198 \pm 558$  mg/kg). This is in line with what was observed in Sweden and Iceland, where Fe, Al, Ca and Mg were also the most abundant metals. Moreover, Bing et al. (2016) reported that these four elements were the most abundant metals in glacier foreland soils at the Tibetan Plateau. Similarly, according to Venkatachalam et al. (2021), Fe, Al, and Ca were abundant in a glacier foreland in Svalbard.

Concentrations of these metals in samples from Greenland were slightly lower than that in Sweden, and much lower than the values detected in Iceland (Appendix 14). Moreover, metal concentrations in Greenland were much lower compared to the glacier forelands in Svalbard (Venkatachalam et al., 2021) and Tibetan Plateau (Bing et al., 2016) (Table 6.4). These comparisons show that sediment and soil samples from Greenland are low in metals, which suggests that TOC is more important factor in the development of the catchment. Moreover, even though major metals detected in Greenland and other catchments are similar, their concentrations vary from country to country, depending on geology of the catchment.

Table 6.4. Metal concentrations from glacier forelands in literature.

Metals	Range			
	from		to	
Fe	8800 <sup>1</sup>	$\pm 2400^1$	28400 <sup>1</sup>	$\pm 6300^1$
	25547 <sup>2</sup>		53774 <sup>2</sup>	
Al	15400 <sup>1</sup>	$\pm 4800^1$	50400 <sup>1</sup>	$\pm 7600^1$
	12561 <sup>2</sup>		39008 <sup>2</sup>	
Ca	19100 <sup>1</sup>	$\pm 3600^1$	33000 <sup>1</sup>	$\pm 2200^1$
	537 <sup>2</sup>		2353 <sup>2</sup>	
Mg	4000 <sup>1</sup>	$\pm 600^1$	14600 <sup>1</sup>	$\pm 3100^1$

References: <sup>1</sup>Bing et al., 2016; <sup>2</sup>Venkatachalam et al., 2021.

As expected, no particular trend was observed in distribution of metal concentrations along the transect in Greenland. This is similar to findings in Sweden and Iceland, and findings in Venkatachalam et al. (2021), reporting no particular pattern in metal distribution along 1.8 km long glacier foreland chronosequence in Svalbard that has been deglaciated for up to 1900 years. This suggests that metals do not accumulate along transects over time and the variation in metal concentrations depend on the geology of the catchment. Moreover, as metals affect the bacterial diversity of the catchment, some variation in microbial community diversity in samples from Greenland was expected.

### 6.3.3 Organic biomarkers

Organic biomarker analysis revealed presence of BHPs and brGDGTs in all samples from the Zackenberg valley. Carbon concentration affects presence of organic biomarkers (Höfle et al., 2015), therefore, it was expected that TOC content affected biomarker distribution in soil samples from Greenland. As predicted, linear regression analysis showed that there was a strong positive relationship between TOC and BHPs (Figure 6.10a). However, there was a weak correlation between TOC and GDGT concentrations (Figure 6.10b). Concentration of total GDGTs was 19 to 111 times less than total BHP concentration, suggesting that OC has higher BHP content compared to GDGTs.

The mean relative concentration of total BHPs was  $5230 \pm 1876$   $\mu\text{g/g}$  TOC, which is higher than that in Iceland ( $2469 \pm 1404$   $\mu\text{g/g}$  TOC) and slightly lower than the findings in Sweden ( $6727 \pm 5250$   $\mu\text{g/g}$  TOC), values observed in Sweden being most likely due to low OC content. On the contrary, the mean absolute concentration of total BHPs was  $90 \pm 76$   $\mu\text{g/g}$  sediment, which is higher than values in Sweden ( $12 \pm 15$   $\mu\text{g/g}$  sediment) and Iceland ( $25 \pm 23$   $\mu\text{g/g}$  sediment). This suggests that biomarker production is higher in Greenland compared to Sweden and Iceland. Moreover, the mean relative concentration is much higher than findings in other studies. For example, according to Doğrul Selver et al. (2015), in river mouth sediments from Siberia, total BHP concentration was in the range of 72 to 489  $\mu\text{g/g}$  TOC in estuary and open bay sediments, while it ranged from 138 to 281  $\mu\text{g/g}$  TOC in river mouth sediments from Siberia (Doğrul Selver et al., 2015). Higher values were recorded in soil samples from Northern England (Cooke et al., 2008), ranging from 53 to 1454  $\mu\text{g/g}$  TOC,

and in Siberian permafrost soils, ranging from 84 to 1111 µg/g TOC (Höfle et al., 2015). These difference in values could be due to higher OC content in other studies: 8-13.6% in river mouth samples from Siberia (Vonk et al., 2010 in Doğrul Selver et al., 2015), 1.7-10.7% in soil samples from Northern England (Cooke et al., 2008) and 1.5-28.4% in Siberian permafrost soils (Höfle et al., 2015) compared to 0.01±0.01 - 4.88±3.56% in Greenland. In line with this, the absolute concentration of BHPs was 4-121 µg/g dry soil in soils from Northern England (Cooke et al., 2008), which is higher than values in Greenland.

### **6.3.3.1 Composition and distribution of soil specific organic biomarkers in Greenland compared to other catchments**

#### **6.3.3.1.1 Soil marker BHPs**

Individual BHPs can be used as biomarkers of past life (Killops and Killops, 2005). This study particularly focused on soil marker BHPs, as they are abundant in soils (Cooke et al., 2008; Doğrul Selver et al., 2012; Höfle et al., 2015), and are used to understand accumulation of soil derived OC along glacier forelands. No such studies have been performed in the past. However, soil marker BHPs were used to trace terrestrial OC along land to sea/ocean transects (e.g., Bischoff et al., 2016; De Jonge et al., 2015; Doğrul Selver et al., 2012; Doğrul Selver et al., 2015). Soil marker BHPs were detected in all samples from the Zackenberg valley and contributed to 8-30% of total BHPs. Fractional contribution of soil marker BHPs in soils from Greenland is similar to Svínafellsjökull (16-28% of total BHPs) and some estuary sediment samples in the Arctic, such as the Mackenzie River (15% of total BHPs) and Indigirka River systems (37% of total BHPs) (Cooke et al., 2009). Relative abundance of soil marker BHPs in Greenland was higher than that in Sweden and Virkisjökull, Kviarjökull, and Fjallsjökull glaciers in Iceland. However, despite the high total BHPs concentration, fractional abundance of soil-specific BHPs was lower than soils from Northern England (20-48%, Cooke et al., 2008) and permafrost regions in the Arctic. For instance, Rethemeyer et al. (2010) reported that soil marker BHPs made up 20-73% of total BHPs in samples from the Bayelva River catchment, while Doğrul Selver et al. (2015) reported that this value went up to 82% in Siberia. These observations suggest that abundance of soil marker BHPs at the Zackenberg valley is comparable to some Arctic catchments and glacier forelands, while much lower than that in the Arctic permafrost regions.

A function of soil marker BHPs,  $R'_{\text{soil}}$  index, ranged from 0.11 to 0.37 across the soil samples from the Zackenberg valley. These values are similar to findings in Sweden (0-0.34) and Svínafellsjökull glacier in Iceland (0.27-0.35), and findings in Doğrul Selver et al. (2012), reporting  $R'_{\text{soil}}$  index in the range of 0.08-0.30 in aquatic sediments in Northern Sweden. Meanwhile, similar to soil marker BHPs, permafrost soils in the Arctic have higher  $R'_{\text{soil}}$  index. For instance, it was 0.57 in Kolyma River in Northeast Siberia (Doğrul Selver et al., 2015) and 0.16-0.62 in river sediment samples from Yenisei River in Siberia (De Jonge et al., 2016). Similar to the conclusion based on soil marker BHP abundance, the  $R'_{\text{soil}}$  index in samples from Greenland suggests that accumulation of soil derived OC in this catchment is comparable to some catchments and glacier forelands in the Arctic but is much lower than organic rich permafrost.

#### **6.3.3.1.2 BrGDGTs**

Another organic biomarker, produced in soils and descriptive of terrestrial OC (Hopmans et al., 2004; Weijers et al., 2006; Doğrul Selver et al., 2012), brGDGTs, was identified in all samples from the Zackenberg valley, while some samples even contained some crenarchaeol, characteristic of aquatic/fluvial environments (Zell et al., 2013). Relative concentration of total GDGTs in Greenland ( $144 \pm 73 \mu\text{g/g TOC}$ ) are comparable to the values recorded at Svínafellsjökull ( $154 \pm 69 \mu\text{g/g TOC}$ ) and Virkisjökull ( $135 \pm 113 \mu\text{g/g TOC}$ ) in Iceland, lower than the values in Sweden ( $403 \pm 163 \mu\text{g/g TOC}$ ) but higher than that at Kviarjökull ( $96 \pm 53 \mu\text{g/g TOC}$ ) and Fjallsjökull ( $59 \pm 27 \mu\text{g/g TOC}$ ) in Iceland. Meanwhile, absolute concentration of total GDGTs were higher in Greenland ( $2.19 \pm 1.25 \mu\text{g/g sediment}$ ) compared to values in Sweden ( $0.77 \pm 1.19 \mu\text{g/g sediment}$ ) and Iceland ( $1.52 \pm 2.14 \mu\text{g/g sediment}$ ). Absolute concentrations of GDGTs detected in this study site were much higher than that recorded in other catchments. For instance, in Northern Sweden, the concentration of total GDGTs was 0.26-0.91  $\mu\text{g/g TOC}$  (Doğrul Selver et al., 2012), at the Kolyma River, the concentration ranged from 3.2 to 4.5  $\mu\text{g/g TOC}$  (Doğrul Selver et al., 2015) and at the Yenisei River, the range was 0.1-35  $\mu\text{g/g POC}$  (De Jonge et al., 2015). Similar to BHPs, this could be due to the relatively low OC content in samples from Greenland ( $0.01 \pm 0.01 - 4.88 \pm 3.56\%$ ) compared to other sites (8-13.6% in river mouth samples from Siberia, Vonk et al., 2010 in Doğrul Selver et al., 2015).

Concentrations of brGDGTs are used as a proxy to study past temperatures (Weijers et al., 2007) and soil pH (Peterse et al., 2012). GDGT proxy values in this chapter are comparable to the findings in literature. For instance, GDGT proxy pH ranged from 4.6 to 7, close to the values reported in Elberling et al. (2004), which varied between 5.0-7.2. GDGT proxy MAT ranged from -2.8 and 5.8 °C, while Hansen et al. (2017) reported annual air temperature of 8.8 °C. This suggests that MAT is lower than the actual annual mean air temperature. On the contrary, De Jonge et al. (2016) reported that MAT in Yenisei watershed soils is above the annual mean air temperature. This suggests that while GDGT proxy pH can be used to predict the soil pH, GDGT proxy MAT is not as accurate and should be considered with care, similar to observations from Sweden and Iceland in previous chapters. Since the proxy has been previously used to access air temperature in other settings, the difference in MAT values here could be due to a relative lack of GDGTs in this region, as well as in Sweden and Iceland.

### 6.3.3.2 Soil development in Greenland

There was no clear pattern of accumulation of OC and organic biomarkers (BHPs and GDGTs) along the Zackenberg valley downstream transect. Based on the observations of TOC downstream distribution, it was concluded that soils in Greenland have stabilised over time (see section 6.3.1). As discussed in previous chapters, soil marker BHPs and their proxies ( $R_{\text{soil}}$  and  $R'_{\text{soil}}$  indexes) were not previously used to investigate accumulation of soil derived OC in deglaciated forelands but rather to trace terrestrial OC along land to sea/ocean transects (e.g., Bischoff et al., 2016; Cooke et al., 2008; Doğrul Selver et al., 2012; Doğrul Selver et al., 2015; Spencer-Jones et al., 2015; Zhu et al., 2011). The  $R'_{\text{soil}}$  index, which was previously used to trace terrestrial OC in other Arctic studies (Doğrul Selver et al., 2012; Doğrul Selver et al., 2015) and to investigate accumulation of soil derived OC in Sweden and Iceland in this study, did not increase along the Zackenberg valley downstream transect. Moreover, there were some discrepancies between OC content and the  $R'_{\text{soil}}$  signal. For instance, TOC was low at the most upstream glacier meltwater soil sample (G-1408), while the  $R'_{\text{soil}}$  index was high. TOC was somewhat stable along the lake transect, while the  $R'_{\text{soil}}$  index indicated that there was a downward trend along the transect of the Sjø Store Lake. This could be due to the masking of soil marker BHP signals by other compounds of OM. Similar to the observations in Iceland, it was seen that BHPs were

dominated by soil marker BHPs along the downstream transect initially but over time abundance of soil marker BHPs decreased. This suggests that the initial soil development plateaus over time and other BHP compound groups, such as BHT, methylated BHT, aminotriol, BHT glucosamine and BHT cyclitol ether might overtake soil marker BHPs, though this does not explain high abundance of soil marker BHPs in sample ZR-BL-0108.

Likewise, brGDGTs were not previously used to study terrestrial soil development. BrGDGTs were used as soil-tracing biomarkers along land to sea/ocean transects (Bischoff et al., 2016; De Jonge et al., 2015; Doğrul Selver et al., 2012; Doğrul Selver et al., 2015). In this chapter, similar to the previous observations in Sweden and Iceland, brGDGTs did not follow a particular pattern of distribution along the Zackenberg valley transect. This suggests that the time of exposure after deglaciation does not affect neither production nor preservation of brGDGTs.

In order to understand the accumulation of soil derived OC in deglaciated catchments it is important to investigate concentration and distribution of both BHPs and GDGTs, as they are produced by different organisms and behave differently (Kim et al., 2011). Moreover, Doğrul Selver et al. (2012) and Doğrul Selver et al. (2015) concluded that it is important to use proxies based on both BHPs and GDGTs in order to trace the transport of terrestrial OC, which might also be applicable to this study.

### 6.3.3.3 Source and nature of organic biomarkers

Similar to Sweden and Iceland, adenosylhopane (3-11% of total BHPs) and adenosylhopane type 3 (2-7% of total BHPs) were the most abundant soil marker BHPs. According to Killips and Killips (2005), individual BHPs are indicative of certain environments. Table 4.4 shows the source organisms of the most abundant organic biomarkers in samples in this study and the environments where they can usually be detected. The most abundant individual BHPs were BHT and methylated BHT, their sum contributing to 56-75% of total BHPs, followed by aminotriol, BHT cyclitol ether and BHT glucosamine. It is worth mentioning that some of the bacteria produce more than one BHP compound. For instance, bacteria (purple non-sulfur bacteria, *Nitrosomonas europaea*, *Bradyrhizobium japonicum*) reported as producing adenosylhopane and related structures (as cited in table 2.2a, Cooke, 2011) also produces other BHP compounds. Purple non-sulfur bacteria produce BHT (Talbot et al., 2003a), while

*Nitrosomonas europaea* and *Bradyrhizobium japonicum* produce aminotriol (Seemann et al., 1999; Bravo et al., 2001). Therefore, even if detected, it might be challenging to pinpoint which bacteria produced specific BHP compound.

GDGTs were almost entirely made up of brGDGTs, which are produced in soils and therefore, indicative of terrestrial OC (Weijers et al., 2006). BrGDGTs are potentially produced by Acidobacteria (Weijers et al., 2009) (Table 4.4). Relatively large amounts of crenarchaeol were also present in three samples along the glacier meltwater stream and the Zackenberg river (G-1408, ZR-BL-0108 and ZR-B-0808). Crenarchaeol is produced by ammonia-oxidizing archaea (Pitcher et al., 2010) and is indicative of aquatic environments (Hopmans et al., 2004; Zell et al., 2013). This suggests that crenarchaeol detected in samples from Greenland was produced in situ in these water systems, while brGDGTs are derived from soils.

### **6.3.4 Microbial community diversity in samples from the Zackenberg valley**

Next, to understand the source of soil OC at the Zackenberg valley, the microbial community of soil and sediment samples from Greenland was analysed.

#### **6.3.4.1 Chemical composition of samples, and their effect on microbial diversity**

Since the chemical properties of samples could be one of the factors that affects microbial community diversity (Venkatachalam et al., 2021), PCA was performed using chemical data of subset of sediment and soil samples in Greenland chosen for microbial investigation. All samples chosen for microbial analysis, including sediments, had detectable amounts of DNA and were amplified with PCR.

Interestingly, PCA indicated that TC, TN, and TOC had low impact on the first two principal components. In line with this, Venkatachalam et al. (2021) suggested that metals were one of the main factors affecting microbial community structure of the ecosystem of deglaciated foreland in Svalbard. This suggests that this is due to a strong influence of major metals rather than OC on the overall chemical parameters of the samples.

Both PCA and PERMANOVA analysis showed that sediments and soils have different chemical properties. This is in line with findings in Colombo et al. (2020), which reported difference between sediment and soil samples from a rock glacier and glacier front in the Italian Alps. Besides, findings in Greenland indicate that OC and metals concentration was higher in soils compared to sediments (see sections 6.2.1.2 and 6.2.1.4).

#### **6.3.4.2 Bacterial diversity**

Alpha diversity measurements showed some difference in microbial community composition by sample type, indicating that sediment samples collected along the meltwater stream and the Zackenberg river had lower bacterial diversity compared to sediments collected along the lake and soils. This difference in bacterial diversity in sediments could be due to the samples collected along the meltwater stream and the river being more mobile than the samples collected along the lake, indicating the importance of sampling location. This is in line with findings in Huang et al. (2019), reporting that sediment samples from lakes in the Taihu basin in China had higher bacterial diversity compared to sediments from the lake wetland and estuary, both of which are a subject to higher water in/outflow compared to a lake. Further, Mapelli et al. (2018) reported that at any stage of development rhizosphere soils had higher microbial diversity than bulk soils, which is in line with soils in Greenland being more vegetated and richer in OM compared to sediments and therefore, having higher bacterial diversity. Moreover, an NMDS ordination plot of family-level bacterial communities also showed difference between bacteria inhabiting soils and sediments. Similar to the findings in alpha diversity distribution of samples, sediment samples collected along the lake were similar to soils, indicating similarity between microbial community composition of soils and sediments along the lake.

As expected, similar to the previous findings in Sweden and Iceland, there was no pattern in distribution of the bacterial community along the Zackenberg downstream transect. This is contrary to findings in Mapelli et al. (2018) and Venkatachalam et al. (2021), suggesting that diversity of the microbial community increases with the age of samples. This suggests that since the Zackenberg valley has been deglaciated for longer (since the Early Holocene (ca. 10.5 ka), Garcia-Oteyza et al., 2022), compared to study sites in Svalbard (1900 years along the Midtre Lovénbreen glacier chronosequence in Mapelli et al., 2018 and

Venkatachalam et al., 2021), the microbial community in samples from Greenland has stabilised over time. This is in line with findings in Schütte et al. (2009) suggesting that community composition stabilizes in older deglaciated soils (older than 150 years) in Spitsbergen. Since soils in Greenland are older than that, it is safe to assume that the microbial community composition stabilised and became homogeneous a long time ago.

#### 6.3.4.3 Bacterial taxa

Dominant phyla identified in samples from Greenland were Acidobacteria, Chloroflexi, Proteobacteria, Verrucomicrobia and Actinobacteria (Figure 6.14a and Figure 6.15a). Similar to the findings in Iceland, candidate phylum AD3 had lower relative abundance compared to Sweden. Literature indicates that previously high relative abundance of this phylum was reported only in samples from the Antarctic (Mitchell Peninsula, Ji et al., 2016) and usually it has lower relative abundance. Generally, relative abundance of phyla identified in Greenland is comparable to the findings in Iceland and to some extent to the observations in Sweden, as well as the findings in other similar studies. For example, Venkatachalam et al. (2021) reported that Acidobacteria, Actinobacteria, Proteobacteria, Chloroflexi and Verrucomicrobia were also the most abundant phyla identified in soil samples from a glacier foreland in Svalbard. Moreover, according to Chu et al. (2010), in soils from the Arctic tundra, Acidobacteria, Actinobacteria and Proteobacteria were the most abundant phyla, while in samples from a glacier foreland in Spitsbergen, Acidobacteria and Actinobacteria were the most abundant phyla (Schütte et al., 2010). The fact that other habitats in the Arctic are also inhabited by similar microbial communities suggests that bacterial communities established in sediments and soils at the Zackenberg valley could be descriptive of the overall Arctic environment.

Differences in microbial communities of soil and sediment samples were observed. As discussed earlier, soils had higher OC content compared to sediments, therefore, this was expected, as the chemical composition of the two environments was also different, which affects microbial community diversity (Venkatachalam et al., 2021). For instance, Proteobacteria (24.8±15.7%), Chloroflexi (14.7±9.6%), Actinobacteria (12.2±6.5%) and Acidobacteria (12.0±6.8%) were dominant in sediments, while Acidobacteria (23.2±5.7%), Chloroflexi (17.1±3.8%), Verrucomicrobia (16.0±3.0%) and Actinobacteria (11.7±3.2%)

were the most abundant in soil samples. Earlier, in samples from Sweden, it was also observed that Proteobacteria had higher relative abundance in sediment samples, while Chloroflexi had higher relative abundance in soils. This is also in line with what Mateos-Rivera et al. (2016) found in Norway, reporting that Proteobacteria was more abundant in recently deglaciated soils, while Chloroflexi had higher relative abundance in older soils. Moreover, according to Ji et al. (2016), Chloroflexi performs photosynthesis in soils, explaining its higher relative abundance in soil samples with higher presence of vegetation. Similar to samples with low OC content from Sweden and Iceland, sediment samples from Greenland also had Bacteroidetes ( $7.0\pm 8.0\%$ ). In line with this, Venkatachalam et al. (2021) also found that Bacteroidetes was abundant in younger samples. This suggests that Bacteroidetes prefer low carbon environments, such as sediment samples. Moreover, Firmicutes ( $3.8\pm 7.4\%$ ) was detected in sediment samples from Greenland, also suggesting that this phylum prefer low carbon environments.

Next, each phylum was investigated in more detail in order to gain a better understanding of microbial community structure along the Zackenberg valley transect. First, Acidobacteria was present and was one of the dominant phyla in all samples from Greenland, except the sediment sample ZR-B-0808\_2. Overall, Acidobacteria had higher relative abundance in soils compared to sediments. In line with this, Acidobacteria has been reported to be one of the most abundant phyla in soils (Janssen, 2006). Moreover, according to Venkatachalam et al. (2021), Acidobacteria was more prevalent in older soils from a glacier foreland in Svalbard. This suggests that sediment samples are younger compared to soils, most likely being brought downstream with the water flow along the valley. As expected, no particular pattern was observed in the distribution of this phylum contrary to the findings in Sweden and Iceland. This could be due to the older age of the entire catchment (Schütte et al., 2010).

Proteobacteria was detected in all samples from the Zackenberg valley. Similar to Acidobacteria, no particular pattern of distribution of this phylum was observed. Overall relative abundance of Proteobacteria was much higher in sediment samples compared to soils. This is in line with the findings in Mateos-Rivera et al. (2016), reporting higher abundance in recently deglaciated soils, and observations in Choudhari et al. (2014), where Proteobacteria was one of the most abundant bacteria in snow and ice samples from an

Alaskan glacier. The most abundant family members of this phylum in samples from Greenland were Comamonadaceae, Hydrogenophilaceae and Bradyrhizobiaceae. Comamonadaceae and Hydrogenophilaceae were abundant in sediments, while Bradyrhizobiaceae had higher relative abundance in soils. Nemergut et al. (2007) proposed that Comamonadaceae could inhabit englacial or subglacial ice, which suggests it could have been brought with sediment samples by a meltwater stream draining the A.P. Olsen ice cap. *Thiobacillus* genus from the Hydrogenophilaceae family was also abundant in sediments. *Thiobacillus* is a chemolithoautotrophic bacteria, which metabolizes reduced sulphur compounds (Orlygsson and Kristjansson, 2014), which explains its higher relative abundance in sediments (Table 6.2). According to Marcondes de Souza et al. (2014), Bradyrhizobiaceae is a nitrogen-fixing bacterium and can be found in different environments, including soils. Members of this family such as *Bradyrhizobium japonicum*, *Rhodopseudomonas palustris* and *Rhodopseudomonas acidophila* have been reported to produce various BHP compounds (see Table 4.4).

Actinobacteria were detected in all samples from Greenland and had higher relative abundance in sediments than soils, similar to what was observed in Sweden. According to Barka et al. (2016), most Actinobacteria are aerobic, can be either heterotrophic or chemoautotrophic, and can be found in different environments, including aquatic and terrestrial habitats. In sediments, the main family-level member of this phylum was Nocardioidaceae, while Gaiellaceae was the most abundant family-level bacteria in soils. Members of the family Nocardioidaceae are chemoorganotrophs and can be found in different environments, including terrestrial ecosystems (Pershina et al., 2018). Gaiellaceae is a strictly aerobic and chemoorganotrophic bacterium (Albuquerque and da Costa, 2014). Based on these observations it can be presumed that Actinobacteria found in samples from Greenland is mostly chemoorganotrophic, which explains why they have higher relative abundance in sediments descriptive of lower carbon environments.

Another major phylum, Chloroflexi, was detected in all samples from Greenland, except ZR-B-0808\_2. Overall, relative abundance of this bacterium was comparable in soil and sediment samples. Interestingly, sediment sample SS-L-1008\_2 had the highest relative abundance. This was not expected, as Ji et al. (2016) suggested that Chloroflexi could perform photosynthesis, therefore, leading to the expectation that more vegetated soil

samples would have higher dominance of this phylum. Moreover, since Mateos-Rivera et al. (2016) reported that relative abundance of Chloroflexi along a glacier chronosequence in Norway increased with the time of exposure, it was also expected that Chloroflexi would have higher relative abundance in soils rather than sediments, which most likely have been washed down recently. This suggests that this discrepancy could be due to the length of the transect, allowing transfer of Chloroflexi from vegetated banks along the valley. oc28, classified at class-level Ellin6529 and Ktedonobacteria, and Thermogemmatissporaceae were the most dominant members of Chloroflexi in samples from Greenland. Literature indicates that most members of this phylum were poorly studied. According to Yabe et al. (2017), members of class Ktedonobacteria can be found in different environments, including extreme and cold environments, which is applicable to the study site in Greenland. Meanwhile, according to Lopes et al. (2015), Ellin6529 fixes nitrogen, which could be contributing to enriching sediments and soils from Greenland in labile nitrogen, fostering growth of vegetation.

Verrucomicrobia were detected in all samples from Greenland, except the sediment sample G-1408\_3. According to Schlesner et al. (2006), Verrucomicrobia can be found in different aquatic and terrestrial ecosystems. This bacterium was also found in samples from Iceland, samples from a glacier foreland in Norway (Mateos-Rivera et al., 2016), as well as samples from Svalbard (Venkatachalam et al., 2021), suggesting that Verrucomicrobia is a common phylum in deglaciated catchments. In samples from Greenland, relative abundance of Verrucomicrobia did not vary in soils, while in sediments, it had higher relative abundance in samples collected along the Store Sø Lake compared to the samples collected along the Zackenberg river. According to Venkatachalam et al. (2021), relative abundance of Verrucomicrobia did not vary much in well-developed soils, while Mateos-Rivera et al. (2016) reported that there was an upward trend in relative abundance of this phylum along the glacier chronosequence in Norway. Therefore, it is suggested that Verrucomicrobia in soils have stabilised over time as Schütte et al. (2010) suggested. As for the sediments, samples from the lake were more soil like, compared to the rocky sediments from the river, suggesting that these samples behave similar to soils. Chthoniobacteraceae was the most dominant family-level member of Verrucomicrobia in samples from Greenland. Chthoniobacteraceae is a free-living aerobic and saccharolytic bacteria living in

soil (Janssen and Hedlund, 2015). In a polar setting, it was one of the most abundant bacteria in soils collected from a glacier foreland in East Antarctica (Yan et al., 2017). This soil organism also had stable relative abundance in soil samples from Greenland and had higher relative abundance in sediments from the lake rather than the river, reinforcing the previous suggestion that the difference in relative abundance is due to the nature of samples.

Some sediment samples (G-1408\_3 and ZR-B-0808\_2) also had high concentration of Bacteroidetes and Firmicutes. Some Firmicutes were detected in soils from a glacier foreland in Spitsbergen (Schütte et al., 2010). Moreover, Kwon et al. (2015) reported that Firmicutes was one of the abundant phyla in deglaciated soils in the early stages of development in Svalbard. Both Bacteroidetes and Firmicutes were abundant in snow and ice samples from an Alaskan glacier (Choudhari et al., 2014), which explains presence of these bacteria in sediment samples rather than soils. Bacteroidetes was also one of the major phyla in some samples from Sweden and Iceland. According to Venkatachalam et al. (2021), Bacteroidetes is representative of younger soils in Svalbard. Moreover, in Sweden and Iceland this phylum was dominant in samples with low TOC content, which is also true for samples from Greenland (0.01% in G-1408\_3 and 0.25% in ZR-B-0808\_2). According to Sheik et al. (2015), Bacteroidetes grows in anoxic environments such as proglacial lake and meltwater stream sediments and glacier terminus and metabolizes inorganic material of subglacial origin. Based on these observations, it can be suggested that Bacteroidetes and Firmicutes prefer low carbon environment, though it doesn't explain their lower relative abundance in other low TOC samples from Greenland.

To summarize, organisms detected in samples from Greenland are very similar to that from other aforementioned studies (e.g., Chu et al., 2010; Costello and Schmidt, 2006; Mateos-Rivera et al., 2016; Schütte et al., 2010; Seok et al., 2016; Venkatachalam et al., 2021) but their relative abundance did not follow a particular trend along the transect. This could be due to the age of catchment, where bacterial communities have stabilised over time as suggested by Schütte et al. (2010).

#### 6.3.4.4 Relationship between microbial community and organic biomarkers

As discussed earlier, specific biomarkers are associated with particular environments and source organisms. Therefore, we attempted to establish a relationship between the bacteria inhabiting the Zackenberg valley catchment and the most abundant organic biomarkers detected in samples from Greenland using the known source organisms that produce these biomarkers (Table 4.4).

First, as this study is aimed at understanding soil development in deglaciated Zackenberg valley, it is important to look at brGDGTs and soil marker BHPs and their source organisms. So, similar to the findings in Sweden and Iceland, Acidobacteria, a potential source of brGDGTs (Weijers et al., 2009), was one of the main phyla in samples from Greenland. Next, species that belong to Bradyrhizobiaceae, which was one of the most abundant family members of Proteobacteria in samples from Greenland are responsible for producing different BHP compounds, including soil marker BHPs. For instance, *Bradyrhizobium japonicum* produces adenosylhopane and related structures (Table 4.4). A member of family Hyphomicrobiaceae (Proteobacteria), purple non-sulfur bacterium *Rhodomicrobium vannielii* (Neunlist et al., 1985), which also produces soil marker BHPs, was detected in samples from Greenland (1-2%). Family Nitrosomonadaceae of a specie *Nitrosomonas europaea*, which produces adenosylhopane and related structures, (as cited in table 2.2a), had a relatively low abundance in few samples.

As previously discussed, other BHP compounds were also detected in samples from Greenland. BHT was the most abundant BHP compound (46-61% of total BHPs) in samples from Greenland, followed by Me-BHT (8-19% of total BHPs), aminotriol (1-9% of total BHPs), BHT glucosamine (1-5% of total BHPs) and BHT cyclitol ether (1-2% of total BHPs). The relative abundance of these biomarkers and the bacteria responsible for their production did not follow a particular trend along the transect. Interestingly, bacteria responsible for producing soil marker BHPs have also been reported as source organisms of other BHP compounds identified in samples from Greenland. For instance, *Bradyrhizobium japonicum* also produces aminotriol (Bravo et al., 2010), while *Rhodomicrobium vannielii* is also responsible for production of BHT (Neunlist et al., 1985). Other species of Bradyrhizobiaceae, *Rhodopseudomonas palustris* and *Rhodopseudomonas*

*acdophila*, produce Me-BHT (as cited in table 2.2a, Cooke, 2011) and BHT (Flesch and Rohmer, 1988) respectively. *Nitrosomonas europaea* responsible for production of soil marker BHPs also produces aminotriol (Seemann et al., 1999).

Other bacteria linked to production of BHP compounds had lower relative abundance. For instance, family Nostocaceae, members of which (*Anabaena cylindrica* (Talbot et al, 2008) and *Nostoc muscorum* (Bisseret et al., 1985; Talbot et al, 2008)) produce BHT, had low relative abundance in samples from Greenland. Family Frankiaceae, of a specie *Frankia*, which produces (Berry et al., 1991; Rosa- Putra et al., 2001) BHT, was also detected but had low relative abundance in samples from Greenland. Similarly, Burkholderiaceae, of a specie *Burkholderia cepacia*, which produces BHT cyclitol ether, (Talbot and Farrimond, 2007; Talbot et al., 2007a) had a minute relative abundance.

Families of other species in samples were reported to produce several BHP compounds and had low relative abundance in samples from Greenland. For example, family Acetobacteraceae had small relative abundance in few samples. Members of Acetobacteraceae produce BHT (genus *Acetobacter* (Peiseler and Rohmer, 1992; Talbot et al., 2007a), genus *Gluconacetobacter* (Talbot et al., 2007a), specie *Acetobacter xylinum* (Ourisson and Rohmer 1992; Herrmann et al., 1996)) and BHT cyclitol ether (specie *Acetobacter xylinum* (Ourisson and Rohmer 1992; Herrmann et al., 1996)). A specie *Azotobacter vinelandii* of Pseudomonadaceae also produces both BHT and BHT cyclitol ether (Vilcheze et al., 1994). Beijerinckiaceae and its members (*Beijerinckia indica* and *Beijerinckia mobilis*) produce aminotriol and BHT (Vilcheze et al., 1994). Cyanobacteria and some of its members produce BHT and Me-BHT (Talbot et al., 2003a; Talbot et al, 2008; as cited in table 2.2a, Cooke, 2011). A specie of Xanthomonadaceae, *Frateuria aurantia*, was reported to produce BHT and BHT cyclitol ether (Joyeux et al., 2004). Members of Methylocystaceae (*Methylocystis parvus* (Talbot et al., 2001) and *Methylosinus trichosporium* (Neunlist and Rohmer, 1985a)) produce BHT and aminotriol. A specie of Sphingomonadaceae, *Zymomonas mobilis*, was reported to produce BHT, BHT cyclitol ether and BHT glucosamine (Flesch and Rohmer, 1988; Flesch and Rohmer, 1989; Moreau 1997; Neunlist et al., 1988; Talbot et al., 2001; as cited in table 2.2a, Cooke, 2011). Family Methylobacteriaceae, of a specie *Methylobacterium organophilum*, which produces various BHP compounds (BHT, BHT cyclitol ether, aminotriol, and Me-BHT) had very low

relative abundance in few soil samples (as cited in table 2.2a, Cooke, 2011; Bisseret et al., 1985; Flesch and Rohmer, 1988; Knani et al., 1994; Neunlist et al., 1988; Renoux and Rohmer, 1985; Talbot et al, 2007b).

Despite being able to link some of the existing organisms with organic biomarkers, relative abundance of most of these bacteria is low. The only exceptions are Bradyrhizobiaceae (Proteobacteria), which produces soil marker BHPs, and aminotriol (as cited in table 2.2a; Bravo et al., 2001; Flesch and Rohmer, 1988), and Acidobacteria, a potential source organism of brGDGTs (Weijers et al., 2009). Since organic biomarkers accumulate over time, bacteria with low relative abundance may have produced organic biomarkers over time. On the other hand, the catchment could have been recolonized or more likely, not all source organisms of organic biomarkers are known, which calls for more studies to establish which organisms are responsible for producing specific biomarkers.

## **6.4 Conclusion**

Compared to the transects studied in Sweden and Iceland, the Zackenberg valley transect has been ice-free for longer. Accumulation of OC along the catchment was not observed, suggesting stabilisation of OC signals over time. Organic biomarkers, BHPs and GDGTs, were detected in all soil samples but did not follow a pattern along the downstream transect. Relative abundance of soil marker BHPs is comparable to that in some other Arctic catchments and glacier forelands but much lower than that in the Arctic permafrost. Meanwhile, the lack of an upward or any trend downstream from the glacier in the distribution of soil marker BHPs and the  $R'_{\text{soil}}$  index suggests that soil along the Zackenberg river transect has reached a certain point of development and stabilised due to the age of exposure. Meanwhile, the stable OC signal and downward trend in the  $R'_{\text{soil}}$  down the lake transect suggests that soil marker BHPs have become less important compound of OC.

Major phyla identified in samples from Greenland were Acidobacteria, Chloroflexi, Proteobacteria, Verrucomicrobia and Actinobacteria, which are similar to microbial communities inhabiting other Arctic catchments, suggesting that bacteria identified in samples from the Zackenberg valley is generally descriptive of the Arctic environment. The relative abundance of bacteria did not vary along the transect, suggesting that similar to

organic biomarkers, microbial community stabilised over time in this catchment. Moreover, as expected, soils and sediments had different chemical properties, with soils having higher OC and major metal concentrations, which affected the bacterial community inhabiting these environments. The attempt to establish a relationship between organic biomarkers and their source organisms showed that only some bacteria can be clearly linked to the production of specific biomarker compounds. For instance, Acidobacteria could be a potential source organism of brGDGTs and some species of Bradyrhizobiaceae (Proteobacteria) produce soil marker BHPs and aminotriol. Other bacteria linked to production of organic biomarkers were detected in smaller quantities, suggesting either accumulation of biomarkers over time or re-colonization of the catchment with bacteria, which are not contributing to the accumulation of biomarkers. To conclude, the Zackenberg valley is an older catchment, where OC, organic biomarkers and microbial community signals have stabilised and homogenized over time.

## **7 Synthesis: Accumulation of soil derived OC in Sweden, Iceland, and Greenland**

### **7.1 Introduction**

Previous chapters looked at three catchments that have been ice free for various periods of time: Tarfala valley in Sweden being bare (Storglaciären and Isfallsglaciären being the youngest glacier forelands), Vatnajökull ice cap in Iceland being an older catchment, and Zackenberg valley in Greenland being the oldest catchment out of the three. The catchments also vary by location and bedrock geology. This chapter investigates similarities and differences between these catchments based on the findings in previous chapters in order to learn how various deglaciated systems in the (sub)Arctic accumulate OC over time.

The main aim of this chapter is to gain better insight to accumulation of soil derived OC in three (Tarfala valley in Sweden, Vatnajökull ice cap in Iceland, and Zackenberg valley in Greenland) deglaciated catchments in the Arctic and Subarctic by comparing catchment chemistry, organic biomarkers and microbial community along transects. In order to achieve this aim, the following objectives were carried out:

1. to compare concentration of TOC, C/N and major metals between the three catchments;
2. to compare concentration and distribution of BHPs and GDGTs between the three catchments;
3. to compare microbial community structure between the three catchments.

Based on these objectives the following was hypothesised:

1. Overall, there is a statistically significant difference between soils and sediments in all countries by:
  - a. environmental chemistry;
  - b. microbial community diversity and composition, where analysed.
2. There is a statistically significant difference between the countries by:
  - a. environmental chemistry;
  - b. organic biomarker concentrations;
  - c. microbial community diversity and composition.
3. TOC concentration is lowest in Sweden and highest in Greenland due to the accumulation of OM over time of exposure.
4. Since the Zackenberg valley had longer time of exposure, samples from this catchment have higher organic biomarker concentrations than samples from the Vatnajökull ice cap.
5. Samples from the Vatnajökull ice cap have higher BHPs and GDGTs concentration than samples from the Tarfala valley.
6. Microbial community diversity is highest in Greenland and lowest in Sweden.

By the end of this chapter, the following is expected:

1. to understand how OC accumulated in soils in deglaciated catchments in Sweden, Iceland and Greenland;
2. to conclude whether organic biomarkers can be used to understand accumulation of soil derived OC across different systems.

## **7.2 Results and discussion**

### **7.2.1 Environmental chemistry**

A total of 94 samples (mostly in triplicates) from Tarfala valley in Sweden, Vatnajökull ice cap in Iceland and Zackenberg valley in Greenland were investigated for environmental chemistry: TC, TOC, TN, and major metal concentrations.

### 7.2.1.1 Comparing chemical properties in sediment and soil samples from Sweden, Iceland, and Greenland

A PCA of environmental chemistry on mean values (of mostly triplicates) of all samples collected in Sweden, Iceland, and Greenland indicated that PC1 explained 36.9% of total variance, while PC2 explained 21.6%. PCA plots in Figure 7.1 show that there is difference between sediments and soils (Figure 7.1a), as well as samples from different countries (Figure 7.1b). Sediment samples are clustered to the top left, away from C and N, while soils are scattered across the plot (Figure 7.1a), suggesting that soil samples vary in chemical composition. PERMANOVA analysis also showed that there was statistically significant difference between soil and sediment samples ( $p$ -value  $< 0.05$ ). This is also similar to other findings (e.g., Colombo et al., 2020) where proglacial soils and sediments varied in chemical composition, in line with the hypothesis that soils and sediments have different environmental chemistry.

Further, the PCA plot in Figure 7.1b shows clustering of samples by country. Samples from Greenland are clustered to the left side of the plot, along PC2, indicating that C and N played an important role in distribution of these samples. Samples from Sweden are clustered in the top left corner and along PC1, demonstrating that metals rather than C and N play a major role in the clustering of these sample. Meanwhile, samples collected in Iceland occupy the centre of the plot and seem to be affected equally by PC1 and PC2. Additionally, according to the PERMANOVA analysis, there was a statistically significant difference between the samples from different countries ( $p$ -value  $< 0.05$ ). This was expected as the catchments have different geology (see below) and have been exposed for different length of time after deglaciation, which plays an important role in the soil development in proglacial areas (Wietrzyk et al., 2018).

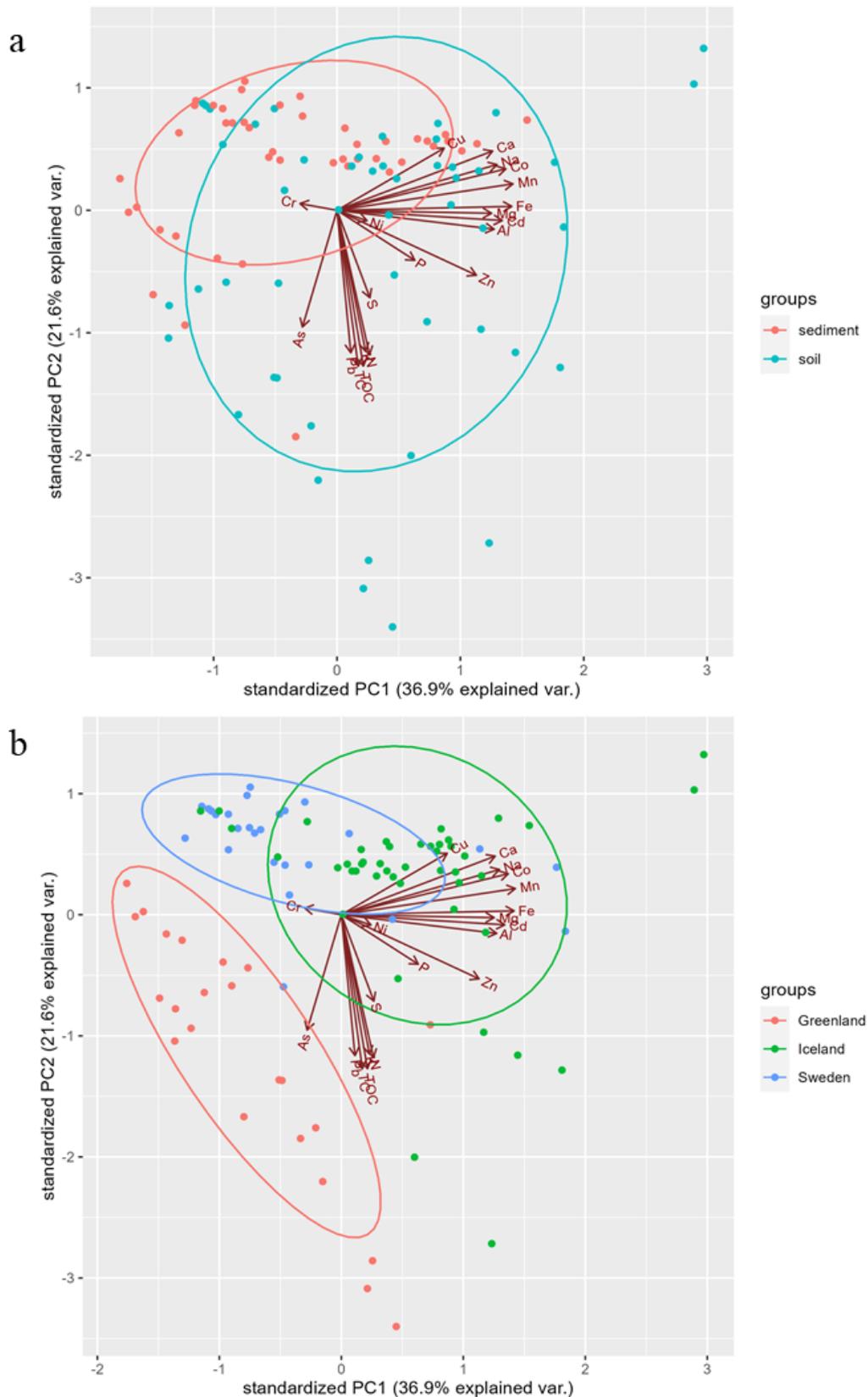


Figure 7.1. PCA ordination plots illustrating the discrimination between all samples from Sweden, Iceland, and Greenland according to chemical properties by the (a) sample type and (b) country. The input data was standardised to have zero mean and variance one. The ellipses represent the core area added by confidence interval of 68%.

### 7.2.1.2 Varying metal concentrations

Total metal concentration and concentrations of the most abundant metals (Fe, Al, Ca, and Mg) were highest in samples collected in Iceland compared to that in Sweden and Greenland (Table 7.1). This difference in metal concentrations is most likely due to the different geological origin of samples, with Iceland having overall higher metal content due to mafic igneous geology. The bedrock of the Tarfala valley is mostly comprised of quartz- and feldspar-rich gneiss (Baird, 2005), while gneiss and granite bedrock, as well as some sandstone and basalt can be found at the Zackenberg valley (Christiansen et al., 2008).

Table 7.1. Mean concentration (mg/kg) of metals in Sweden, Iceland, and Greenland.

Country\ Metals	Total		Fe		Al		Ca		Mg	
<b>Sweden</b>	12963	±4447	5227	±2230	2998	±791	2658	±905	1247	±342
<b>Iceland</b>	18757	±4974	9423	±2209	3419	±1158	3120	±1290	1609	±683
<b>Greenland</b>	10741	±4027	4857	±1862	2517	±1196	1348	±441	1198	±558

Overall, soil samples from all three countries had higher total metal concentrations compared to sediment samples (Table 7.2). This is in line with observations from the PCA plot (Figure 7.1a), indicating that soils and sediments have different chemical compositions.

Table 7.2. Mean metal concentration (mg/kg) of sediment and soil samples from Sweden, Iceland and Greenland.

Country	Sediment		Soil	
<b>Sweden</b>	12109	±3102	13813	±5667
<b>Iceland</b>	16434	±3979	20790	±4933
<b>Greenland</b>	8192	±2741	12701	±3810

### 7.2.1.3 Comparing TOC, TN and C/N across the three catchments

TOC analysis showed that mean TOC concentration of field triplicates of samples collected from the Tarfala valley in Sweden was the lowest of three sites, followed by Iceland and Greenland (Table 7.3). Across all catchments, soils had higher TOC content compared to sediment samples, similar to other studies (e.g., Guzella et al., 2016), suggesting that soils are more developed and organic rich compared to sediments.

Table 7.3. Mean TOC concentration (%) of sediment and soil samples from Sweden, Iceland and Greenland.

Country	Sediment		Soil	
Sweden	0.07	±0.13	0.32	±0.47
Iceland	0.04	±0.96	0.96	±1.62
Greenland	0.25	±0.50	1.62	±1.54

Variation in TOC values across the catchments, suggests that the site in Sweden has accumulated less soil OC compared to other sites, while the Vatnajökull ice cap and the Zackenberg valley have accumulated similar concentrations of soil OC, despite the latter being exposed for longer after the deglaciation. Taking into account age of exposure of the Zackenberg valley, it is suggested that OC content plateaus out after reaching the age of the oldest moraines in Iceland, undergoing transformations in soil composition without changes to the OC concentration. Moreover, in Iceland, there was a clear trend of TOC build-up with the age of moraines (Figure 5.3), which was not observed in Sweden or Greenland, suggesting that in young catchments, such as Sweden, there is not enough OC to create a similar pattern, while in older catchments, such as Greenland, downstream distribution of OC stabilizes. It is worth mentioning that some of the samples, especially the ones with higher TOC content, had large variations (i.e., sample SV-LIAM-3005 had a mean TOC of  $5.24 \pm 2.68\%$ , VK-RM-2709 –  $4.71 \pm 3.07\%$ , SS-LI-1408 –  $4.88 \pm 3.56\%$ ). All samples were collected in triplicates in a close proximity from each other, suggesting that the sampling sites are very heterogeneous and there may be implications for studies that only collect one sample per site.

Similar to the TOC, the mean TN values were the lowest in Sweden ( $0.03 \pm 0.02\%$ ). The mean TN concentration being higher in Iceland ( $0.05 \pm 0.07\%$ ) and Greenland ( $0.07 \pm 0.07\%$ ). The sample collected from a moraine heavily vegetated by the invasive species Nootka lupine (*Lupinus nootkatensis*) (SV-LIAM-3005) had the highest TN of  $0.44 \pm 0.19\%$ . This sample also had a high TOC concentration ( $5.24 \pm 2.68\%$ ), which is most likely due to vegetation. This is in line with findings in Dümig et al. (2011), where increase in OC in soils from a glacier foreland in Switzerland was linked to vegetation as well. Overall, across all three sites, TOC and TN are generally correlated, suggesting that nitrogen has an organic nature (Stein and Rack, 1995 in Huon et al., 2002) and available for uptake by plants (Brust, 2019). This

strengthens the previous observations that high TN and TOC values are often due to the presence of vegetation.

Further, across all three sites, C/N values were lower in sediment samples compared to soil samples (Table 7.4). Lower C/N ratios observed in sediment samples from three catchments, as well as soil samples from Sweden is descriptive of aquatic environments (from  $7.5 \pm 1.1$  in marine environments to  $12.7 \pm 2.3$  in freshwater environments, Yu et al., 2010), while higher values observed in soil samples from Iceland and Greenland are representative of soils (from  $8.9 \pm 1.1$  to  $17.9 \pm 3.6$  in soils, Yu et al., 2010). The highest C/N ratios were recorded in vegetated moraines in Iceland and correspond with plant material recorded in other studies (e.g., from  $22.7 \pm 11.6$  to  $24.6 \pm 9.4$  in plants, Yu et al., 2010). This, along with the fact that older moraines in Iceland had more vegetation than sampling sites in Greenland suggest that higher C/N ratio and TOC concentration in Iceland are due to the accumulation of OC due to soil development caused by vegetation.

Table 7.4. Mean C/N values of sediment and soil samples from Sweden, Iceland and Greenland.

Country	Sediment		Soil	
Sweden	3.19	$\pm 3.80$	7.25	$\pm 7.82$
Iceland	1.20	$\pm 1.82$	10.36	$\pm 8.54$
Greenland	6.77	$\pm 5.82$	14.13	$\pm 4.66$

Now, that it has been established that soil and sediment samples differ by catchment in terms of environmental chemistry, it would be interesting to see the differences in organic biomarkers.

## 7.2.2 Organic biomarkers

Looking at all samples studied for organic biomarkers from the three catchments, even though BHPs form a small fraction of the total OC, while GDGTs even less so, it can be seen that based on the Pearson correlation coefficient there was a strong correlation between TOC and total BHPs, and TOC and total brGDGTs (Table 7.5). This was expected, since organic biomarkers depend on presence and composition of organic carbon content (Höfle et al., 2015). Meanwhile, a weak correlation between total BHPs and total brGDGTs was observed (Table 7.5). As explored in previous chapters, different biomarkers are produced

by different bacteria, suggesting that BHPs and brGDGTs accumulate irrespective of each other.

Table 7.5. Correlation between total organic carbon, total brGDGTs and total BHPs.

	TOC, %	$\Sigma$ brGDGTs, $\mu\text{g/g}$ sediment	$\Sigma$ BHPs, $\mu\text{g/g}$ sediment
TOC, %	1		
$\Sigma$ brGDGTs, $\mu\text{g/g}$ sediment	0.75	1	
$\Sigma$ BHPs, $\mu\text{g/g}$ sediment	0.70	0.49	1

### 7.2.2.1 Absolute versus relative concentration of organic biomarkers

In this study, organic biomarker concentrations in soil samples were normalised by TOC, which is representative of a relative concentration. Comparison of absolute concentration ( $\mu\text{g/g}$  sediment) versus relative concentration of BHPs ( $\mu\text{g/g}$  TOC) along the Tarfalajaure, Svínafellsjökull and Virkisjökull transects are presented in Figure 7.2.

At the Tarfalajaure transect, the absolute concentration indicates that the amount of total BHPs initially increases, followed by a subsequent decrease (Figure 7.2a), which is in line with OC distribution along the transect (Figure 4.3, samples KG-MW-1308, T-L-1308, TL-RB-1408 and MS-TL-1408). The relative concentration of total BHPs increases along the transect (Figure 7.2b).

At Svínafellsjökull, the absolute concentration of total BHPs first increases, followed by a subsequent decrease (Figure 7.2c), similar to the trend observed in OC concentration (Figure 5.3, samples SV-PL-2905, SV-LIAM-3005 and SV-2.5K-3105). Meanwhile, the relative concentration of total BHPs first decreases and subsequently increases (Figure 7.2d).

At Virkisjökull, the absolute concentration of total BHPs increases with the age of samples (Figure 7.2c) in line with OC distribution (Figure 5.3, samples VK-YM-0206, VK-OM-0206 and VK-RM-2709), while relative concentration decreases (Figure 7.2d).

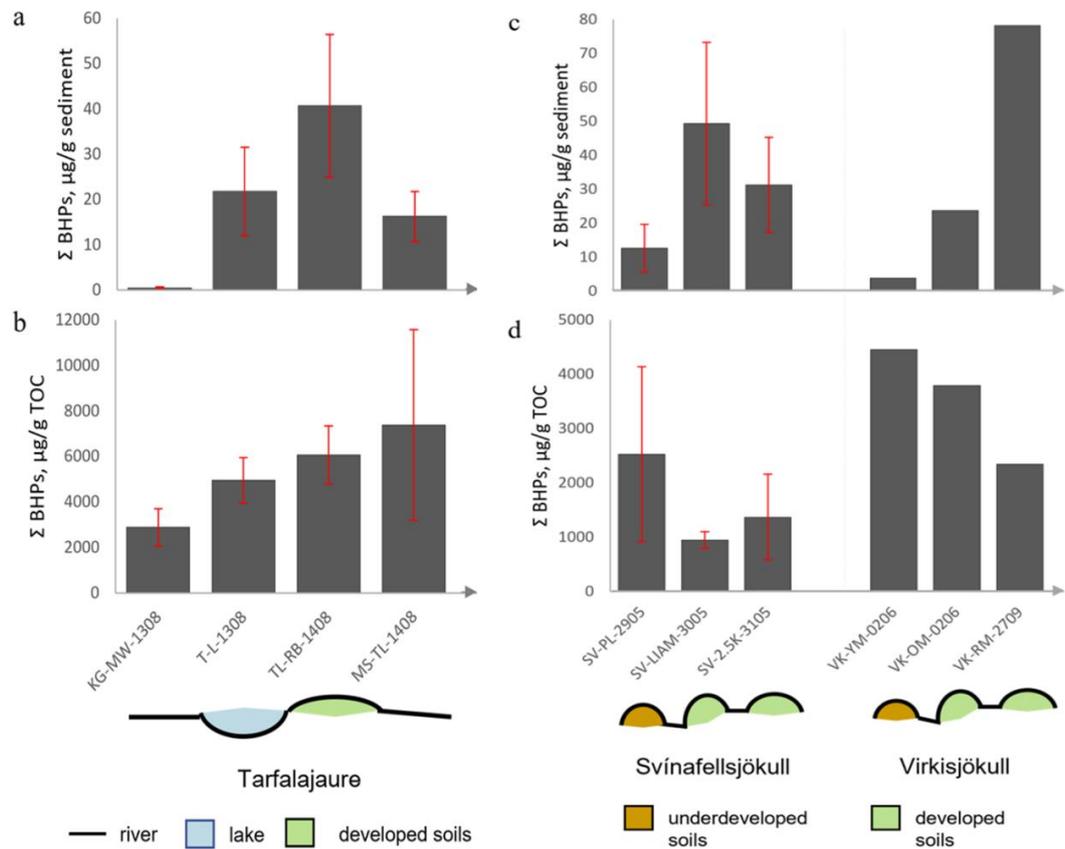


Figure 7.2. Average distribution of (a) absolute concentration ( $\mu\text{g/g}$  sediment) and (b) relative concentration of total BHPs ( $\mu\text{g/g}$  TOC) in selected samples along the Tarfalajaure transect in Sweden, and (a) absolute concentration ( $\mu\text{g/g}$  sediment) and (b) relative concentration of total BHPs ( $\mu\text{g/g}$  TOC) in selected samples along the Svínafellsjökull and Virkisjökull transects in Iceland. Error bars represent  $\pm$  one standard deviation on three field replicate samples collected from individual sampling locations at the Tarfalajaure and Svínafellsjökull. Charts at the bottom of the graph show downstream (left to right) transect profiles.

Distribution of the absolute concentration of total BHPs along the downstream transects follow the same trend as the change in OC concentrations, reinforcing observations in previous chapters that the two are strongly correlated. At Tarfalajaure and Svínafellsjökull, it seems that less BHPs are produced in older soils. Along with the decreased OC concentration, this might be due to the increased presence of other OC compounds. Meanwhile, the increase in the relative concentration of total BHPs suggests that the role of BHPs in OC becomes more important over time. On the contrary, at Virkisjökull the absolute concentration of BHPs increases, however, a decrease in relative concentration suggests that the role of BHPs in OC becomes less significant. Therefore, it seems that BHPs behave differently at each transect.

### **7.2.2.2 Comparing organic biomarker concentrations and distribution along the transects of three different catchments**

Environmental chemistry data showed that there was difference between the catchments, therefore, it was expected to see difference in organic biomarkers as well. PERMANOVA analysis showed a significant variance in total BHP compounds ( $p$ -value  $< 0.05$ ) and total brGDGTs between the countries ( $p$ -value  $< 0.05$ ), suggesting that each catchment has a different ecosystem.

Organic biomarker analysis revealed presence of BHPs and GDGTs in all samples across the three catchments, except samples with low TOC content in Sweden, strengthening the observation that detection of organic biomarkers is affected by OC content. The mean absolute concentration of total BHPs was  $12 \pm 15$   $\mu\text{g/g}$  sediment in Sweden,  $25 \pm 23$   $\mu\text{g/g}$  sediment in Iceland and  $90 \pm 76$   $\mu\text{g/g}$  sediment in Greenland, suggesting that biomarker production increases over time. On the contrary, relative concentration of total BHPs was lowest in samples from the Vatnajökull ice cap ( $2469 \pm 1404$   $\mu\text{g/g}$  TOC), while the values were higher at the Zackenberg valley ( $5230 \pm 1876$   $\mu\text{g/g}$  TOC) and the Tarfala valley ( $6727 \pm 5250$   $\mu\text{g/g}$  TOC). High relative concentration of BHPs in Sweden is most likely due to low OC content. Meanwhile, concentration of total BHPs was higher in samples from Greenland compared to Iceland, confirming that the older catchment in Greenland has more BHPs compared to the younger sites in Iceland. Moreover, compared to other studies, relative BHP concentrations detected in study sites in this project are generally much higher than in other studies (e.g., Cooke et al., 2008; Doğrul Selver et al., 2012; Doğrul Selver et al., 2015; Höfle et al., 2015; Rethemeyer et al., 2010), while this is not true for absolute concentrations. For instance, in soils from Northern England, the absolute concentration was similar to that in study sites in this project (4-121  $\mu\text{g/g}$  dry soil), while the relative concentration was lower (53-1454  $\mu\text{g/g}$  TOC) (Cooke et al., 2008). This could be due to the lower TOC content in samples in this study (Table 7.3) compared to soils in Northern England (1.7-10.7%; Cooke et al., 2008).

Further, since individual BHP compounds can be used as markers of specific environments (Killops and Killops, 2005), distributions of soil marker BHPs along downstream transects were studied to understand accumulation of soil OC in proglacial areas across the three

catchments. Literature indicates that this is a first such study, with previous studies looking into soil marker BHPs to trace terrestrial OC along land to ocean transects (e.g., Bischoff et al., 2016; De Jonge et al., 2015; Dođrul Selver et al., 2012; Dođrul Selver et al., 2015). Similar to total BHPs, soil marker BHPs were detected in all samples, except some samples from Sweden. Their fractional abundance varied between 0-21% in Sweden, 7-28% in Iceland, and 8-30% in Greenland. Abundance of soil marker BHPs slightly increased from the youngest (Sweden) to the oldest (Greenland) catchments in this study, indicative of accumulation of soil specific BHPs with time. Meanwhile, function of soil marker BHPs, the  $R'_{\text{soil}}$  of the samples collected across the three sites had similar values, slightly increasing from 0.07-0.34 in Sweden, to 0.09-0.35 in Iceland and to 0.11-0.37 in Greenland. Overall, contribution of soil marker BHPs to total BHPs and consequently, the  $R'_{\text{soil}}$  in this study are similar to that in sediment samples elsewhere (e.g., Cooke et al., 2009) but lower than the values from soils (e.g., Cooke et al., 2008; Rethemeyer et al., 2010) and especially permafrost soils (e.g., Dođrul Selver et al., 2015). This suggests that even the oldest proglacial areas in this study have less organic soils than that in Northern England (Cooke et al., 2008) and in Siberian Arctic (Dođrul Selver et al., 2015).

BrGDGTs, which are also produced in soils and used to trace terrestrial OC (Hopmans et al., 2004; Weijers et al., 2006; Dođrul Selver et al., 2012) were investigated in this study. Similar to BHPs, GDGTs were identified in all samples, except some samples from Sweden with low TOC concentration. Majority of GDGTs was made up off brGDGTs, however, crenarchaeol, descriptive of aquatic/fluviol environments (Zell et al., 2013) was detected in some samples from the Tarfala valley and the Zackenberg valley. Absolute concentration of total GDGTs was  $0.77 \pm 1.19 \mu\text{g/g}$  sediment in Sweden,  $1.52 \pm 2.14 \mu\text{g/g}$  sediment in Iceland and  $2.19 \pm 1.25 \mu\text{g/g}$  sediment in Greenland, along with TOC concentration being lower in Sweden and comparable in Iceland and Greenland. This suggests that similar to BHPs, GDGT content in soils increases with the time of exposure. Meanwhile, relative concentration of total GDGTs was highest in Sweden ( $403 \pm 163 \mu\text{g/g TOC}$ ), while comparable in Iceland ( $112 \pm 74 \mu\text{g/g TOC}$ ) and Greenland ( $144 \pm 73 \mu\text{g/g TOC}$ ). Since, concentrations were normalized by TOC, this could be due to the low OC content in samples from Sweden. As discussed in earlier chapters, relative concentration of GDGTs in samples in this study are much higher compared to other studies (e.g., De Jonge et al., 2015; Dođrul Selver et al., 2012; Dođrul

Selver et al., 2015), most likely due to the lower TOC concentration (values in Table 7.3 compared to 8-13.6% in Kolyma River samples; Dođrul Selver et al., 2015).

GDGT proxies, pH and MAT, were also calculated. Comparing findings in three catchments in this study to literature, it was concluded that GDGT proxy pH is reliable, while MAT is either lower or higher than the findings in literature and should be used with care. This is in line with findings in De Jonge et al. (2016), reporting difference between annual mean air temperature and GDGT proxy MAT.

### **7.2.2.3 Source and nature of organic biomarkers**

Individual BHP compounds can be used to describe a certain environment. Therefore, specific BHPs were studied in more detail. Overall it seems that, despite different concentrations, organic biomarker content across the study sites was similar. For instance, the most abundant BHP compound in all three sites was BHT, followed by 35-aminotriol in Sweden and methylated BHT in Iceland and Greenland, BHT glucosamine and BHT cyclitol ether being other common biomarkers in samples. Moreover, in all study sites, adenosylhopane and adenosylhopane type 3 were the most abundant soil marker BHPs. Source organisms of the most abundant organic biomarkers in samples in this study and environments where they are found are presented in Table 4.4.

Further, as mentioned earlier, brGDGTs, produced by Acidobacteria and indicative of terrestrial OC (Weijers et al., 2009), almost entirely made up total GDGTs. Some crenarchaeol was also detected in few samples from Sweden and Greenland. According to Pitcher et al. (2010), crenarchaeol is produced by ammonia-oxidizing Archaea and is indicative of aquatic environments (Hopmans et al., 2004; Zell et al., 2013). This suggests that crenarchaeol detected in samples from Sweden and Greenland came from in situ fluvial production, perhaps being transferred to samples during spring melt events.

### **7.2.2.4 Soil development**

There was a significant difference between sampling locations of all sampling sites in Greenland, Iceland and Sweden for total BHP compounds (PERMANOVA p-value < 0.05) but no significant difference for total GDGTs (PERMANOVA p-value > 0.05) when grouping samples by their location along the transect (upstream, midstream and downstream).

The significant difference in total BHPs by sampling location along the downstream transects suggests that soil development has different dynamics based on the time of exposure after deglaciation, while the lack of a significant difference in total GDGTs indicates that this organic biomarker develops differently regardless of the time of exposure. This is in line with findings in Kim et al. (2011), suggesting that soil-specific BHPs and brGDGTs are synthesized by different microbial organisms depending on the depth of soil horizon. For this study, soil samples were collected from accessible topsoil, which have better developed soil-specific BHPs but not brGDGTs (Kim et al., 2011).

In agreement with the difference in samples by sampling location, no pattern of total GDGTs along the downstream transects across the three catchments was observed. On the contrary, looking at distribution of soil specific BHPs and thus  $R'_{\text{soil}}$  across the catchments, an upward trend along the Tarfalajaure transect in Sweden was observed, suggesting soil development over time. Meanwhile, in Iceland and in Greenland, there was no overall trend in total BHPs or the  $R'_{\text{soil}}$  distribution along the downstream transects. However, it seems that at the Vatnajökull ice cap soil development plateaus in older moraines, while at the oldest catchment, Zackenberg valley, no trend of OC or soil marker BHPs accumulation was observed, suggesting that in older catchments other factors and processes might be involved in soil development. These observations are discussed further in more detail.

#### **7.2.2.5 Accumulation of soil derived OC in three catchments so far**

The Tarfala valley in Sweden is the youngest catchment, representative of a bare catchment with very low TOC concentration. In this catchment, the absolute concentration of organic biomarkers ( $\mu\text{g/g}$  sediment) is low, while the relative concentration ( $\mu\text{g/g}$  TOC) is high due to the low OC content. The upward trend observed in the distribution of total BHPs ( $\mu\text{g/g}$  TOC), soil marker BHPs and the  $R'_{\text{soil}}$  along the Tarfalajaure transect indicates how soil derived OC accumulates initially without intervention of other external factors.

The Vatnajökull ice cap in Iceland is an older catchment compared to the Tarfala valley in Sweden. TOC content is much higher and increases with distance from glacier front, indicating an OC build-up along the downstream transects of studied glaciers. Total BHP concentration per g TOC was lower than that in Sweden but due to the higher TOC content the absolute BHP concentration was higher. As opposed to the TOC distribution, there was

no pattern of build-up of total BHPs or soil marker BHPs, indicating that accumulation of soil derived OC in this catchment is more complicated and could be affected by other external factors. The upside-down parabola pattern of the  $R'_{\text{soil}}$  distribution along the transects, suggests that soil development stalls after reaching a certain point, allowing microbes to produce non-soil specific biomarkers. Assuming that the moraines are representing different time slices, the youngest ones being closest to the glaciers and the oldest ones being the farthest, soil development reached this point pre-LIA (pre-LIA moraines at Virkisjökull (Everest et al., 2017); 2500-years old Stóralda moraine at the Svínafellsjökull glacier). It is suggested that (1) the recent ecosystem (after the LIA) across the glacier forelands of the Vatnajökull ice cap build up OC due to the accumulation of soil biomarkers, (2) older soils that have been developing pre-LIA, reach a point when OC accumulates due to the non-soil specific biomarkers.

The Zackenberg valley in Greenland is an older catchment compared to the sites in Sweden and Iceland. TOC concentration of soil samples in the valley is comparable to the ones recorded in the Icelandic soils, suggesting that soils in these two deglaciated areas reached a certain level of OC accumulation. Compared to Iceland, OC distribution did not follow a clear upward trend, suggesting that there are other processes involved in the accumulation of soil OC. It is worth mentioning that other factors such as erosion, weathering and latitude also play a role. Despite the similar TOC content, both absolute and relative BHP concentrations were higher than the values recorded across the soils collected from the Vatnajökull ice cap, indicative of more developed soils. Similar to the TOC, total BHPs and the  $R'_{\text{soil}}$  do increase downstream, reacting to the input from the tributaries that dilute organic biomarker signals. Moreover, it is suggested that at the Zackenberg valley, over time production of non-soil marker BHPs also affect the composition of OC.

### **7.2.3 Microbial analysis**

#### **7.2.3.1 Comparing microbial community structures in soil and sediment samples from Sweden, Iceland and Greenland**

Next, the microbial community structure of soil and sediment samples from three catchments were compared. It was expected that microbial community composition of

samples would be different by country, with changes to microbial diversity due to the time of exposure after deglaciation at the study sites.

### **7.2.3.2 Chemical composition of samples, and their effect on microbial diversity**

Principal component analysis was conducted on chemical properties of samples selected for further microbial community analysis. PC1 explained 36.0% and PC2 explained 20.4% of total variance. There was a strong negative correlation between PC1 and Fe, Mn, Co, Ca, Na, Cd, Al and a less strong negative correlation with Mg, Cu and Zn. PC2 had a strong negative correlation with TC, TOC, K, N, S and Pb, and negative correlation with As and Zn (Figure 7.3).

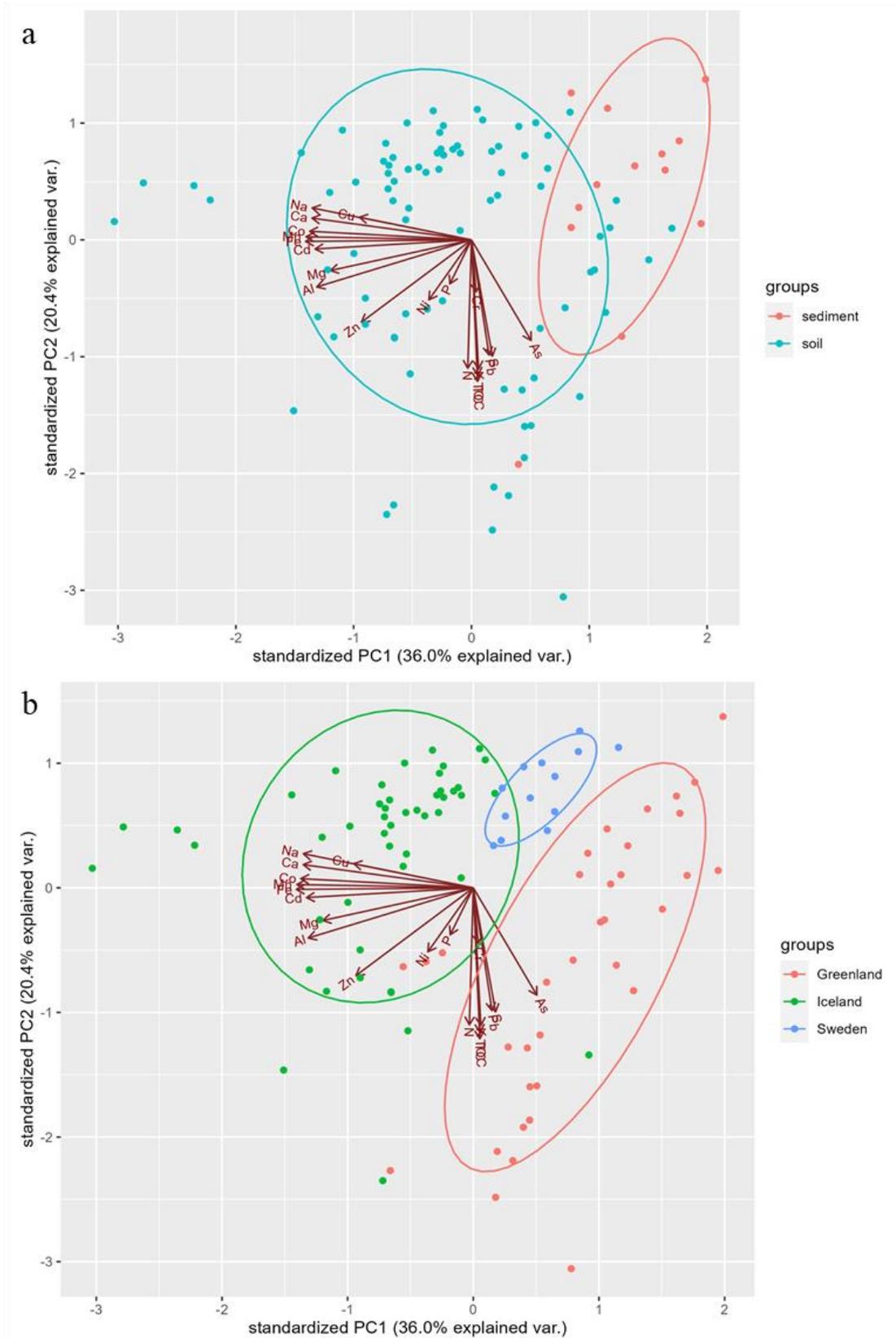


Figure 7.3. PCA ordination plots illustrating the discrimination between samples from Sweden, Iceland, and Greenland according to chemical properties by the (a) sample type and (b) country. The input data was standardised to have zero mean and variance one. The ellipses represent the core area added by confidence interval of 68%.

A PCA ordination plot (Figure 7.3a) and PERMANOVA analysis ( $p$ -value  $< 0.05$ ) showed that sediment and soil samples had different environmental properties. On Figure 7.3a sediment samples are mostly clustered together to the top right corner of the plot, away from metals, TC, TOC and TN, in line with previous findings. Previous observations and findings in other studies (e.g., Colombo et al., 2020) also suggest that soil and sediment samples have different chemical properties. Similarly, PCA ordination plot (Figure 7.3b) and PERMANOVA analysis ( $p$ -value  $< 0.05$ ) indicated that there was difference in environmental chemistry of samples by catchment. The plot shows that samples from Greenland are mostly clustered along PC2 (TC, TOC, TN, As), while samples from Iceland are mostly influenced by PC1, which correlates with most of the metals. Meanwhile, samples collected in Sweden are grouped away from vectors, in line with previous observations of lower concentration of most chemical elements in this study site. Since chemical properties of samples play an important role in the microbial diversity of samples (Venkatachalam et al., 2021), based on the PCA plots, it was expected to see difference in microbial community distribution of samples by the type of sample and by country.

Looking at the difference in bacteria by sample type on the NMDS ordination plot of family-level bacterial communities (Figure 7.4), sediment samples are mostly scattered to the left side of soil samples. Moreover, PERMANOVA analysis showed that there was a statistically significant difference between family-level bacterial communities of soil and sediment samples ( $p$ -value  $< 0.05$ ).

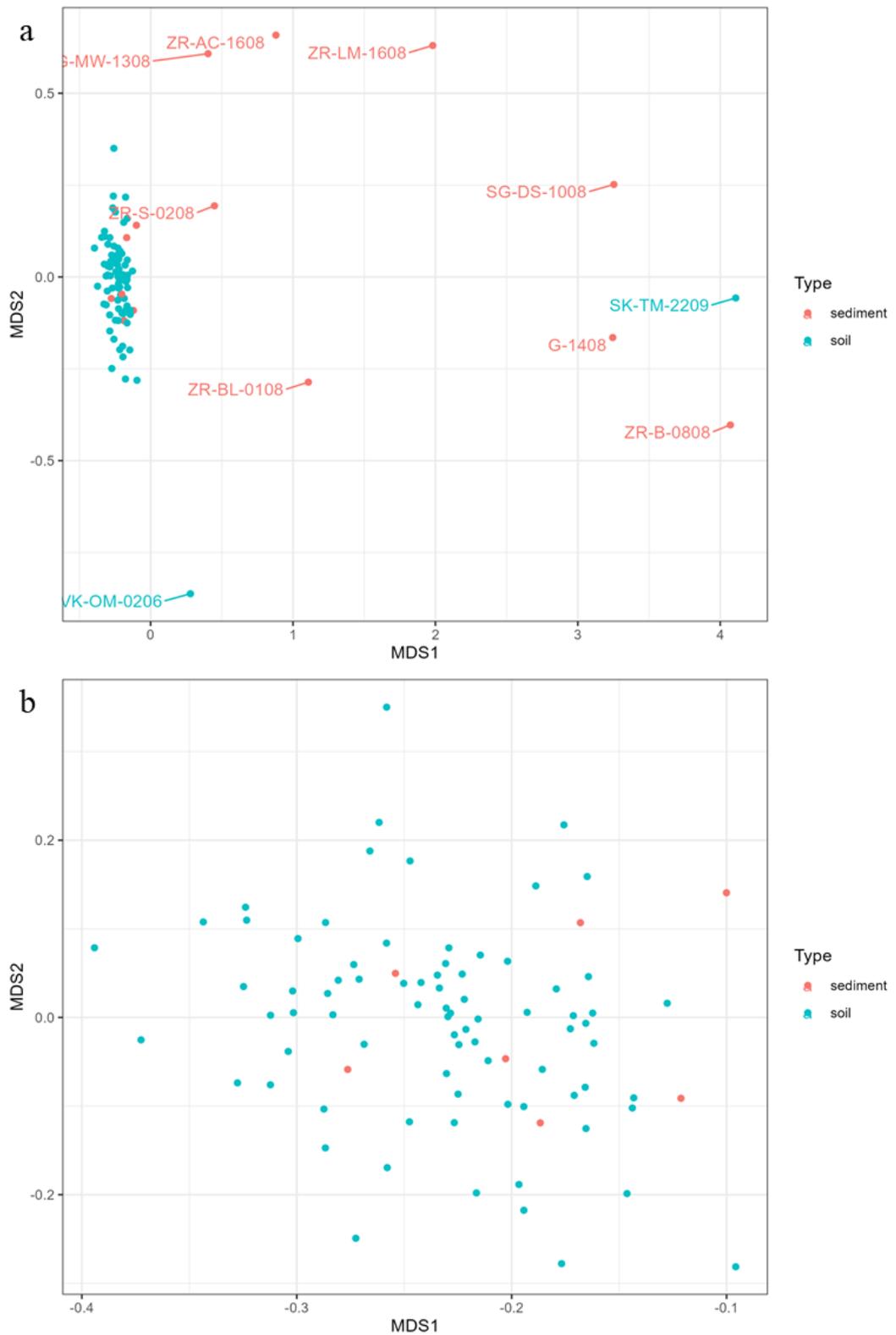


Figure 7.4. (a) Full scale and (b) zoomed-in two-dimensional NMDS plots of family-level bacterial communities in samples from Sweden, Iceland and Greenland by sample type. When the data values were larger than common abundance class scales, Wisconsin double standardisation was performed; when values looked very large, sqrt transformation was also performed; and distance matrix based on Bray–Curtis dissimilarity was obtained.

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There is no clear clustering of samples by site in each country in the two-dimensional NMDS plots of family-level bacterial communities (Figure 7.5). Overall, samples from Iceland are mostly grouped higher than samples from Sweden. Though not clearly shown on NMDS plots, PERMANOVA analysis showed that there was statistically significant difference between samples from each country ( $p$ -value  $< 0.05$ ).

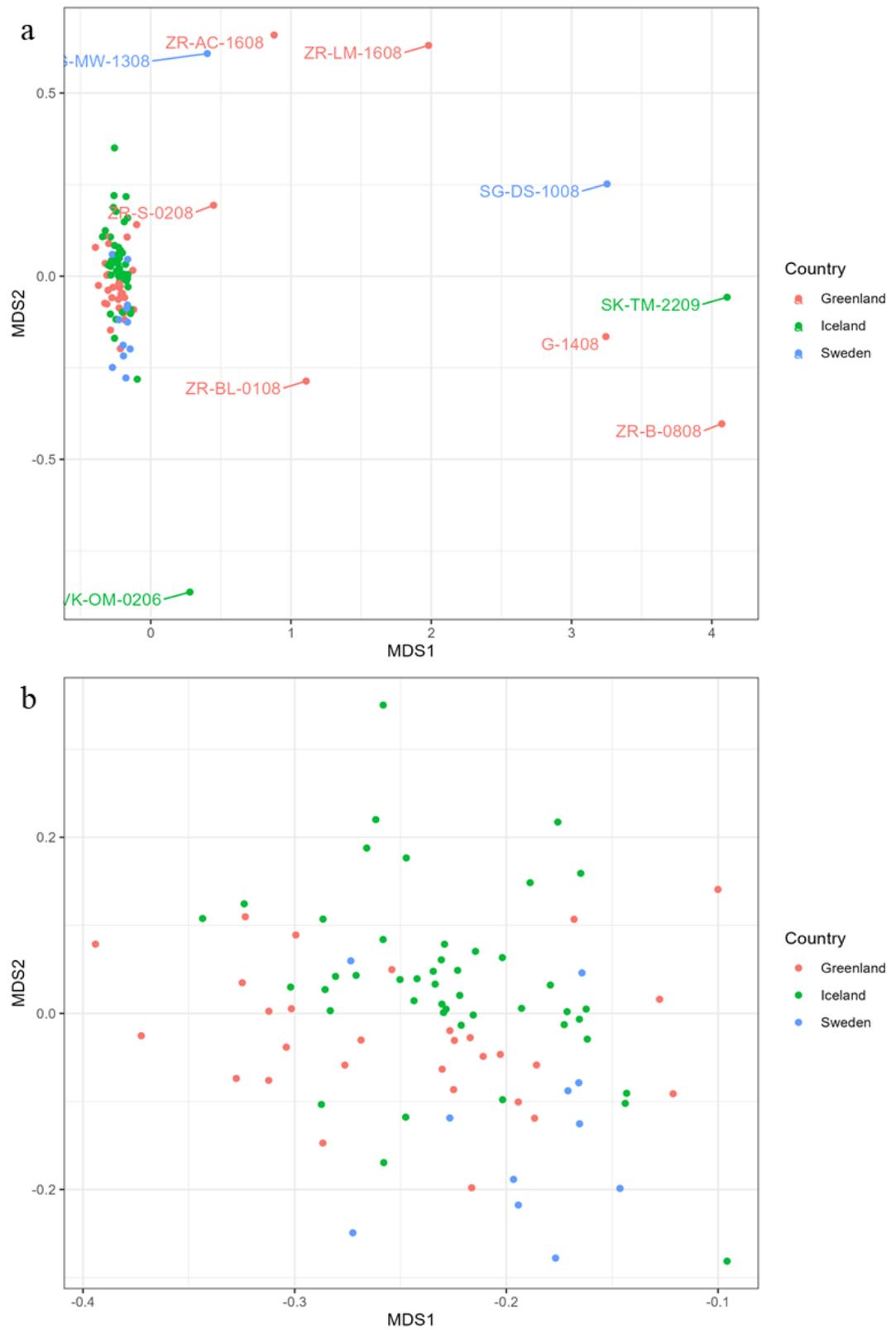


Figure 7.5. (a) Full scale and (b) zoomed-in two-dimensional NMDS plots of family-level bacterial communities in samples from Sweden, Iceland and Greenland by country. When the data values were larger than common abundance class scales, Wisconsin double standardisation was performed; when values looked very large, sqrt transformation was also performed; and distance matrix based on Bray–Curtis dissimilarity was obtained.

### 7.2.3.3 Alpha diversity

Shannon–Weiner diversity index and Chao1 species richness estimate values were plotted to compare the microbial alpha diversity in samples from Sweden, Iceland, and Greenland (Figure 7.6). Both the Shannon–Weiner diversity index and Chao1 species richness estimate indicated that samples from Iceland had the largest range in diversity in soil samples, having the lowest and the highest values. Soil samples from Sweden had slightly less diversity compared to samples from Iceland and Greenland. As for the sediment samples, Chao1 species richness estimate indicated that samples from Sweden had lower diversity than soil samples from this catchment. Meanwhile, in Greenland, some sediment samples (SS-D-0508 and SS-L-1308) had very high diversity, while others were low and comparable to that indicated in Sweden. Samples SS-D-0508 and SS-L-1308 were collected from vegetated delta and lake bank, indicating the importance of plant material, in line with findings in Mapelli et al. (2018), suggesting higher microbial diversity in rhizosphere soils.

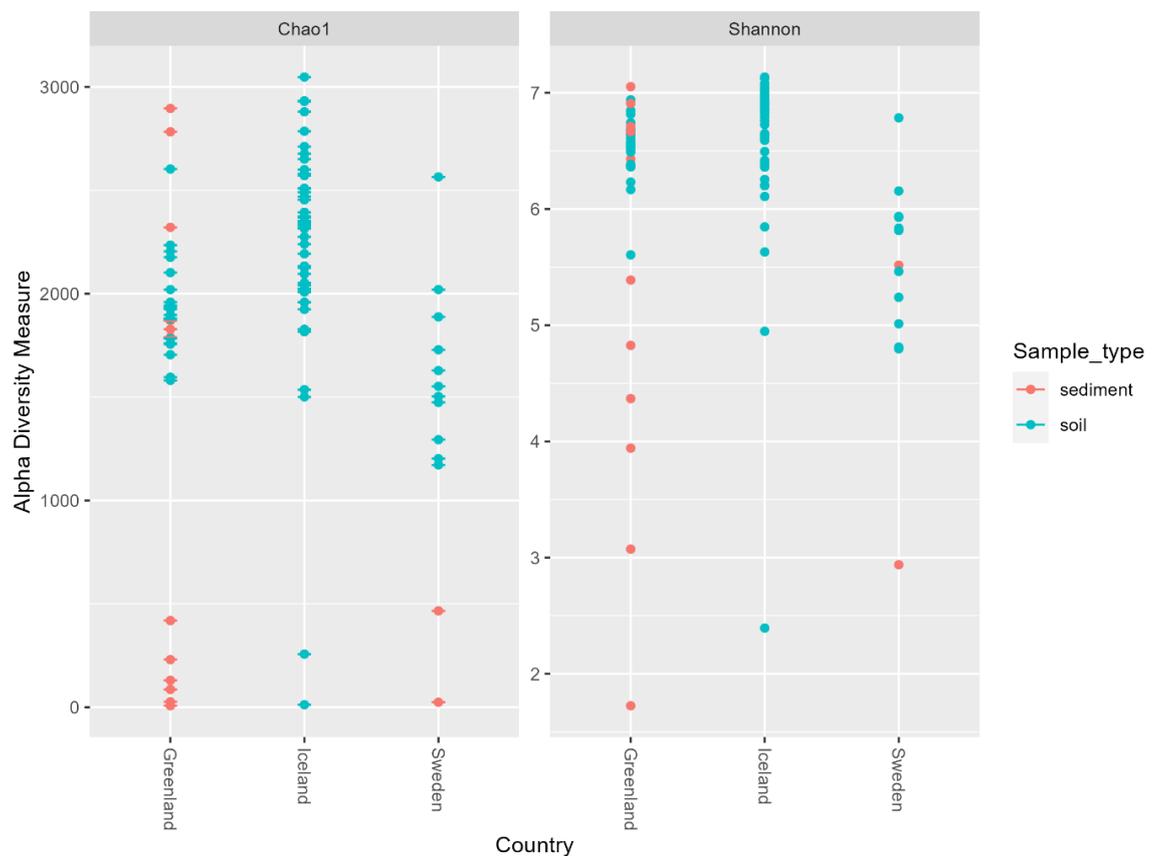


Figure 7.6. Alpha diversity (Shannon) and richness (Chao1) variation in soil and sediment samples from Sweden, Iceland, and Greenland.

As expected, the analysis showed that samples from Sweden, Iceland, and Greenland chosen for further microbial investigation had different environmental chemistry and microbial community composition. This is in line with findings in Mapelli et al. (2018), reporting variation in bacterial community by soil development stages. To understand this difference in a greater detail, bacterial communities across the three catchments in this study are compared.

#### **7.2.3.4 Bacterial diversity of samples from Sweden, Iceland and Greenland: Phylum-level community composition**

Major phyla identified in samples from Sweden, Iceland and Greenland are shown in Table 7.6 and Figure 7.7. There was some difference in relative abundance of bacterial 16S rRNA gene derived phyla between the samples from Sweden, Iceland, and Greenland (PERMANOVA,  $p$ -value < 0.05). Acidobacteria, Chloroflexi and Proteobacteria were dominant phyla in samples from all three countries. Relative abundance of Actinobacteria, Planctomycetes and Verrucomicrobia were also high, though their contribution to the total abundance varied. Samples collected in Sweden also had high relative abundance of the candidate phylum AD3 compared to samples from Iceland and Greenland. Generally, microbial composition of the samples is comparable to that identified in other cold habitats discussed in previous chapters (e.g., Chu et al., 2010; Costello and Schmidt, 2006; Ji et al., 2016; Mateos-Rivera et al., 2016; Schütte et al., 2010; Seok et al., 2016; Shivaji et al., 2011; Venkatachalam et al., 2021). These suggests that catchments investigated in this study are populated by microbial communities similar to other cold catchments and are descriptive of the polar ecosystem.

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Table 7.6. Relative abundance (%) of the dominant phyla in samples from Sweden, Iceland, and Greenland.

Country/ bacteria	candidate phylum AD3		<i>Acidobacteria</i>		<i>Actinobacteria</i>		<i>Chloroflexi</i>		<i>Planctomycetes</i>		<i>Proteobacteria</i>		<i>Verrucomicrobia</i>		<i>Bacteroidetes</i>	
Sweden	16.8	±13.6	17.2	±10.5	5.6	±4.3	15.7	±6.7	7.0	±3.5	15.4	±13.6	5.5	±2.1	2.5	±4.6
Iceland	1.5	±2.8	22.1	±6.3	10.5	±5.5	17.5	±4.5	10.7	±2.8	13.3	±4.4	11.0	±3.4	3.2	±5.2
Greenland	2.8	±4.0	19.6	±8.0	11.8	±4.4	16.3	±6.2	8.1	±3.1	15.4	±11.0	14.2	±5.5	3.1	±5.1

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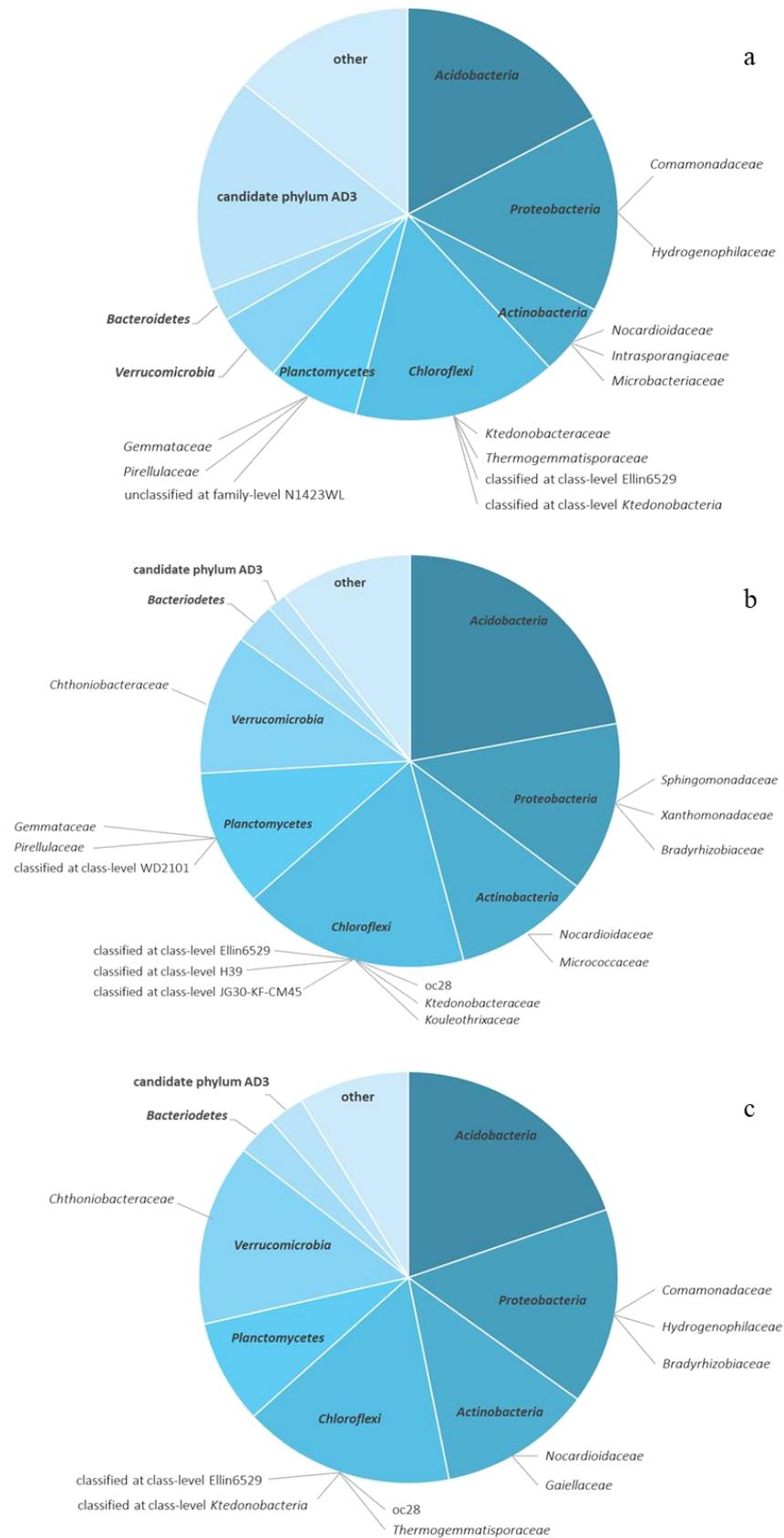


Figure 7.7. Average relative abundance of bacterial phyla (bold font) and previously discussed most abundant family-level bacteria (normal font) in each phylum in sediment and soil samples from (a) Sweden, (b) Iceland, and (c) Greenland.

Figure 7.7 shows that bacterial phyla identified in samples from Iceland and Greenland are similar, while samples from Sweden have different average phylum distribution mostly due to the high relative abundance of the candidate phylum AD3 (Table 5.6). High relative abundance of the candidate phylum AD3 in samples from Sweden highlights the uniqueness of this catchment compared to the catchments in Iceland and Greenland and other polar and alpine studies. Since Sweden is the youngest catchment in this study, it is predicted that the microbial community in this catchment may evolve to be more like the ones detected in Iceland and Greenland. Moreover, it is suggested that bacterial communities have homogenized in samples from Iceland and Greenland. This is in line with findings in Schütte et al. (2010), suggesting that bacterial communities stabilize in deglaciated soils older than 150 years. Moreover, even though catchments in Sweden, Iceland, and Greenland are colonized by similar phyla, family-level bacteria inhabiting each site are different (Figure 7.7, see previous chapters for more detail). This suggests that each catchment in this study is a habitat for different family-level bacteria, and predictions can be made only regarding the establishment of microbial communities at the phylum-level.

Further, it was observed that there was some difference between soil and sediment samples as well (PERMANOVA,  $p$ -value  $< 0.05$ ). For instance, sediment samples from Sweden had high relative abundance of Bacteroidetes ( $12.3 \pm 5.2\%$ ), as well as much higher relative abundance of Proteobacteria ( $44.2 \pm 11.2\%$ ) compared to soil samples ( $0.8 \pm 0.5\%$  – Bacteroidetes,  $10.2 \pm 3.8\%$  – Proteobacteria). Similarly, in Greenland, Proteobacteria had higher relative abundance in sediment samples ( $23.4 \pm 14.8\%$ ) compared to soil samples ( $10.7 \pm 2.9\%$ ). Mateos-Rivera et al. (2016) also reported that Proteobacteria was more abundant in recently deglaciated soils in Norway. Meanwhile, Bacteroidetes was prevalent in glacial ice and snow samples in Alaska (Choudhari et al., 2014), as well as proglacial sediment samples (Sheik et al., 2015), in agreement with findings in this study. This suggests that at least in catchments in Sweden and Greenland, while being dominant in sediment samples, relative abundance of Bacteroidetes and Proteobacteria decreases as other bacteria colonize older soils.

Though there was a difference between the microbial community composition of sediment and soil samples, particular pattern of microbial community succession along any of the transects across the three catchments was not observed. It is assumed that this is due to the adaptation and stabilisation of microbial communities in soils after deglaciation, as proposed by Schütte et al. (2010).

#### **7.2.3.5 Relationship between microbial community and organic biomarkers**

In previous chapters an attempt was made to establish a relationship between the current microbial communities inhabiting the catchments and organic biomarkers, using knowledge of source organisms that produce specific biomarkers (Table 4.4). Some biomarkers detected in samples were linked to some findings about the microbial communities inhabiting catchments. The main challenge was that the relative abundance of most of these bacteria was low, apart from Acidobacteria, which was present in high concentrations in all three catchments (up to 30%) and is suggested to produce brGDGTs (Weijers et al., 2009). Other bacteria that had relatively high relative abundance in samples from Iceland and Greenland and are linked to production of specific BHPs are some members of Proteobacteria (see previous chapters 4, 5 and 6 for more detail). Moreover, some organisms produce more than one BHP compound (Table 4.4), making it more complicated to establish a relationship between a bacterium and a specific biomarker. This suggests that processes taking place in the catchments in this study and perhaps in other catchments in the Arctic and Subarctic regions are complex, and more studies aimed at establishing the link between bacteria and specific organic biomarkers should be carried out.

### **7.3 Research limitations**

Some research limitations were encountered while conducting this study. There was a lack of soil samples along transects in Sweden and Iceland. This was due to the limited time allocated for soil sampling, as the initial aim of the study was to understand meltwater chemistry. Whilst the transects do not represent a full chronosequence of soil development, they provide understanding of organic biomarker and bacteria distribution along transects from recently deglaciated areas up to areas that have been ice free for

several millennia. In order to address this limitation, future sampling campaigns in study sites in this thesis should have higher spatial sample resolution. Moreover, when analysing triplicate (sometimes duplicate) samples, some samples had large relative standard deviation (SD of TOC data in sample SS-LI-1408 was 73%). This could be due to natural variations in the environment as samples were collected next to each other (within a radius of 0.5-1 m), rather than from the same spot at each location. Therefore, data from studies with one replicate should be considered with care. Future studies should collect replicates closer to each other and analyse more replicates if possible to overcome this limitation.

Some limitations were due to the lack of already available data. For instance, lack of available sequence data in gene banks led to not being able to identify some bacteria. To overcome this limitation, bacteria were analysed at the family level. Future microbial studies in polar and alpine regions will help to close this gap in sequencing data, adding more information about different taxa in the gene banks. Next, due to the lack of studies linking organic biomarkers and their source organisms, it was difficult to establish a link between some living bacteria and biomarkers. In order to overcome this limitation, more studies aimed at establishing this relationship should be conducted.

## **7.4 Conclusion**

This chapter was a synthesis of findings in previous chapters, where an attempt was made to compare the three catchments (Sweden, Iceland, and Greenland) investigated in this study. As expected, it was observed that soil and sediment samples had different environmental chemistry and were inhabited by different bacteria. Moreover, the catchments differ by environmental chemistry, organic biomarker concentrations and microbial community composition and diversity. TOC concentration was lowest in Sweden, and had comparable values in Iceland and Greenland, suggesting stabilisation of OC accumulation in older catchments. Concentration of both total BHPs and GDGTs, in soils increased from youngest to oldest catchment, suggest accumulation of soil derived OC with the time of exposure after deglaciation. However, the lack of a particular pattern in the  $R'_{\text{soil}}$  along the downstream transects in Iceland and Greenland, as well as similar bacterial phyla in these two catchments, suggests that soil OC stabilizes and becomes homogeneous over time.

## 8 Conclusion

The aim of this study was to understand how microbial metabolism and bioavailability of OC affects accumulation of soil derived OC in three nutrient rich deglaciated catchments (Zackenberget in Greenland, Vatnajökull ice cap in Iceland, and Tarfala in Sweden). To achieve this aim, soil and sediment samples from pro-glacial areas of the catchments were studied in detail for catchment chemistry, including OC and metals, organic biomarkers and microbial community composition. Chapters 4, 5, 6, and 7 aimed to address the sub-aims identified in introduction.

This is the first study to look at organic biomarkers to understand the source and nature of soil OC in deglaciated catchments. Moreover, current microbial community was investigated, creating an opportunity to establish relationship between the current bacterial population and organic biomarkers of past life in post-glaciated areas.

This chapter aims to provide summary of chapters 4, 5, 6, and 7, and an overall conclusion to this PhD research project. Further, recommendations for future research are provided based on the knowledge gaps encountered throughout this study.

**Chapter 4** discussed findings at the Tarfala valley in Sweden. Tarfala valley is a catchment with no to low vegetation in this study. Two sub-areas were studied in detail: Storglaciären glacier foreland, which has been deglaciated since the glacier maximum in 1910 (Holmlund et al., 2005) and the Tarfalajaure transect, which could have been deglaciated for as long as 9000-8500 years BP (Karlen, 1979), though the exact date of deglaciation of the surroundings of the lake is unknown.

Throughout all three transects (including Isfallsglaciären), soils had higher OC than sediments. Despite potentially being deglaciated for a long time, soil samples from Sweden had low OC content (0 - 1.08±0.09%), especially those that have not been exposed for a

long time. Abundance of soil marker BHPs increased along the Tarfalajaure downstream transect, and brGDGTs were only detected in samples with higher TOC content. Moreover, microbial diversity was also higher in older soil samples compared to sediments, indicative of accumulation of OC in soils in this catchment over time. The main bacteria detected in samples from Sweden were candidate phylum AD3, Acidobacteria, Actinobacteria, Chloroflexi, Proteobacteria, and Planctomycetes. Previously, candidate phylum AD3 was the most abundant phylum only in samples from the Mitchell Peninsulain Antarctica (Ji et al., 2016). This suggests that the Tarfala valley in Sweden might have a unique bacterial community and should be studied in more detail. No variation in microbial community along the downstream transect was observed, suggesting that microbial community in deglaciated catchments homogenises and adapts to the new environment after deglaciation. Linking biomarkers with bacterial community showed that soil marker BHPs in samples from Sweden were mainly produced by Rhodospirillaceae or purple non-sulfur bacteria, while brGDGTs were produced by Acidobacteria.

The Tarfala valley in Sweden is an example of a bare and relatively young catchment in the Arctic, which provides an opportunity to understand the microbial community establishment and OC accumulation in recently deglaciated soils.

**Chapter 5** was aimed at studying soil OC at the more developed catchment, Vatnajökull ice cap in Iceland. Six glaciers at the southern margin of the ice cap were studied in this chapter: Svínafellsjökull, Virkisjökull, Kviarjökull, Fjallsjökull, Skaftafellsjökull and Skeiðarárjökull. The youngest samples along the downstream transects were from recently deglaciated areas, while the oldest ones were as old as 5000-6000 years old (Virkisjökull; Guðmundsson, 1998).

The Vatnajökull ice cap is older catchment compared to the Tarfala valley in Sweden. OC content at the Vatnajökull ice cap was higher ( $0.01 - 5.24 \pm 2.68\%$ ) and increased along the downstream transects. Distribution of soil marker BHPs along the glacier forelands in Iceland suggests that soil development plateaus out after reaching a certain stage, soil marker BHPs being overtaken by other BHP compounds. Similar to the findings in Sweden, brGDGTs did not follow a pattern along the downstream transects, suggesting that this organic biomarker is not affected by the time of exposure after deglaciation. Moreover,

microbial community distribution did not follow a trend along the downstream transects either, suggesting that similar to the observations in Sweden, bacterial community has homogenised over time. Acidobacteria, Actinobacteria, Chloroflexi, Planctomycetes, Proteobacteria and Verrucomicrobia were the most abundant phyla in samples from Iceland. The relationship between bacteria and organic biomarkers, indicated that soil marker BHPs in this catchment were produced by Bradyrhizobiaceae, Hyphomicrobiaceae, and Nitrosomonadaceae, while brGDGTs were produced by Acidobacteria.

Overall, Vatnajökull ice cap in Iceland has recently deglaciated areas (after the LIA) still accumulating organic biomarkers and areas that have been ice free for longer having more established soils.

**Chapter 6** investigated soil OC in the oldest catchment in this study, Zackenberg valley in Greenland, which has been ice free since the Early Holocene (ca. 10.5 ka) (Garcia-Oteyza et al., 2022). This chapter looked at the accumulation of soil derived OC in soils along the Zackenberg valley transect.

The Zackenberg valley transect is an older catchment compared to study sites in Sweden and Iceland. No particular pattern in the downstream distribution of OC ( $0.01 \pm 0.01$  -  $4.88 \pm 3.56\%$ ) and organic biomarkers was observed, suggesting stabilisation of OM due to the age of exposure. Moreover, microbial community did not follow a trend along the downstream transects away from the glacier front, also suggesting that bacterial community has homogenised over time. Acidobacteria, Chloroflexi, Proteobacteria, Verrucomicrobia and Actinobacteria were the most abundant phyla in samples from Greenland. Soil marker BHPs in Greenland were produced by Bradyrhizobiaceae, Hyphomicrobiaceae, and Nitrosomonadaceae, which are the same bacteria linked to soil specific BHPs in study sites in Iceland. This suggests that similar bacterial communities responsible for production of soil marker BHPs might have been established in catchments in Iceland and Greenland. Similar to the observation in Sweden and Iceland, brGDGTs in Greenland could have been produced by Acidobacteria.

The Zackenberg valley is an example of an older catchment, where OC, organic biomarkers, and microbial community have stabilised over time.

**Chapter 7** was aimed at bringing together and synthesising findings from the three catchments. Across the three catchments, soils had higher OC content compared to sediment samples, as well as different metal concentrations, which led to differences in microbial communities. Samples from Sweden, had the lowest OC content, while OC concentrations in Iceland and Greenland were comparable, indicative of OC content reaching a certain value and plateauing in older catchments. Variation in the distribution of soil marker BHPs across the catchments suggests that soil marker BHPs accumulate in recently deglaciated areas along the glacier foreland transects, followed by stabilisation in older soils. Meanwhile, time of exposure after deglaciation does not affect the distribution of microbial community composition. However, more unique bacterial community has been established in the younger catchment (Sweden) compared to more developed catchments, (Iceland and Greenland).

An attempt to establish the relationship between bacterial community and organic biomarkers indicated that while some bacteria could have been responsible for production of certain organic biomarkers, most bacteria had low relative abundance, while some (i.e. Acidobacteria - a potential source of brGDGTs) had high relative abundance. This suggests that even though detected in small quantities, some bacteria could have produced organic biomarkers that have accumulated over time, or the catchments could have been re-colonised, therefore, suggesting that current bacteria detected in the catchments are not representative of past bacteria responsible for the production of organic biomarkers. Moreover, since a specie of Acidobacteria responsible for the production of brGDGTs is unknown, it is difficult to establish a firm relationship between this phylum and the organic biomarker. Based on the observations in this study, the main hypothesis set at the beginning of this thesis "Organic biomarkers and microbial analysis can be used to understand accumulation of soil derived OC in newly-developed soils following glacier retreat in northern latitudes" is rejected. This study showed that while the hypothesis is true in some instances, such as OC accumulation in Iceland and soil marker BHPs in Sweden, processes of microbial colonization and OC accumulation are unique to each catchment and are complex. While time of exposure after deglaciation is an important factor, there are other factors such the bedrock geology, weathering, erosion and proximity to proglacial lakes that can affect accumulation of SOC and soil development.

Further, the following observations can be concluded based on the findings of this study:

1. The Tarfala valley catchment, as well as recently deglaciated areas in Iceland, rapidly accumulate OC, as well as soil marker BHPs, subsequently stabilising over time in older soils in Iceland and Greenland.
2. BrGDGTs in deglaciated areas are present in areas with sufficient OC content.
3. Microbial communities are different in the younger catchment in Sweden compared to older soils in Iceland and Greenland. In catchments in Iceland and Greenland microbial communities are generally representative of other Arctic catchments and consist of members of Acidobacteria, Actinobacteria, Chloroflexi, Proteobacteria and Verrucomicrobia,
4. Soil marker BHPs in Sweden were mainly produced by Rhodospirillaceae or purple non-sulfur bacteria and by members of Bradyrhizobiaceae, Hyphomicrobiaceae, and Nitrosomonadaceae in Iceland and Greenland, while presence of brGDGTs in all three catchments could be due to Acidobacteria.

This study attempted to understand source and nature of accumulation of OC in soils in three deglaciated catchments in the Arctic and Subarctic regions. However, in order to gain a better understanding of accumulation of soil derived OC in the catchments investigated in this study, as well as other regions in the Arctic and Subarctic, further research should be conducted addressing the following issues:

1. Conducting a larger scale study of microbial community of sediment and soil samples from the Tarfala valley, Vatnajökull ice cap and Zackenberg valley.
2. Conducting more microbial studies in polar and alpine regions to contribute to the expansion of the gene banks.
3. Conducting a study to identify which species of Acidobacteria produce brGDGTs.
4. Conducting more studies aimed at establishing relationships between organic biomarkers and specific organisms producing them.
5. Investigating carbon implication of accumulation of SOC in post-glaciated areas.

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## Appendices

Appendix 1. pH, temperature (T), electric conductivity (EC), and discharge data from glacier meltwater streams and proglacial lakes in Sweden, Iceland and Greenland.

Sampling Site	Location at the site	Date	pH	T, °C	EC, mS/cm	Discharge, method 1, m <sup>3</sup> /s	Discharge, method 2, m <sup>3</sup> /s
<b>SWEDEN</b>							
Storglaciären	Main meltwater stream	08/08/2018	8.17	2.4	0.03	1.39	2.2
		16/08/2018	8.47	2.4	0.01	0.85	1
	Southern meltwater stream	10/08/2018	8.45	1.8	0.01	0.17	0.17
		15/08/2018	8.41	1.5	0.01	0.1	0.09
	Storglaciären meltwater streams before joining Tarfalajåkk river	09/08/2018	7.96	4.4	0.01	-	-
		16/08/2018	8.2	2.2	0.01	2.91	1.78
	Storglaciären meltwater streams and Tarfalajåkk river joined together	09/08/2018	8.5	4.3	0.04	1.29	2.45
16/08/2018		7.78	5	0.02	5.48		
Isfallsglaciären	Northern meltwater steam	12/08/2018	8.09	2.9	0.01	0.63	0.58
		18/08/2018	8.56	2.4	0.01	1.06	0.98
	Southern meltwater stream	12/08/2018	8.1	1.6	0	1.67	1.78
		18/08/2018	8.68	2.3	0	0.77	0.68
	Eastern lake – closest to the glacier, poorly fed by meltwater streams	12/08/2018	7.65	6.6	0.01	-	-
	Main lake	11/08/2018	8.42	4.1	0.01	-	-
		18/08/2018	8.13	4.7	0.01	-	-
	Downstream river	11/08/2018	8.06	3.7	0.01	0.76	0.76
18/08/2018		7.78	4.4	0.01	0.88	0.89	
Kebnepakte-glaciären	Meltwater stream	13/08/2018	8.55	1.9	0.01	0.46	0.43
Sydöstra Kaskatjåkkoglaciären	Meltwater stream	14/08/2018	8.13	3.8	0.02	0.25	0.23
Tarfalajaure	Lake, eastern bank	13/08/2018	7.95	6.9	0.02	-	-
Tarfalajåkk	Downstream from Tarfalajaure lake	14/08/2018	7.85	7.4	0.02	1.24	2.13

	Tarfalajákk river after joining Isfallsglaciären meltwater stream	19/08/2018	8.14	7.1	0.02	2.55	2.19
	Lake at the Tarfalajákk river	19/08/2018	7.92	7.9	0.02	-	-
	Tarfalajákk river before joining Storglaciären meltwater streams	10/08/2018	7.79	8.5	0.02	2.51	-
		19/08/2018	7.83	7.1	0.02	3.31	3.22
<b>ICELAND</b>							
Virkiðsjökull	Meltwater stream draining the glacier	26/05/2018	6.69	2.4	-	4.94	4.34
		28/05/2018	6.63	6.8	0.04	8.14	7.17
		02/06/2018	-	-	-	9.57	8.75
		19/09/2019	7.76	0.9	0.06	2.53	3.87
	Proglacial lake	26/05/2018	7.14	3.5	-	-	-
		28/05/2018	6.41	6.6	0.05	-	-
		19/09/2019	7.95	1.7	0.05	-	-
	Downstream river	27/05/2018	6.57	9		7.73	4.6
		28/05/2018	6.06	13.1	0.05	11.77	15.97
20/09/2019		7.54	2.7	0.04	4.94	8.42	
Svínafellssjökull	Proglacial lake	21/09/2019	7.87	6.7	0.15	-	-
	Downstream river	29/05/2018	-	-	-	9.97	10.91
		30/05/2018	-	-	-	7.15	8.17
		30/05/18 evening	-	-	-	6.57	7.14
		03/06/2018	-	-	-	9.58	13.64
		21/09/2019	9.28	0.9	0.06	3.45	19.49
Háalda	River	02/06/2018	-	-	-	7.02	12.57
Skaftafellssjökull	Proglacial lake	22/09/2019	8	3.5	0.04	-	-
	Downstream river	22/09/2019	8.07	1.8	0.04	5.54	25.83
Fjallsjökull	Proglacial lake	23/09/2019	7.87	5	0.1	-	-
	Downstream river	23/09/2019	7.48	4.7	0.05	2.04	2.65
Kviarjökull	Proglacial lake	23/09/2019	7.11	2.1	0.06	-	-
	Downstream river	23/09/2019	7.65	1.5	0.04	2.33	12.13
<b>GREENLAND</b>							
A.P. Olsen Land glacier meltwater stream	Meltwater stream draining the glacier	14/08/2019	6.36	5	0.02	5.1	10.7
River at Antonsens Hytte	River at Antonsens Hytte	05/08/2019	6.94	12	0.01	0.61	2.67

Store Sjø Lake	Delta at the inlet where glacier meltwater stream inflows the lake	05/08/2019	6.9	10.5	0.01	3.74	12.59
	Inlet area of the lake	14/08/2019	6.81	11.2	0.02	-	-
	Middle of the lake transect	10/08/2019	6.42	8.7	0.01	-	-
	Eastern lobe of the lake in the mid-section transect	13/08/2019	6.43	10.2	0.02	-	-
	Lake outlet	10/08/2019	6.42	7.2	0.01	0.89	1.81
Zackenber River	Zackenber River after outflowing from the lake	15/08/2019	6.99	9.1	0.03	0.64	8.45
	Before Lindemanselven River inflows to Zackenber River	01/08/2019	6.53	10.5	0.03	4.78	23.00
	After Lindemanselven and Palnatokeelv rivers inflow to Zackenber River	16/08/2019	6.65	6.7	0.1	2.59	11.04
	Aucellaelv River inflows to Zackenber River	16/08/2019	6.43	7.2	0.05	3.48	11.12
	Bridge by the station	01/08/2019	6.68	11.2	0.03	6.93	35.44
		08/08/2019	6.49	9.1	0.04	5.82	28.10
		19/08/2019	6.73	7.5	0.04	3.59	24.57
Station	02/08/2019	6.89	10.9	0.04	8.5	32.42	

Appendix 2. Elements measured by ICP-OES, wavelength in nm and calibration standard details.

Element	Analysed wavelength (nm)	C1, ppm	C2, ppm	C3, ppm	C4, ppm	C5, ppm	C6, ppm	C7, ppm	C8, ppm
Na	589.592	0.100	0.500	1.000	10.000	20.000	50.000	200.000	500.000
K	769.896	0.100	0.500	1.000	10.000	20.000	50.000	200.000	500.000
Mg	285.213	0.050	0.250	0.500	5.000	10.000	25.000	100.000	250.000
Ca	422.673	0.100	0.500	1.000	10.000	20.000	50.000	200.000	500.000
Cr	267.716	0.020	0.100	0.200	2.000	4.000	10.000	40.000	100.000
Mn	257.610	0.020	0.100	0.200	2.000	4.000	10.000	40.000	N/A
Fe	240.488	0.100	0.500	1.000	10.000	20.000	50.000	200.000	500.000
Co	228.616	0.020	0.100	0.200	2.000	4.000	10.000	40.000	100.000
Ni	231.604	0.020	0.100	0.200	2.000	4.000	10.000	N/A	N/A
Cu	324.754	0.050	0.250	0.500	5.000	10.000	25.000	100.000	250.000
Zn	206.200	0.020	0.100	0.200	2.000	4.000	10.000	40.000	100.000
Cd	214.438	0.020	0.100	0.200	2.000	4.000	10.000	40.000	100.000
Al	396.152	0.100	0.500	1.000	10.000	20.000	50.000	200.000	500.000
P	178.284	0.050	0.250	0.500	5.000	10.000	25.000	100.000	250.000
S	182.034	0.050	0.250	0.500	5.000	10.000	25.000	100.000	250.000
As	189.042	0.020	0.100	0.200	2.000	4.000	10.000	40.000	100.000
Pb	220.353	0.020	0.100	0.200	2.000	4.000	10.000	40.000	100.000
Mo	202.030	0.020	0.100	0.200	2.000	4.000	10.000	40.000	100.000

Appendix 3. Specifications of the certified reference material used for ion chromatography analysis.

Measurand	Value in mg/L		$\mu$	N
Calcium	13.6	$\pm$	1.3	155
Chloride	1.40	$\pm$	0.15	141
Magnesium	2.83	$\pm$	0.26	153
Nitrate + Nitrite (as N)	0.358	$\pm$	0.036	178
Potassium	0.502	$\pm$	0.072	131
Silica (as Si)	1.06	$\pm$	0.11	111
Sodium	1.43	$\pm$	0.18	136
Sulfate (as SO <sub>4</sub> )	3.50	$\pm$	0.50	138
Total Nitrogen	0.430	$\pm$	0.055	112

Appendix 4. Dilution of anion and cation calibration standards used for calibrating the ion chromatography equipment.

<b>Anions</b>		<b>Cations</b>	
Calibration 6	1:5	Calibration 6	1:10
Calibration 5	1:10	Calibration 5	1:20
Calibration 4	1:20	Calibration 4	1:100
Calibration 3	1:100	Calibration 3	1:1000
Calibration 2	1:200	Calibration 2	1:2000
Calibration 1	1:2000	Calibration 1	1:10000

Appendix 5. Elements measured by ICP-OES, wavelength in nm and calibration details. Detected metals are shaded in grey. C1 is calibration 1, C2, C3, C3.5, C4 and C5 – calibration 2, 3, 3.5, 4 and 5 accordingly.

Element	Analysed wavelength (nm)	C1, ppm	C2, ppm	C3, ppm	C3.5, ppm	C4, ppm	C5, ppm
Phosphorous (P)	178.284	0.050	0.250	0.500	2.500	5.000	10.000
Sodium (Na)	589.592	0.100	0.500	1.000	5.000	10.000	20.000
Potassium (K)	769.896	0.100	0.500	1.000	5.000	10.000	20.000
Calcium (Ca)	422.673	0.100	0.500	1.000	5.000	10.000	20.000
Chromium (Cr)	267.716	0.020	0.100	0.200	1.000	2.000	4.000
Molybdenum (Mo)	202.030	0.020	0.100	0.200	1.000	2.000	4.000
Manganese (Mn)	257.610	0.020	0.100	0.200	1.000	2.000	4.000
Iron (Fe)	259.940	0.100	0.500	1.000	5.000	10.000	20.000
Cobalt (Co)	228.616	0.020	0.100	0.200	1.000	2.000	4.000
Nickel (Ni)	231.604	0.020	0.100	0.200	1.000	2.000	4.000
Copper (Cu)	324.754	0.050	0.250	0.500	2.500	5.000	10.000
Zinc (Zn)	206.200 and 213.856	0.020	0.100	0.200	1.000	2.000	4.000
Cadmium (Cd)	228.802	0.020	0.100	0.200	1.000	2.000	4.000
Aluminium (Al)	396.152	0.100	0.500	1.000	5.000	10.000	20.000
Sulfur (S)	182.034	0.050	0.250	0.500	2.500	5.000	10.000
Arsenic (As)	189.042	0.020	0.100	0.200	1.000	2.000	4.000
Lead (Pb)	220.353	0.020	0.100	0.200	1.000	2.000	4.000
Magnesium (Mg)	280.270	0.050	0.250	0.500	2.500	5.000	10.000

Appendix 6. Sampling sites at the Tarfala valley, Sweden.

Water and sediment					
No	Name	Sampling Site	Location at the site	Coordinates	Date
1	SG-R-0808	Storglaciären	Main meltwater stream	67°54'11"N 18°36'18"E	08-08-2018
2	SG-MX-0908	Storglaciären	Storglaciären meltwater streams and Tarfalajåkk river joined together	67°54'08"N 18°37'09"E	09-08-2018
3	SG-DS-0908	Storglaciären	Storglaciären meltwater streams before joining Tarfalajåkk river	67°54'10"N 18°37'02"E	09-08-2018
4	SG-SS-1008	Storglaciären	Southern meltwater stream	67°54'04"N 18°36'14"E	10-08-2018
5	MS-1008	Tarfalajåkk	Tarfalajåkk river before joining Storglaciären meltwater streams	67°54'11"N 18°37'05"E	10-08-2018
6	IG-DL-1108	Isfallsglaciären	Main lake	67°54'57"N 18°35'50"E	11-08-2018
7	IG-DR-1108	Isfallsglaciären	Downstream river	67°54'53"N 18°35'59"E	11-08-2018
8	IG-NS-1208	Isfallsglaciären	Northern meltwater stream	67°54'58"N 18°35'15"E	12-08-2018
9	IG-SS-1208	Isfallsglaciären	Southern meltwater stream	67°54'49"N 18°35'11"E	12-08-2018
10	IG-EL-1208	Isfallsglaciären	Eastern lake – closest to the glacier, poorly fed by meltwater streams	67°54'56"N 18°35'29"E	12-08-2018
11	KG-MW-1308	Kebnepakteglaciären	Meltwater stream	67°55'32"N 18°34'30"E	13-08-2018
12	T-L-1308	Tarfalajaure	Lake	67°55'14"N 18°35'20"E	13-08-2018
13	CB-1408	Sydöstra Kaskatjåkkoglaciären	Meltwater stream	67°55'41"N 18°36'17"E	14-08-2018
14	MS-TL-1408	Tarfalajåkk	Downstream from Tarfalajaure lake	67°55'10"N 18°35'52"E	14-08-2018
15	SG-SS-1508	Storglaciären	Southern meltwater stream	67°54'04"N 18°36'14"E	15-08-2018
16	SG-R-1608	Storglaciären	Main meltwater stream	67°54'11"N 18°36'22"E	16-08-2018
17	SG-DS-1608	Storglaciären	Storglaciären meltwater streams before joining Tarfalajåkk river	67°54'13"N 18°36'57"E	16-08-2018
18	SG-MX-1608	Storglaciären	Storglaciären meltwater streams and Tarfalajåkk river joined together	67°54'06"N 18°37'20"E	16-08-2018
19	IG-SS-1808	Isfallsglaciären	Southern meltwater stream	67°54'49"N 18°35'13"E	18-08-2018
20	IG-NS-1808	Isfallsglaciären	Northern meltwater stream	67°54'58"N 18°35'15"E	18-08-2018
21	IG-MS-1808	Isfallsglaciären	Downstream river	67°54'54"N 18°35'59"E	18-08-2018
22	IG-L-1808	Isfallsglaciären	Main lake	67°54'57"N 18°35'48"E	18-08-2018

23	MS-bef.SG-1908	Tarfalajåkk	Tarfalajåkk river before joining Storglaciären meltwater streams	67°54'23"N 18°36'58"E	19-08-2018
24	MS+IG-1908	Tarfalajåkk	Tarfalajåkk river after joining Isfallsglaciären meltwater stream	67°54'45"N 18°36'25"E	19-08-2018
25	MS-L-1908	Tarfalajåkk	Lake at the Tarfalajåkk river	67°54'33"N 18°36'42"E	19-08-2018
<b>Soil</b>					
1	SG-OM-0808	Storglaciären	Outer moraine	67°54'22"N 18°36'47"E	08-08-2018
2	SG-M2-0808	Storglaciären	Inner moraine	67°54'20"N 18°36'46"E	08-08-2018
3	SG-VP-0808	Storglaciären	Vegetated patch	67°54'19"N 18°36'45"E	08-08-2018
4	SG-RB-0808	Storglaciären	River bank	67°54'15"N 18°36'43"E	08-08-2018
5	IG-LM-1208	Isfallsglaciären	Large moraine		12-08-2018
6	TL-RB-1408	Tarfalajaure	West bank of the lake	67°55'20"N 18°35'60"E	14-08-2018
7	IG-Lit.Mor-1808	Isfallsglaciären	Little moraine between the lakes	67°54'55"N 18°35'36"E	18-08-2018
8	IG-OM-1808	Isfallsglaciären	Old moraine (outer moraine, close to Tarfalajåkk)	67°54'55"N 18°36'10"E	18-08-2018
<b>Ice</b>					
1	SG-SI-0708	Storglaciären	Surface ice	67°54'07"N 18°34'59"E	07-08-2018
2	SG-BI-0808	Storglaciären	Basal ice	67°54'07"N 18°36'14"E	08-08-2018
3	CG-BI-1308	Kebnepakteglaciären	Basal ice	67°55'34"N 18°34'30"E	13-08-2018

Appendix 7. Sampling sites at the Vatnajökull ice cap, Iceland, 2018.

Water and sediment					
No	Name	Sampling Site	Location at the site	Coordinates	Date
1	VK-US-2605	Virkisjökull	Meltwater stream draining the glacier	63°58'03"N 16°48'06"W	26-05-2018
2	VK-PL-2605	Virkisjökull	Proglacial lake	63°57'57"N 16°48'22"W	26-05-2018
3	VK-DS-2705	Virkisjökull	Downstream river	63°57'10"N 16°50'54"W	27-05-2018
4	VK-US-2805	Virkisjökull	Meltwater stream draining the glacier	63°58'03"N 16°48'06"W	28-05-2018
5	VK-PL-2805	Virkisjökull	Proglacial lake	63°57'57"N 16°48'22"W	28-05-2018
6	VK-DS-2805	Virkisjökull	Downstream river	63°57'10"N 16°50'54"W	28-05-2018
7	SV-R-2905	Svínafellsjökull	Downstream river	63°59'01"N 16°52'22"W	29-05-2018
8	SV-PL-2905	Svínafellsjökull	Proglacial lake	63°59'26"N 16°51'38"W	29-05-2018
9	SV-R-3005	Svínafellsjökull	Downstream river	63°59'01"N 16°52'22"W	30-05-2018
10	SV-PL-3005	Svínafellsjökull	Proglacial lake	63°59'26"N 16°51'38"W	30-05-2018
11	SV-R-3005E	Svínafellsjökull	Downstream river	63°59'01"N 16°52'22"W	30-05-2018
12	VK-US-0206	Virkisjökull	Meltwater stream draining the glacier	63°58'03"N 16°48'06"W	02-06-2018
13	VK-PL-0206	Virkisjökull	Proglacial lake	63°57'57"N 16°48'22"W	02-06-2018
14	VK-DS-0206	Virkisjökull	Downstream river	63°57'10"N 16°50'54"W	02-06-2018
15	SV-R-0306	Svínafellsjökull	Downstream river	63°59'01"N 16°52'22"W	03-06-2018
16	SV-PL-0306	Svínafellsjökull	Proglacial lake	63°59'26"N 16°51'38"W	03-06-2018
Only water					
1	SK-R-106	Skaftafellsjökull	Downstream river	64°00'29"N 16°55'52"W	01-06-2018
2	KV-R-106	Kvíárjökull	Downstream river	63°56'23"N 16°26'21"W	01-06-2018
3	KV-PL-106	Kvíárjökull	Proglacial lake	63°56'21"N 16°27'28"W	01-06-2018
4	FL-R-106	Fjallsjökull	Downstream river	64°00'44"N 16°22'28"W	01-06-2018
5	HL-R-0206	Háalda	River	63°55'13"N 16°46'53"W	02-06-2018
Soil					
1	SV-LM-2905	Svínafellsjökull	Lake moraine	63°59'27"N 16°51'37"W	29-05-2018
2	SV-LIAM-3005	Svínafellsjökull	Little Ice Age moraine	63°59'12"N 16°52'11"W	30-05-2018

3	SV-2.5K-3105	Svínafellsjökull	2500 years old moraine	63°59'29"N 16°53'29"W	31-05-2018
4	VK-YM-0206	Virkisjökull	Young moraine	63°57'49"N 16°48'44"W	02-06-2018
5	VK-OM-0206	Virkisjökull	Old moraine	63°58'15"N 16°48'08"W	02-06-2018
<b>Ice</b>					
1	SV-BI-2705	Svínafellsjökull	Basal ice	63°59'40"N 16°52'37"W	27-05-2018
2	SV-SI-3105	Svínafellsjökull	Surface ice	63°59'55"N 16°52'00"W	31-05-2018
3	VK-BI-0206	Virkisjökull	Basal ice	63°58'05"N 16°47'46"W	02-06-2018
4	VK-SI-0206	Virkisjökull	Surface ice	63°58'13"N 16°48'10"W	02-06-2018

Appendix 8. Sampling sites at the Vatnajökull ice cap, Iceland, 2019.

Water and sediment					
No	Name	Sampling Site	Location at the site	Coordinates	Date
1	VK-US-1909	Virkisjökull	Meltwater stream draining the glacier	63°58'05"N 16°48'03"W	19-09-2019
2	VK-PL-1909	Virkisjökull	Proglacial lake	63°57'54"N 16°48'33"W	19-09-2019
3	VK-DS-2009	Virkisjökull	Downstream river	63°57'11"N 16°50'59"W	20-09-2019
4	SV-PL-2109	Svínafellsjökull	Proglacial lake	63°59'26"N 16°51'38"W	21-09-2019
5	SV-R-2109*	Svínafellsjökull	Downstream river	63°59'01"N 16°52'22"W	21-09-2019
6	SK-PL-2209	Skaftafellsjökull	Proglacial lake	64°01'14"N 16°53'55"W	22-09-2019
7	SK-R-2209	Skaftafellsjökull	Downstream river	64°00'31"N 16°55'44"W	22-09-2019
8	FJ-PL-2309	Fjallsjökull	Proglacial lake	64°01'08"N 16°22'16"W	23-09-2019
9	FJ-R-2309	Fjallsjökull	Downstream river	64°00'24"N 16°22'08"W	23-09-2019
10	KV-PL-2409	Kvíárjökull	Proglacial lake	63°56'20"N 16°27'32"W	24-09-2019
11	KV-R-2409*	Kvíárjökull	Downstream river	63°56'21"N 16°26'12"W	24-09-2019
*Water and soil samples					
Soil					
1	VK-YM-1909	Virkisjökull	Young moraine	63°57'55"N 16°48'22"W	19-09-2019
2	VK-OM-1909	Virkisjökull	Old moraine	63°57'55"N 16°48'16"W	19-09-2019
3	VK-M2-1909	Virkisjökull	Moraine #2	63°57'48"N 16°48'33"W	19-09-2019
4	SV-YM-2009	Svínafellsjökull	Youngest moraine	63°59'49"N 16°52'46"W	20-09-2019
5	SV-LM-2109	Svínafellsjökull	Lake moraine	63°59'02"N 16°52'22"W	21-09-2019
6	SV-LIAM-2109	Svínafellsjökull	Little Ice Age moraine	63°59'28"N 16°52'33"W	21-09-2019
7	SV-2.5K-2109	Svínafellsjökull	2500 years old moraine	63°59'29"N 16°53'29"W	21-09-2019
8	SK-TM-2209	Skaftafellsjökull	Terminal moraine	64°01'02"N 16°54'02"W	22-09-2019
9	SK-M2-2209	Skaftafellsjökull	Moraine #2	64°00'44"N 16°54'22"W	22-09-2019
10	FJ-LM-2309	Fjallsjökull	Lake moraine	64°01'08"N 16°22'16"W	23-09-2019
11	FJ-M2-2309	Fjallsjökull	Moraine #2		23-09-2019
12	FJ-M3-2309	Fjallsjökull	Moraine #3	64°00'16"N 16°22'24"W	23-09-2019

13	KV-LM-2409	Kvíárjökull	Lake moraine	63°56'20"N 16°27'32"W	24-09-2019
14	KV-M2-2409	Kvíárjökull	Moraine #2	63°56'15"N 16°27'20"W	24-09-2019
15	KV-M3-2409	Kviarjökull	Moraine #3	63°56'18"N 16°26'27"W	24-09-2019
16	SD-LM-2609	Skeiðarárjökull	Lake moraine	63°59'39"N 17°11'07"W	26-09-2019
17	SD-M2-2609	Skeiðarárjökull	Moraine #2	63°58'32"N 17°11'55"W	26-09-2019
18	VK-RM-2709	Virkisjökull	Road moraine	63°57'01"N 16°51'17"W	27-09-2019

Appendix 9. Sampling sites at Zackenberg valley, Greenland.

Water and sediment					
No	Name	Sampling Site	Location at the site	Coordinates	Date
1	ZR-BL-0108	Zackenberg River	Before Lindemanselven River inflows to Zackenberg River	74°30'39"N 20°38'57"W	01-08-2019
2	ZR-B-0108	Zackenberg River	Bridge by the station	74°28'33"N 20°33'59"W	01-08-2019
3	ZR-S-0208	Zackenberg River	Station	74°28'15"N 20°34'43"W	02-08-2019
4	SS-D-0508	Store Sø Lake	Delta at the inlet where glacier meltwater stream inflows the lake	74°31'18"N 21°04'38"W	05-08-2019
5	AHR-0508*	River at Antonsens Hytte	River at Antonsens Hytte	74°30'56"N 21°10'50"W	05-08-2019
6	ZR-B-0808	Zackenberg River	Bridge by the station	74°28'32"N 20°34'01"W	08-08-2019
7	SS-L-1008	Store Sø Lake	Middle of the lake transect	74°31'12"N 20°55'16"W	10-08-2019
8	SS-O-1008*	Store Sø Lake	Lake outlet	74°31'08"N 20°50'17"W	10-08-2019
9	ZR-LM-1608	Zackenberg River	After Lindemanselven and Palnatokeelv rivers inflow to Zackenberg River	74°30'12"N 20°34'54"W	16-08-2019
10	ZR-AC-1608	Zackenberg River	Aucellaelv River inflows to Zackenberg River	74°29'41"N 20°35'24"W	16-08-2019
11	ZR-B-1908	Zackenberg River	Bridge by the station	74°28'33"N 20°34'01"W	19-08-2019
Modified water sampling and sediment					
1	SS-L-1308	Store Sø Lake	Eastern lobe of the lake in the mid-section transect	74°31'20"N 20°52'33"W	13-08-2019
2	G-1408	A.P. Olsen Land glacier meltwater stream	Meltwater stream draining the glacier	74°32'03"N 21°07'11"W	14-08-2019
3	SS-LI-1408*	Store Sø Lake	Inlet area of the lake	74°31'28"N 21°00'07"W	14-08-2019
4	ZR-SS-1508	Zackenberg River	Zackenberg River after outflowing from the lake	74°30'57"N 20°45'17"W	15-08-2019
*Water and soil samples					
Soil					
1	ZR-BL-0108	Zackenberg River	Before Lindemanselven River inflows to Zackenberg River	74°30'41"N 20°38'48"W	01-08-2019
2	ZR-S-0208	Zackenberg River	Station	74°28'15"N 20°34'43"W	02-08-2019
3	SS-D-0508	Store Sø Lake	Delta at the inlet where glacier meltwater stream inflows the lake	74°31'17"N 21°04'48"W	05-08-2019
4	ZR-B-0808	Zackenberg River	Bridge by the station	74°28'32"N 20°34'01"W	08-08-2019

5	SS-L-1008	Store Sø Lake	Middle of the lake transect	74°31'07"N 20°55'17"W	10-08-2019
6	SS-L-1308	Store Sø Lake	Eastern lobe of the lake in the mid-section transect	74°31'20"N 20°52'33"W	13-08-2019
7	G-1408	A.P. Olsen Land glacier meltwater stream	Meltwater stream draining the glacier	74°32'03"N 21°07'11"W	14-08-2019
8	ZR-SS-1508	Zackenbergl River	Zackenbergl River after outflowing from the lake	74°30'57"N 20°45'17"W	15-08-2019
9	ZR-LM-1608	Zackenbergl River	After Lindemanselven and Palnatokeelv rivers inflow to Zackenbergl River	74°30'12"N 20°34'54"W	16-08-2019
10	ZR-AC-1608	Zackenbergl River	Aucellaelv River inflows to Zackenbergl River	74°29'41"N 20°35'24"W	16-08-2019

Appendix 10. Mean concentration of major ions (mg/L) in water samples from Sweden, Iceland and Greenland.

Samples	Na <sup>+</sup>	NH <sub>4</sub> <sup>+</sup>	K <sup>+</sup>	Mg <sup>2+</sup>	Ca <sup>2+</sup>	Cl <sup>-</sup>	NO <sub>2</sub> <sup>-</sup>	SO <sub>4</sub> <sup>2-</sup>	NO <sub>3</sub> <sup>-</sup>	PO <sub>4</sub> <sup>3-</sup>
KG-MW-1308	0.178±0.1	0±0	0.036±0.049	0.037±0.008	0.151±0.045	0.133±0.045	0±0	0.508±0.039	0±0	0.125±0.143
CB-MW-1408	0.286±0.034	0±0	0.108±0.09	0.188±0.021	0.427±0.09	0.179±0.075	0±0	2.33±0.088	0±0	n.d.
T-L-1308	0.277±0.017	0±0	0.059±0.015	0.173±0.005	0.454±0.021	0.184±0.011	0±0	2.141±0.01	0±0	n.d.
MS-TL-1408	0.312±0.028	0±0	0.065±0.021	0.214±0.022	0.588±0.067	0.201±0.038	0±0	2.748±0.034	0±0	n.d.
MS+IG-1908	0.315±0.011	0±0	0.076±0.009	0.18±0.016	0.474±0.028	0.173±0.01	0±0	2.158±0.031	0±0	n.d.
MS-L-1908	0.378±0.025	0±0	0.162±0.176	0.198±0.009	0.477±0.059	0.167±0.017	0±0	2.313±0.086	0±0	n.d.
MS-bef.SG-1908	0.287±0.038	0±0	0.083±0.03	0.171±0.004	0.447±0.071	0.152±0.009	0±0	2.094±0.028	0±0	n.d.
MS-1008	0.257±0.015	0±0	0.058±0.015	0.15±0.008	0.41±0.07	0.138±0.006	0.002±0	1.871±0.075	0±0	n.d.
SG-R-0808	0.076±0.006	0±0	0.058±0.009	0.032±0.006	0.148±0.037	0.025±0.019	0±0	0.294±0.032	0±0	0.006±0.006
SG-R-1608	0.209±0.045	0±0	0.081±0.035	0.089±0.002	0.323±0.012	0.136±0.02	0±0	1.413±0.052	0±0	n.d.
SG-SS-1008	0.137±0.021	0±0	0.081±0.02	0.066±0.015	0.27±0.104	0.037±0.021	0±0	1.054±0.027	0±0	0.009±0.001
SG-DS-0908	0.13±0.023	0±0	0.068±0.008	0.071±0.002	0.263±0.009	0.048±0.004	0±0	1.09±0.03	0±0	n.d.
SG-DS-1608	0.255±0.012	0.057±0	0.173±0.104	0.119±0.005	0.422±0.015	0.154±0.018	0±0	1.674±0.034	0±0	n.d.
SG-MX-0908	0.77±1.042	0±0	0.117±0.047	0.091±0.009	0.296±0.023	0.113±0.053	0±0	1.135±0.058	0±0	n.d.
SG-MX-1608	0.375±0.069	0.047±0	0.158±0.078	0.219±0.037	0.652±0.096	0.168±0.026	0.001±0	2.822±0.323	0.03±0	n.d.
IG-NS-1208	0.079±0.011	0±0	0.037±0.016	0.065±0.011	0.216±0.064	0.064±0.009	0±0	0.808±0.024	0±0	n.d.
IG-NS-1808	0.081±0.01	0±0	0.035±0.016	0.042±0.002	0.146±0.025	0.068±0.005	0±0	0.634±0.021	0±0	n.d.
IG-SS-1208	0.171±0.118	0±0	0.027±0.018	0.039±0.011	0.147±0.076	0.125±0.056	0±0	0.516±0.037	0±0	n.d.
IG-SS-1508	0.166±0.015	0±0	0.071±0.014	0.095±0.004	0.377±0.026	0.024±0.001	0±0	1.701±0.053	0.012±0	0.008±0.008
IG-SS-1808	0.157±0.133	0±0	0.007±0.011	0.034±0.006	0.104±0.017	0.091±0.012	0±0	0.5±0.023	0±0	n.d.
IG-EL-1208	0.19±0.023	0±0	0.061±0.012	0.089±0.003	0.302±0.048	0.139±0.065	0±0	1.562±0.102	0±0	n.d.
IG-DL-1108	0.141±0.029	0±0	0.212±0.286	0.069±0.019	0.271±0.113	0.113±0.038	0±0	1.018±0.034	0±0	n.d.
IG-L-1808	0.104±0.019	0±0	0.087±0.114	0.067±0.005	0.23±0.006	0.118±0.081	0.011±0	0.931±0.004	0.084±0	n.d.
IG-DR-1108	0.102±0.022	0±0	0.025±0.014	0.074±0.014	0.272±0.056	0.093±0.022	0±0	1.007±0.03	0±0	n.d.
IG-MS-1808	0.089±0.015	0±0	0.032±0.017	0.055±0.006	0.146±0.01	0.087±0.017	0±0	0.91±0.039	0±0	n.d.

VK-US-2605	3.252±0.142	0.006±0	0.394±0.021	0.303±0.033	0.833±0.159	1.867±0.163	0±0	0.639±0.063	0.024±0.041	0.072±0.031
VK-US-2805	4.402±0.269	0±0	0.472±0.045	0.483±0.035	1.094±0.177	3.899±0.191	0±0	1.062±0.035	0±0	0.044±0.006
VK-US-0206	3.268±0.113	0±0	0.427±0.047	0.446±0.056	0.921±0.078	1.757±0.084	0±0	0.786±0.029	0±0	0.072±0.021
VK-US-1909	8.065±0.305	0±0	0.424±0.094	2.377±0.12	5.999±0.092	2.851±0.15		1.998±0.145	0.021±0.022	0.047±0.007
VK-PL-2605	6.67±0.161	0±0	0.668±0.046	0.977±0.067	2.809±0.223	3.988±0.246	0±0	3.484±0.102	0.133±0.134	0.158±0.015
VK-PL-2805	5.778±0.138	0±0	0.616±0.023	0.631±0.129	1.641±0.378	3.319±0.265	0±0	1.693±0.116	0±0	0.094±0.019
VK-PL-0206	4.933±0.074	0±0	0.534±0.05	0.433±0.027	1.051±0.118	2.965±0.102	0±0	1.283±0.043	0.002±0.003	0.084±0.026
VK-PL-1909	5.799±0.092	0±0	0.419±0.051	1.871±0.061	4.603±0.309	2.337±0.083		1.424±0.055		0.063±0.005
VK-DS-2705	5.768±0.191	0±0	0.578±0.077	0.554±0.038	1.436±0.069	3.146±0.101	0±0	1.492±0.06	0±0	0.086±0.007
VK-DS-2805	5.364±0.044	0±0	0.528±0.056	0.495±0.005	1.199±0.092	2.915±0.131	0±0	1.333±0.027	0.02±0.034	0.083±0.003
VK-DS-0206	5.161±0.065	0±0	0.516±0.038	0.475±0.033	1.029±0.077	3.124±0.08	0±0	1.308±0.017	0.019±0.033	0.095±0.023
VK-DS-2009	5.711±0.123	0±0	0.777±0.661	1.83±0.052	3.835±0.338	2.242±0.025		1.313±0.025	0±0	0.051±0.012
SV-PL-2905	5.171±0.343	0±0	0.352±0.025	1.029±0.272	9.096±4.127	3.753±0.571	0.004±0.005	6.703±3.17	0±0	n.d.
SV-PL-3005	5.065±0.117	0±0	0.316±0.011	0.924±0.048	8.793±0.445	3.304±0.282	0.002±0.002	8.624±0.266	0±0	0.007±0.006
SV-PL-2109	4.913±0.116	0±0	0.275±0.013	3.098±0.046	n.d.	4.007±0.074		4.829±0.17	n.d.	0.011±0.007
SV-R-2905	4.384±0.127	0±0	0.305±0.017	0.726±0.048	6.149±0.509	2.713±0.052	0±0	3.219±0.069	0±0	0.015±0.001
SV-R-3005	4.242±0.076	0±0	0.256±0.021	0.59±0.01	5.031±0.076	2.838±0.121	0.002±0.003	3.046±0.091	0±0	0.007±0.007
SV-R-3005E	4.34±0.104	0±0	0.279±0.018	0.603±0.04	5.667±0.057	2.704±0.063	0±0	3.07±0.115	0±0	0.023±0.021
SV-R-0306	4.279±0.055	0±0	0.283±0.046	0.61±0.022	5.503±0.331	2.692±0.089	0±0	2.999±0.106	0±0	0.011±0.001
SV-R-2109	4.157±0.26	0±0	0.21±0.008	1.922±0.099	11.052±0.66	1.875±0.206	n.d.	2.829±0.1	n.d.	0.016±0.012
SK-PL-2209	3.961±0.948	0±0	0.073±0.012	0.851±0.069	8.681±0.532	1.345±0.367	n.d.	1.338±0.498	n.d.	0.005±0.002
SK-R-2209	3.867±0.509	0±0	0.088±0.055	0.856±0.034	8.865±0.388	1.17±0.098	n.d.	1.131±0.195	n.d.	n.d.
FJ-PL-2309	3.054±0.361	0±0	0.198±0.022	3.21±0.271	n.d.	2.835±0.269	n.d.	7.796±0.744	n.d.	n.d.
FJ-R-2309	1.991±0.186	0±0	0.077±0.014	1.332±0.031	11.76±0.73	1.407±0.066	n.d.	2.857±0.095	n.d.	n.d.
KV-PL-2409	5.18±0.313	0±0	0.142±0.013	2.057±0.09	9.744±0.362	2.101±0.092	n.d.	1.428±0.127	n.d.	0.004±0.004
KV-R-2409	4.461±0.087	0±0	0.115±0.005	1.552±0.113	6.862±0.312	1.648±0.033	n.d.	1.048±0.042	n.d.	n.d.
G-1408	0.426±0.037	0.151±0.187	0.557±0.022	0.82±0.11	3.506±0.035	0.212±0.008	n.d.	1.275±0.094	0.091±0.006	n.d.
AHR-0508	0.37±0.014	0.041±0.05	0.217±0.092	0.543±0.121	1.787±0.292	0.537±0.15	n.d.	0.664±0.04	0.024±0.003	n.d.

SS-D-0508	0.235±0.059	0.035±0.019	0.229±0.02	0.337±0.064	1.178±0.291	0.255±0.104	n.d.	0.436±0.039	0.007±0.006	n.d.
SS-LI-1408	0.39±0.027	0.046±0.025	0.558±0.067	0.844±0.053	2.923±0.359	0.208±0.017	n.d.	0.83±0.009	0.02±0.003	n.d.
SS-L-1008	0.415±0.059	0.047±0.058	1.067±0.989	0.722±0.103	3.108±0.535	0.305±0.074	n.d.	0.991±0.013	0.028±0.01	n.d.
SS-LW-1308	0.419±0.018	0.056±0.033	0.612±0.05	0.867±0.086	3.084±0.503	0.245±0.021	n.d.	1.21±0.005	0.034±0.003	n.d.
SS-O-1008	0.393±0.03	0.02±0.014	0.517±0.109	0.55±0.013	2.24±0.21	0.206±0.013	n.d.	0.837±0.005	0.077±0.008	n.d.
ZR-SS-1508	0.592±0.021	0.041±0.024	0.446±0.035	1.632±0.126	5.538±0.66	0.276±0.021	0.004±0.001	4.349±0.086	0.024±0.018	n.d.
ZR-BL-0108	0.549±0.212	1.089±1.85	0.642±0.175	0.861±0.271	3.714±1.621	0.386±0.21	n.d.	1.105±0.386	0.042±0.033	n.d.
ZR-LM-1608	0.937±0.019	0.014±0.024	0.71±0.39	5.848±0.063	n.d.	0.258±0.032	n.d.	30.467±0.1	0.008±0.005	n.d.
ZR-AC-1608	0.739±0.007	0.026±0.005	0.59±0.149	3.159±0.115	8.39±0.409	0.226±0.017	n.d.	12.397±0.164	0.024±0.006	n.d.
ZR-B-0108	0.519±0.063	0.388±0.642	0.559±0.115	1.719±0.175	5.4±0.484	0.241±0.043	n.d.	4.627±0.548	0.054±0.031	n.d.
ZR-B-0808	0.539±0.033	0.021±0.026	0.549±0.055	2.255±0.273	6.32±0.346	0.23±0.044	0.003±0.002	7.035±0.065	0.023±0.006	n.d.
ZR-B-1908	0.699±0.173	0.285±0.459	0.949±0.788	2.842±0.348	7.705±0.482	0.267±0.016	0.014±0.015	9.769±0.403	0.112±0.159	n.d.
ZR-S-0208	0.67±0.024	0±0	0.549±0.078	1.704±0.194	4.654±0.887	0.5±0.054	n.d.	4.615±0.094	n.d.	n.d.

Appendix 11. Mean concentration of dissolved metals (mg/L) in water samples from Sweden, Iceland and Greenland.

Samples	P	Na	K	Ca	Mn	Fe	Zn	Al	S	Mg
KG-MW-1308	0.001±0	n.d.	0.001±0.002	0.386±0.003	0.001±0	0.004±0.002	n.d.	n.d.	0.195±0.008	0.069±0.002
CB-MW-1408	0.001±0.001	0.135±0.036	0.065±0.007	1.097±0.033	0.002±0	0.002±0.001	n.d.	n.d.	0.873±0.035	0.33±0.005
T-L-1308	n.d.	0.112±0.014	0.057±0.002	1.25±0.025	0.001±0	0.008±0.001	n.d.	n.d.	0.755±0.008	0.313±0.002
MS-TL-1408	0±0	0.156±0.014	0.062±0.003	1.514±0.003	0±0	0.007±0.001	n.d.	n.d.	0.962±0.008	0.369±0.002
MS+IG-1908	0.001±0.001	0.106±0.013	0.058±0	1.248±0.008	0±0	0.006±0.001	n.d.	n.d.	0.793±0.005	0.332±0.003
MS-L-1908	0±0	0.172±0	0.057±0	1.326±0	0±0	0.006±0	0±0	0±0	0.82±0	0.379±0
MS-bef.SG-1908	0±0.001	0.106±0.016	0.054±0.001	1.191±0.007	0±0	0.008±0	n.d.	n.d.	0.773±0.002	0.309±0.002
MS-1008	0±0	0.127±0.023	0.053±0.001	1.113±0.009	0±0	0.011±0.001	n.d.	0.002±0.002	0.699±0.001	0.292±0.001
SG-R-0808	0.028±0.007	0.106±0.056	0.092±0.014	0.539±0.062	0.005±0.001	0.327±0.059	n.d.	0.253±0.059	0.14±0.009	0.203±0.024
SG-R-1608	0.011±0.003	0.082±0.012	0.114±0.006	1.074±0.03	0.005±0	0.239±0.047	n.d.	0.179±0.041	0.512±0.002	0.264±0.02
SG-SS-1008	0.015±0.005	0.455±0.573	0.097±0.021	0.855±0.143	0.006±0	0.207±0.042	n.d.	0.16±0.037	0.552±0.261	0.22±0.042
SG-DS-0908	0.017±0.003	0.169±0.027	0.122±0.001	0.922±0.009	0.006±0	0.297±0.012	n.d.	0.23±0.009	0.401±0.008	0.257±0.004
SG-DS-1608	0.012±0.002	0.072±0.005	0.122±0.001	1.238±0.029	0.005±0	0.256±0.013	n.d.	0.19±0.011	0.63±0.019	0.322±0.01
SG-MX-0908	0.011±0.002	0.186±0.025	0.102±0.005	0.901±0.014	0.005±0	0.311±0.031	n.d.	0.238±0.025	0.41±0.004	0.284±0.015
SG-MX-1608	0.006±0.001	0.231±0.051	0.159±0.005	1.673±0.051	0.002±0	0.152±0.013	n.d.	0.109±0.011	0.964±0.035	0.419±0.009
IG-NS-1208	0.004±0.001	n.d.	0.021±0.001	0.483±0.006	0.002±0	0.065±0.001	n.d.	0.047±0.003	0.301±0.004	0.121±0.002
IG-NS-1808	0.004±0.001	n.d.	0.019±0.002	0.446±0.004	0.002±0	0.092±0.009	n.d.	0.068±0.007	0.247±0.005	0.124±0.004
IG-SS-1208	0.002±0.001	0.058±0.041	0.006±0.004	0.301±0.015	0.002±0	0.013±0.001	n.d.	0.004±0	0.228±0.041	0.069±0.003
IG-SS-1508	0.013±0.001	0.08±0.064	0.097±0.003	1.076±0.046	0.006±0	0.139±0.028	n.d.	0.105±0.024	0.684±0.088	0.238±0.009
IG-SS-1808	0.001±0.001	n.d.	n.d.	0.299±0.015	0.002±0	0.022±0.001	n.d.	0.012±0.002	0.189±0.013	0.071±0.001
IG-EL-1208	0.003±0.002	0.11±0.071	0.069±0.009	0.873±0.013	0.001±0	0.035±0.003	n.d.	0.022±0.003	0.601±0.028	0.193±0.002
IG-DL-1108	0.007±0.006	1.955±0.747	0.06±0.042	0.847±0.333	0.002±0	0.028±0	n.d.	0.021±0.003	1.027±0.631	0.18±0.065
IG-L-1808	0.001±0.001	n.d.	0.021±0.001	0.55±0.003	0.002±0	0.061±0.002	n.d.	0.044±0.001	0.362±0.004	0.135±0.002
IG-DR-1108	0.001±0	n.d.	0.019±0.002	0.534±0.009	0.002±0	0.022±0.001	n.d.	0.012±0.001	0.375±0.003	0.117±0.001
IG-MS-1808	0.002±0.001	n.d.	0.022±0.002	0.55±0.018	0.002±0	0.072±0.006	n.d.	0.051±0.005	0.378±0.041	0.14±0.004

VK-US-2605	0.097±0.008	2.672±0.081	0.463±0.031	1.744±0.018	0.026±0.003	1.015±0.102	0.001±0	0.666±0.065	0.217±0.004	0.694±0.027
VK-US-2805	0.218±0.049	4.121±0.08	0.724±0.045	3.055±0.264	0.083±0.02	1.905±0.448	0.008±0.005	1.535±0.336	0.379±0.004	1.168±0.127
VK-US-0206	0.125±0.013	3.158±0.045	0.513±0.008	2.32±0.097	0.031±0.004	0.987±0.093	0.009±0.013	0.722±0.066	0.299±0.011	0.861±0.036
VK-US-1909	0.208±0.003	9.261±0.147	0.967±0.029	5.022±0.18	0.06±0.002	2.829±0.138	0.021±0.009	1.893±0.131	0.803±0.004	1.612±0.028
VK-PL-2605	0.136±0.02	5.759±0.075	0.811±0.032	5.022±0.308	0.024±0.007	0.876±0.237	0.006±0.008	0.483±0.126	1.178±0.053	1.517±0.128
VK-PL-2805	0.131±0.007	4.785±0.049	0.715±0.007	3.509±0.279	0.031±0.003	1.273±0.19	n.d.	0.729±0.102	0.613±0.036	1.167±0.042
VK-PL-0206	0.217±0.136	4.474±0.197	0.73±0.082	3.19±0.816	0.068±0.048	2.027±1.276	0.008±0.001	1.243±0.722	0.472±0.004	1.226±0.433
VK-PL-1909	0.195±0.043	6.86±0.09	0.929±0.038	4.006±0.283	0.052±0.013	2.691±0.536	0.01±0.004	1.739±0.353	0.585±0.005	1.282±0.152
VK-DS-2705	0.115±0.004	4.838±0.163	0.692±0.012	2.884±0.077	0.027±0.002	1.04±0.084	0.001±0.001	0.581±0.05	0.528±0.013	0.984±0.043
VK-DS-2805	0.114±0.008	4.473±0.027	0.665±0.01	2.678±0.085	0.027±0.003	1.031±0.097	n.d.	0.601±0.066	0.486±0.002	0.922±0.035
VK-DS-0206	0.158±0.03	4.542±0.071	0.687±0.022	2.782±0.207	0.045±0.009	1.51±0.322	0.008±0.003	0.934±0.205	0.471±0.006	1.049±0.109
VK-DS-2009	0.176±0.033	6.593±0.126	0.94±0.056	3.853±0.31	0.05±0.008	2.813±0.3	0.015±0.008	1.827±0.23	0.567±0.006	1.255±0.091
SV-PL-2905	0.043±0.011	4.617±0.192	0.454±0.018	10.638±2.809	0.013±0.002	0.475±0.163	n.d.	0.269±0.091	2.223±0.942	1.413±0.215
SV-PL-3005	0.04±0.011	4.523±0.084	0.422±0.007	11.004±0.217	0.015±0.004	0.492±0.199	n.d.	0.276±0.095	2.945±0.135	1.382±0.035
SV-PL-0306	0.05±0.028	4.218±0.108	0.401±0.011	9.418±0.435	0.019±0.009	0.688±0.434	n.d.	0.366±0.209	1.593±0.207	1.252±0.064
SV-PL-2109	0.055±0.01	5.638±0.07	0.595±0.004	13.998±0.28	0.019±0.004	1.007±0.237	n.d.	0.634±0.129	1.727±0.07	1.728±0.046
SV-R-2905	0.047±0.026	3.836±0.079	0.353±0.019	8.003±0.176	0.016±0.009	0.582±0.397	n.d.	0.319±0.197	1.113±0.01	1.046±0.128
SV-R-3005	0.039±0.009	3.731±0.077	0.33±0.006	7.751±0.106	0.012±0.004	0.443±0.203	n.d.	0.251±0.104	1.037±0.01	0.953±0.073
SV-R-3005E	0.06±0.004	3.749±0.046	0.344±0.007	8.033±0.085	0.023±0.001	0.857±0.041	n.d.	0.467±0.021	1.043±0.007	1.085±0.016
SV-R-0306	0.176±0.203	3.922±0.262	0.385±0.075	8.703±1.449	0.061±0.073	2.599±3.11	n.d.	1.279±1.497	1.017±0.016	1.621±0.98
SV-R-2109	0.151±0.029	4.887±0.077	0.588±0.007	9.332±0.262	0.057±0.011	3.219±0.5	0.002±0.002	1.985±0.272	1.113±0.01	1.755±0.156
KV-PL-106	0.053±0.012	7.459±0.169	0.471±0.027	5.784±0.146	0.044±0.003	1.173±0.171	0.016±0.014	0.533±0.082	0.983±0.039	1.274±0.061
KV-PL-2409	0.07±0.005	5.696±0.45	0.483±0.031	6.48±0.104	0.023±0.001	1.743±0.101	0.004±0.001	1.099±0.077	0.537±0.034	1.263±0.028
KV-R-106	0.074±0.031	7.368±0.094	0.469±0.029	5.816±0.239	0.05±0.009	1.712±0.691	n.d.	0.763±0.307	0.963±0.013	1.451±0.267
KV-R-2409	0.182±0.102	5.423±0.152	0.514±0.069	5.942±0.969	0.053±0.027	3.662±1.694	0.004±0.001	2.259±1.037	0.447±0.007	1.813±0.672
FJ-PL-2309	0.021±0.008	3.283±0.352	0.391±0.037	15.245±1.091	0.007±0.002	0.384±0.109	0.008±0.006	0.29±0.083	2.793±0.285	1.532±0.162
FJ-R-0106	0.02±0.003	3.512±0.153	0.247±0.016	9.069±0.106	0.009±0.002	0.345±0.102	n.d.	0.225±0.059	1.941±0.05	0.933±0.022
FJ-R-2309	0.029±0.026	2.289±0.384	0.212±0.027	8.299±0.264	0.014±0.009	0.668±0.468	0.007±0.003	0.503±0.352	1.163±0.121	0.662±0.135

SK-PL-2209	0.065±0.006	4.095±0.266	0.24±0.016	5.752±0.253	0.021±0.002	1.422±0.134	0.003±0.001	0.896±0.085	0.458±0.072	0.73±0.056
SK-R-0106	0.062±0.019	3.934±0.045	0.237±0.014	5.435±0.306	0.023±0.008	1.351±0.478	n.d.	0.688±0.248	0.558±0.006	0.812±0.17
SK-R-2209	0.087±0.011	4.399±0.467	0.257±0.006	5.957±0.112	0.029±0.007	1.963±0.463	0.007±0.009	1.257±0.29	0.504±0.187	0.922±0.165
HL-R-0206	0.041±0.001	10.482±0.204	1.266±0.007	4.379±0.054	0.009±0	0.317±0.026	n.d.	0.284±0.013	0.677±0.037	2.796±0.061
G-1408	0.029±0.009	0.588±0.072	0.72±0.088	3.162±0.041	0.029±0.004	1.512±0.294	0.024±0.003	1.379±0.346	0.567±0.011	0.922±0.127
AHR-0508	0.005±0.001	0.313±0.025	0.366±0.006	1.715±0.051	0.005±0	0.328±0.024	0.002±0.001	0.336±0.02	0.325±0.013	0.349±0.008
SS-D-0508	0.009±0.003	0.482±0.524	0.461±0.078	1.574±0.346	0.007±0.001	0.4±0.044	0.001±0.001	0.384±0.022	0.239±0.045	0.312±0.001
SS-LI-1408	0.038±0.04	0.419±0.026	0.647±0.239	2.604±0.227	0.021±0.011	1.36±0.785	0.022±0.002	1.235±0.676	0.403±0.004	0.817±0.297
SS-L-1008	0.015±0.005	0.798±0.637	0.86±0.071	2.692±0.494	0.013±0.003	0.577±0.225	0.006±0.002	0.549±0.194	0.49±0.088	0.508±0.082
SS-LW-1308	0.025±0.021	0.513±0.202	0.532±0.134	2.539±0.147	0.019±0.006	0.941±0.38	0.022±0.001	0.858±0.345	0.535±0.004	0.672±0.143
SS-O-1008	0.038±0.046	1.286±1.093	0.886±0.188	2.821±0.743	0.014±0.005	0.673±0.394	0.005±0.004	0.675±0.445	0.671±0.362	0.545±0.201
ZR-SS-1508	0.003±0.002	0.55±0.163	0.385±0.027	4.001±0.07	0.004±0.001	0.203±0.055	0.03±0.001	0.199±0.073	1.797±0.046	0.741±0.029
ZR-BL-0108	0.022±0.019	1.91±2.697	1.065±0.423	3.229±1.573	0.011±0.003	0.609±0.248	0.003±0.003	0.555±0.242	0.621±0.386	0.549±0.176
ZR-LM-1608	0.006±0.003	1.064±0.011	0.863±0.024	11.427±0.111	0.012±0.001	0.322±0.069	0.003±0	0.286±0.059	11.225±0.065	3.646±0.06
ZR-AC-1608	0.013±0.01	0.814±0.092	0.865±0.083	6.364±0.133	0.011±0.003	0.509±0.25	0.005±0.003	0.446±0.217	4.803±0.021	1.738±0.061
ZR-B-0108	0.006±0.001	0.522±0.011	0.802±0.007	3.755±0.034	0.01±0	0.394±0.019	0.001±0	0.362±0.03	1.777±0.01	0.83±0.011
ZR-B-0808	0.01±0.006	1.135±0.911	0.919±0.162	5.004±0.71	0.009±0.002	0.465±0.164	0.001±0.001	0.412±0.161	2.796±0.09	1.214±0.164
ZR-B-1908	0.009±0.002	0.697±0.055	0.841±0.024	5.455±0.119	0.009±0.001	0.457±0.048	0.002±0.001	0.415±0.046	3.619±0.012	1.387±0.028
ZR-S-0208	0.008±0.007	0.797±0.108	0.869±0.087	3.826±0.139	0.011±0.003	0.486±0.202	n.d.	0.446±0.184	1.837±0.038	0.909±0.084

Appendix 12. Mean concentration of particulate metals (mg/L) in water samples from Sweden, Iceland and Greenland.

Samples	Ca	Cr	Mn	Co	Ni	Cu	Zn	Al	P	S	Pb	Na	K	Mg	Fe	Cd
KG-MW-1308	2.914 ±2.543	0 ±0	0.009 ±0.001	0.001 ±0	0.001 ±0	0.004 ±0	9.037 ±8.092	7.063 ±6.12	0.031 ±0.002	0.012 ±0.01	0.001 ±0.001	17.358 ±15.193	5.461 ±4.755	0.406 ±0.354	0.99 ±0.103	0 ±0
CB-MW-1408	4.322 ±4.545	0 ±0	0.043 ±0.013	0.003 ±0.001	0.003 ±0.001	0.018 ±0.004	14.745 ±18.613	9.844 ±10.632	0.108 ±0.042	0.014 ±0.013	0.001 ±0	23.59 ±26.346	7.284 ±8.009	0.824 ±0.69	3.331 ±1.205	0 ±0
T-L-1308	4.203 ±3.664	0 ±0	0.006 ±0.004	0.001 ±0	0.001 ±0.001	0.004 ±0.001	17.116 ±15.179	11.43 ±10	0.039 ±0.011	0.012 ±0.009	0.001 ±0	26.158 ±22.763	8.354 ±7.277	0.582 ±0.504	0.842 ±0.191	0 ±0
MS-TL-1408	5.016 ±4.466	0 ±0	0.003 ±0.002	0 ±0	0 ±0	0.003 ±0.001	10.287 ±17.818	13.787 ±12.18	0.033 ±0.008	0.043 ±0.029	0.001 ±0.001	31.667 ±27.841	10.07 ±8.924	0.632 ±0.562	0.522 ±0.098	0 ±0
MS+IG-1908	1.385 ±1.294	0 ±0	0.001 ±0.001	0 ±0	0 ±0	0.005 ±0.001	2.325 ±2.266	2.781 ±2.978	0.027 ±0.006	0.011 ±0.013	0.001 ±0	9.165 ±8.122	2.812 ±2.548	0.15 ±0.158	0.515 ±0.097	0 ±0
MS-L-1908	3.162 ±0	0 ±0	0.012 ±0	0.001 ±0	0.001 ±0	0.005 ±0	7.39 ±0	6.811 ±0	0.045 ±0	0.019 ±0	0.002 ±0	16.639 ±0	5.186 ±0	0.542 ±0	1.205 ±0	0 ±0
MS-befSG-1908	5.42 ±2.074	0 ±0	0.007 ±0.001	0.001 ±0	0.001 ±0	0.006 ±0	26.094 ±17.331	14.411 ±5.429	0.037 ±0.003	0.029 ±0.035	0.001 ±0.001	33.476 ±12.805	10.702 ±3.898	0.801 ±0.275	0.868 ±0.008	0 ±0
MS-1008	3.162 ±5.476	0 ±0	0.004 ±0.006	0.001 ±0	0.001 ±0	0.004 ±0.001	0 ±0	7.516 ±13.019	0.029 ±0.015	0.003 ±0.004	0.001 ±0	19.724 ±34.162	6.403 ±11.09	0.43 ±0.745	0.816 ±0.322	0 ±0
SG-R-0808	77.603 ±14.899	0.279 ±0.042	2.108 ±0.217	0.086 ±0.008	0.088 ±0.01	0.219 ±0.024	3.993 ±6.916	73.416 ±18.4	2.433 ±0.122	0.55 ±0.147	0.002 ±0.001	18.566 ±27.969	6.941 ±9.718	39.828 ±6.834	109.954 ±13.107	0.01 ±0.001
SG-R-1608	27.162 ±4.559	0.115 ±0.016	0.85 ±0.102	0.046 ±0.003	0.054 ±0	0.131 ±0.003	0 ±0	17.414 ±6.821	1.283 ±0.033	0.229 ±0.012	0.001 ±0.001	0 ±0	0 ±0	18.649 ±1.768	50.95 ±4.634	0.004 ±0
SG-SS-1008	13.249 ±6.17	0.077 ±0.022	0.618 ±0.131	0.038 ±0.006	0.046 ±0.008	0.109 ±0.017	0 ±0	3.315 ±5.742	0.982 ±0.124	0.148 ±0.055	0.002 ±0	0 ±0	0 ±0	13.61 ±3.164	40.001 ±7.637	0.003 ±0.001
SG-DS-0908	78.738 ±9.952	0.282 ±0.029	2.271 ±0.238	0.104 ±0.005	0.11 ±0.005	0.292 ±0.019	0 ±0	66.758 ±12.14	3.234 ±0.127	0.567 ±0.008	0.004 ±0.001	0 ±0	0.682 ±1.181	43.419 ±3.793	124.311 ±9.612	0.011 ±0.001
SG-DS-1608	31.228 ±10.299	0.123 ±0.026	0.845 ±0.102	0.044 ±0.003	0.05 ±0.003	0.117 ±0.009	0.039 ±0.068	25.733 ±16.028	1.104 ±0.075	0.197 ±0.026	0.001 ±0.001	8.597 ±14.89	2.917 ±5.052	19.532 ±3.315	50.457 ±5.402	0.004 ±0.001
SG-MX-0908	46.661 ±8.333	0.17 ±0.014	1.385 ±0.095	0.065 ±0.004	0.069 ±0.003	0.179 ±0.006	0 ±0	40.296 ±13.196	2.073 ±0.041	0.322 ±0.05	0.002 ±0.001	0.142 ±0.246	0.728 ±0.774	25.114 ±2.627	74.894 ±7.902	0.005 ±0.001
SG-MX-1608	10.792 ±10.439	0.044 ±0.017	0.388 ±0.046	0.021 ±0	0.023 ±0	0.053 ±0.002	4.774 ±8.268	8.947 ±15.496	0.553 ±0.017	0.293 ±0.301	0.001 ±0.001	10.881 ±18.847	3.526 ±6.108	8.351 ±1.981	23.7 ±1.528	0.002 ±0
IG-NS-1208	4.231 ±4.048	0.018 ±0.006	0.196 ±0.016	0.013 ±0.001	0.016 ±0.001	0.04 ±0.004	0 ±0	4.114 ±4.974	0.393 ±0.036	0.094 ±0.012	0.001 ±0.001	1.86 ±3.222	0.617 ±1.069	3.436 ±0.693	13.173 ±1.004	0.001 ±0

IG-NS-1808	12.555 ±9.122	0.029 ±0.018	0.197 ±0.054	0.012 ±0.002	0.016 ±0.001	0.039 ±0.005	30.301 ±26.242	21.335 ±18.482	0.365 ±0.029	0.089 ±0.059	0.001 ±0	36.121 ±31.282	11.396 ±9.874	4.9 ±1.973	13.007 ±2.621	0.001 ±0.001
IG-SS-1208	0.455 ±0.788	0 ±0	0.052 ±0.006	0.003 ±0	0.004 ±0	0.011 ±0.001	0 ±0	0 ±0	0.082 ±0.006	0.009 ±0.016	0.002 ±0.001	0 ±0	0 ±0	0.584 ±0.378	3.426 ±0.142	0 ±0
IG-SS-1508	29.887 ±14.123	0.108 ±0.032	0.721 ±0.139	0.039 ±0.003	0.045 ±0.003	0.112 ±0.007	14.123 ±20.609	31.564 ±25.303	0.885 ±0.055	0.097 ±0.017	0.003 ±0.001	28.325 ±30.879	10.038 ±10.75	17.614 ±4.095	44.165 ±6.629	0.004 ±0.001
IG-SS-1808	0 ±0	0 ±0	0.067 ±0.012	0.005 ±0	0.006 ±0	0.015 ±0.001	0 ±0	0 ±0	0.116 ±0.014	0.027 ±0.028	0.001 ±0	0 ±0	0 ±0	0.509 ±0.442	4.572 ±0.568	0 ±0
IG-EL-1208	2.523 ±4.058	0 ±0.001	0.028 ±0.004	0.003 ±0	0.006 ±0	0.016 ±0.002	8.456 ±14.646	5.877 ±10.179	0.086 ±0.008	0.018 ±0.008	0.003 ±0.002	12.109 ±20.974	3.924 ±6.796	1.006 ±0.653	2.877 ±0.099	0 ±0
IG-DL-1108	3.123 ±3.599	0.001 ±0.002	0.029 ±0.006	0.003 ±0	0.005 ±0	0.014 ±0.001	5.911 ±10.237	7.324 ±8.626	0.068 ±0.009	0.027 ±0.021	0.001 ±0	12.712 ±17.554	4.051 ±5.565	0.958 ±0.864	2.882 ±0.309	0 ±0
IG-L-1808	5.266 ±4.92	0.008 ±0.01	0.085 ±0.044	0.006 ±0.004	0.011 ±0.002	0.03 ±0.005	5.73 ±6.06	9.002 ±8.429	0.17 ±0.061	0.037 ±0.032	0.001 ±0.001	14.972 ±13.438	4.519 ±4.161	2.642 ±1.265	6.931 ±2.276	0 ±0
IG-DR-1108	1.179 ±2.043	0 ±0	0.032 ±0.003	0.003 ±0	0.006 ±0.001	0.016 ±0.002	1.549 ±2.683	2.319 ±4.016	0.074 ±0.005	0.032 ±0.017	0.001 ±0	4.604 ±7.974	1.375 ±2.381	0.619 ±0.618	3.206 ±0.335	0 ±0
IG-MS-1808	2.55 ±3.056	0.002 ±0.003	0.064 ±0.005	0.006 ±0	0.011 ±0	0.028 ±0.001	1.73 ±2.996	3.918 ±6.384	0.141 ±0.004	0.013 ±0.011	0.001 ±0	5.771 ±9.995	1.906 ±3.301	1.926 ±0.543	5.705 ±0.215	0 ±0
VK-US-2605	12.231 ±6.181	0 ±0	0.801 ±0.019	0.03 ±0.004	0.007 ±0.001	0.039 ±0.002	1.663 ±2.88	14.588 ±13.114	1.07 ±0.065	0.062 ±0.045	0.002 ±0.002	10.449 ±13.557	3.469 ±4.174	6.517 ±0.445	43.948 ±3.85	0.003 ±0.001
VK-US-2805	26.224 ±8.889	0 ±0	1.168 ±0.091	0.038 ±0.002	0.008 ±0.001	0.05 ±0.004	17.572 ±16.304	37.33 ±17.214	1.769 ±0.085	0.169 ±0.045	0.003 ±0.001	42.606 ±37.249	13.958 ±12.224	9.375 ±1.587	60.285 ±3.973	0.005 ±0.001
VK-US-0206	20.248 ±2.015	0 ±0	1.301 ±0.049	0.044 ±0.001	0.009 ±0	0.058 ±0.001	0 ±0	25.18 ±4.764	1.969 ±0.049	0.255 ±0.078	0.003 ±0.001	1.869 ±3.237	0.738 ±1.278	9.876 ±0.399	70.349 ±2.647	0.006 ±0
VK-US-1909	22.461 ±10.357	0 ±0	1.361 ±0.068	0.043 ±0.002	0.007 ±0.001	0.044 ±0.002	7.163 ±6.348	32.424 ±19.696	2.214 ±0.094	0.548 ±0.105	0.002 ±0	30.385 ±26.357	11.463 ±9.931	11.784 ±1.538	72.281 ±2.807	0.006 ±0
VK-PL-2605	14.368 ±0.902	0 ±0	0.289 ±0.035	0.009 ±0.001	0.002 ±0	0.015 ±0.003	18.298 ±15.848	30.617 ±2.573	0.539 ±0.068	0.005 ±0.006	0.002 ±0	56.019 ±3.829	18.627 ±1.935	3.984 ±0.366	15.771 ±1.882	0.001 ±0
VK-PL-2805	7.964 ±4.183	0 ±0	0.483 ±0.036	0.016 ±0.001	0.003 ±0	0.024 ±0.002	0 ±0	10.38 ±8.583	0.886 ±0.056	0.021 ±0.018	0.001 ±0.001	7.28 ±9.665	2.326 ±2.789	4.484 ±0.403	26.132 ±1.953	0.002 ±0
VK-PL-0206	9.206 ±12.435	0 ±0	0.713 ±0.244	0.023 ±0.006	0.005 ±0.002	0.029 ±0.01	7.774 ±13.465	12.054 ±20.879	1.178 ±0.329	0.053 ±0.026	0.002 ±0.001	16.726 ±28.971	5.533 ±9.583	5.239 ±2.865	36.059 ±11.002	0.003 ±0.001
VK-PL-1909	5.874 ±2.584	0 ±0	0.651 ±0.003	0.019 ±0.001	0.005 ±0.001	0.027 ±0.001	0 ±0	3.817 ±5.565	1.288 ±0.098	0.041 ±0.005	0.001 ±0	0 ±0	0 ±0	5.685 ±0.177	34.601 ±1.515	0.003 ±0
VK-DS-2705	5.775 ±4.215	0 ±0	0.482 ±0.02	0.015 ±0	0.004 ±0	0.025 ±0.001	0 ±0	6.582 ±8.392	0.894 ±0.021	0.018 ±0.024	0.002 ±0.001	4.25 ±7.362	1.623 ±2.811	4.494 ±0.648	27.055 ±0.604	0.002 ±0

VK-DS-2805	5.166 ±4.507	0 ±0	0.632 ±0.039	0.021 ±0.002	0.004 ±0.001	0.032 ±0.003	0 ±0	5.146 ±7.618	1.092 ±0.082	0.016 ±0.01	0.001 ±0	0.5 ±0.865	0.403 ±0.698	5.307 ±0.149	35.437 ±3.547	0.003 ±0
VK-DS-0206	11.877 ±5.461	0 ±0	0.582 ±0.078	0.018 ±0.001	0.004 ±0.001	0.028 ±0.004	2.685 ±2.72	17.975 ±14.063	1.141 ±0.112	0.016 ±0.005	0.001 ±0.001	16.752 ±14.554	6.124 ±5.305	5.699 ±1.126	31.397 ±3.301	0.003 ±0
VK-DS-2009	9.035 ±7.046	0 ±0	0.763 ±0.035	0.022 ±0	0.007 ±0.002	0.03 ±0.001	1.677 ±2.905	10.355 ±14.402	1.395 ±0.01	0.062 ±0.022	0.001 ±0.001	8.32 ±14.41	3.166 ±5.484	6.763 ±0.995	39.807 ±0.291	0.003 ±0
SV-PL-2905	16.647 ±3.506	0.006 ±0.002	0.545 ±0.194	0.02 ±0.007	0.011 ±0.004	0.037 ±0.013	34.477 ±20.401	35.295 ±6.924	0.808 ±0.277	0.074 ±0.007	0.001 ±0	46.072 ±14.973	16.175 ±5.439	10.016 ±3.097	31.414 ±10.907	0.003 ±0.001
SV-PL-3005	8.628 ±8.468	0.002 ±0.004	0.793 ±0.029	0.032 ±0.001	0.019 ±0.001	0.055 ±0.003	2.944 ±5.099	11.585 ±19.566	1.204 ±0.039	0.078 ±0.011	0.002 ±0.001	9.869 ±17.093	3.411 ±5.908	13.803 ±0.939	49.219 ±0.66	0.004 ±0
SV-PL-0306	13.421 ±0.913	0.001 ±0.001	0.678 ±0.124	0.026 ±0.004	0.016 ±0.003	0.046 ±0.008	2.11 ±1.863	24.951 ±0.155	1.009 ±0.17	0.086 ±0.08	0.001 ±0	18.076 ±9.274	6.484 ±2.702	12.764 ±1.584	41.59 ±6.599	0.003 ±0.001
SV-PL-2109	7.863 ±1.645	0 ±0	0.636 ±0.04	0.024 ±0.001	0.016 ±0	0.04 ±0.002	0 ±0	9.072 ±4.964	1.003 ±0.026	0.059 ±0.017	0.001 ±0.001	0 ±0	0 ±0	10.743 ±0.302	39.093 ±2.289	0.003 ±0
SV-R-2905	26.965 ±6.085	0.012 ±0.007	1.296 ±0.042	0.047 ±0.003	0.027 ±0.002	0.092 ±0.004	6.017 ±10.422	49.088 ±15.782	1.871 ±0.105	0.231 ±0.212	0.002 ±0	35.365 ±33.886	12.713 ±12.004	21.699 ±0.466	74.57 ±4.581	0.006 ±0
SV-R-3005	14.737 ±7.102	0.002 ±0.003	1.207 ±0.072	0.048 ±0	0.028 ±0	0.088 ±0.006	0 ±0	18.208 ±13.904	1.856 ±0.046	0.123 ±0.005	0.002 ±0.001	0.453 ±0.784	0.346 ±0.599	20.57 ±1.267	74.324 ±1.959	0.006 ±0
SV-R-3005E	14.592 ±4.407	0.001 ±0.002	1.266 ±0.028	0.05 ±0.002	0.029 ±0.001	0.089 ±0.003	0 ±0	18.285 ±9.149	1.905 ±0.074	0.107 ±0.039	0.002 ±0.001	0 ±0	0 ±0	21.66 ±0.613	79.16 ±2.507	0.006 ±0.001
SV-R-0306	21.639 ±7.869	0.008 ±0.007	1.047 ±0.395	0.038 ±0.014	0.021 ±0.008	0.07 ±0.027	8.822 ±8.982	38.911 ±12.988	1.489 ±0.541	0.107 ±0.046	0.002 ±0	29.385 ±13.534	10.969 ±4.708	17.665 ±6.782	60.32 ±22.878	0.005 ±0.002
SV-R-2109	22.826 ±3.631	0.008 ±0.005	1.19 ±0.023	0.042 ±0.002	0.027 ±0	0.076 ±0.002	5.391 ±9.337	34.832 ±7.191	1.758 ±0.076	0.141 ±0.016	0.002 ±0	13.988 ±21.875	5.798 ±7.884	19.499 ±0.23	69.724 ±1.19	0.006 ±0
KV-PL-106	17.176 ±2.436	0 ±0	0.689 ±0.009	0.034 ±0.001	0.016 ±0	0.071 ±0.001	0 ±0	23.126 ±5.989	1.445 ±0.033	0.299 ±0.397	0.002 ±0.001	6.313 ±10.934	2.565 ±4.443	15.656 ±0.656	54.724 ±2.765	0.004 ±0.001
KV-PL-2409	22.63 ±8.828	0.003 ±0.005	0.847 ±0.068	0.041 ±0.002	0.022 ±0.003	0.087 ±0.006	3.718 ±6.44	27.719 ±16.973	1.858 ±0.064	0.122 ±0.022	0.002 ±0	14.607 ±25.301	5.289 ±9.162	17.018 ±1.669	58.738 ±2.816	0.005 ±0
KV-R-106	31.157 ±3.77	0.006 ±0.005	1.066 ±0.093	0.049 ±0.004	0.024 ±0.002	0.107 ±0.01	9.355 ±8.476	47.803 ±10.604	2.137 ±0.236	0.183 ±0.043	0.004 ±0	29.081 ±21.472	11.832 ±7.45	23.903 ±2.106	80.056 ±6.867	0.007 ±0.001
KV-R-2409	22.062 ±6.941	0.002 ±0.004	0.955 ±0.073	0.045 ±0.004	0.023 ±0.001	0.099 ±0.006	1.912 ±3.312	25.643 ±14.892	2.114 ±0.221	0.148 ±0.019	0.002 ±0	10.452 ±18.103	4.011 ±6.947	18.485 ±0.989	66.856 ±5.4	0.006 ±0
FJ-PL-2309	5.84 ±7.389	0.003 ±0.005	0.464 ±0.05	0.017 ±0.002	0.013 ±0.003	0.03 ±0.002	6.941 ±12.022	9.776 ±15.868	0.636 ±0.086	0.072 ±0.011	0 ±0	13.145 ±22.768	4.49 ±7.776	7.2 ±1.535	25.085 ±2.632	0.002 ±0
FJ-R-0106	10.674 ±7.125	0.003 ±0.004	0.541 ±0.026	0.02 ±0.001	0.012 ±0	0.036 ±0.001	2.015 ±3.491	22.879 ±17.246	0.693 ±0.039	0.036 ±0.014	0.001 ±0.001	28.178 ±31.864	9.635 ±10.767	9.8 ±0.924	30.993 ±1.654	0.003 ±0

FJ-R-2309	11.547 ±2.599	0.004 ±0.003	0.666 ±0.043	0.023 ±0.001	0.018 ±0.001	0.045 ±0.004	0.31 ±0.537	19.864 ±5.39	0.849 ±0.04	0.102 ±0.015	0.001 ±0	6.793 ±8.141	2.446 ±2.887	10.79 ±1.019	35.5 ±2.703	0.003 ±0
SK-PL-2209	19.383 ±3.896	0.012 ±0.006	1.017 ±0.06	0.046 ±0.002	0.035 ±0.002	0.088 ±0.005	0 ±0	19.658 ±7.125	1.314 ±0.059	0.039 ±0.015	0.001 ±0	0 ±0	0 ±0	20.841 ±1.503	62.768 ±3.374	0.005 ±0
SK-R-106	15.916 ±8.903	0.006 ±0.009	0.863 ±0.036	0.04 ±0.003	0.027 ±0.002	0.075 ±0.003	4.877 ±8.448	16.362 ±18.989	1.123 ±0.065	0.042 ±0.013	0.001 ±0.001	13.341 ±23.108	4.443 ±7.696	17.295 ±0.505	54.912 ±3.864	0.004 ±0
SK-R-2209	28.082 ±5.539	0.021 ±0.007	1.12 ±0.04	0.051 ±0.002	0.04 ±0.001	0.101 ±0.003	1.551 ±2.686	33.262 ±11.169	1.478 ±0.039	0.096 ±0.071	0 ±0	10.619 ±15.989	3.314 ±4.981	23.738 ±0.566	68.908 ±3.222	0.006 ±0
HL-R-0206	6.027 ±1.526	0 ±0	0.398 ±0.029	0.011 ±0.003	0.006 ±0.002	0.026 ±0.002	0 ±0	8.318 ±3.231	0.534 ±0.037	0.04 ±0.058	0.001 ±0.001	1.38 ±2.391	0.425 ±0.736	2.8 ±0.12	20.475 ±1.428	0.001 ±0.001
AHR-0508	6.607 ±5.763	0.006 ±0.005	0.17 ±0.018	0.007 ±0.001	0.007 ±0	0.01 ±0.003	23.189 ±20.722	17.86 ±15.601	0.192 ±0.027	0.106 ±0.096	0.002 ±0	31.666 ±27.564	12.161 ±10.624	4.535 ±1.14	10.849 ±0.861	0.001 ±0
SS-D-0508	1.721 ±2.273	0.006 ±0.006	0.341 ±0.016	0.014 ±0.001	0.014 ±0.001	0.021 ±0.001	0 ±0	3.203 ±5.478	0.527 ±0.049	0.051 ±0.019	0.004 ±0	0 ±0	0.379 ±0.656	7.53 ±0.635	21.718 ±0.812	0.001 ±0
SS-L-1008	3.511 ±2.158	0.05 ±0.008	0.93 ±0.081	0.039 ±0.003	0.041 ±0.003	0.068 ±0.006	0 ±0	14.818 ±4.095	0.992 ±0.087	0.067 ±0.005	0.011 ±0.001	0 ±0	0 ±0	23.338 ±1.951	62.801 ±4.433	0.005 ±0
SS-O-1008	4.696 ±6.029	0.044 ±0.013	0.801 ±0.143	0.033 ±0.006	0.035 ±0.005	0.058 ±0.008	0 ±0	18.019 ±12.669	0.799 ±0.179	0.084 ±0.032	0.009 ±0.002	0 ±0	2.703 ±4.682	20.131 ±3.499	53.297 ±8.305	0.004 ±0.001
ZR-BL-0108	11.041 ±5.661	0.069 ±0.011	1.063 ±0.071	0.043 ±0.002	0.046 ±0.002	0.084 ±0.005	0 ±0	32.462 ±12.908	1.075 ±0.047	0.093 ±0.025	0.013 ±0.001	0.422 ±0.731	5.399 ±6.889	26.659 ±1.856	69.519 ±2.81	0.006 ±0
ZR-LM-1608	7.394 ±3.223	0.032 ±0.005	0.523 ±0.045	0.021 ±0.002	0.024 ±0.001	0.041 ±0.004	2.571 ±4.453	25.048 ±8.06	0.484 ±0.036	0.235 ±0.021	0.007 ±0.001	8.712 ±15.09	7.867 ±6.594	13.567 ±1.118	35.503 ±2.21	0.003 ±0
ZR-AC-1608	12.719 ±4.282	0.045 ±0.006	0.643 ±0.044	0.023 ±0.006	0.029 ±0.002	0.05 ±0.004	1.383 ±2.396	36.936 ±8.422	0.597 ±0.036	0.244 ±0.151	0.005 ±0.005	19.484 ±12.088	15.397 ±6.079	17.486 ±1.174	43.942 ±2.488	0.003 ±0.001
ZR-B-0108	16.123 ±12.204	0.074 ±0.011	0.948 ±0.05	0.036 ±0.005	0.045 ±0.004	0.085 ±0.009	9.367 ±16.224	48.152 ±25.76	0.982 ±0.093	0.482 ±0.054	0.01 ±0.005	29.353 ±25.647	18.436 ±16.004	24.439 ±3.075	64.968 ±7.606	0.005 ±0.001
ZR-B-0808	13.398 ±4.072	0.06 ±0.007	0.853 ±0.08	0.033 ±0.003	0.036 ±0.004	0.064 ±0.006	6.361 ±11.018	39.902 ±10.189	0.799 ±0.071	0.164 ±0.072	0.01 ±0.001	16.767 ±20.061	14.266 ±8.389	21.93 ±1.944	54.989 ±5.034	0.005 ±0
ZR-B-1908	7.12 ±3.588	0.042 ±0.006	0.721 ±0.122	0.028 ±0.007	0.032 ±0.005	0.054 ±0.008	0 ±0	22.29 ±6.744	0.7 ±0.163	0.103 ±0.052	0.006 ±0.006	0 ±0	4.224 ±4.357	19.164 ±2.219	49.898 ±6.723	0.003 ±0.001
ZR-S-0208	13.577 ±5.814	0.082 ±0.007	1.106 ±0.022	0.045 ±0.002	0.051 ±0.003	0.09 ±0.002	1.806 ±3.128	43.613 ±14.292	1.147 ±0.057	0.577 ±0.043	0.014 ±0.001	8.688 ±15.049	9.324 ±10.682	27.664 ±0.303	74.879 ±1.829	0.006 ±0

Appendix 13. Total carbon (TC), total organic carbon (TOC), nitrogen (N) before (N1) and after decarbonation (N2) and carbon to nitrogen ratio (C/N) per field replicate, N, TOC and C/N means and standard deviations (%) across sampling sites in Sweden, Iceland and Greenland. Samples analysed further for organic biomarkers are in *red*.

Sample name	Replicate #	Soil/sediment	N1, %	TC, %	N2, %	Mean N, %	SD N, %	TOC, %	Mean TOC, %	SD TOC, %	C/N	Mean C/N	SD C/N
<b>SWEDEN</b>													
<b>Storglaciären</b>													
SG-BI-0808		sediment	0	0.02	0.03	0.03		0.03	0.03		1.20	1.20	
<i>SG-R-0808</i>	1	sediment	0	0.02	0.05	0.03	0.02	0.01	0.01	0	0.13	0.47	0.58
	2		0.01	0.01	0.04			0.01			0.14		
	3		0.03	0.01	0.01			<i>0.01</i>			1.14		
SG-SS-1008	1	sediment	0.01	0.02	0.01	0.02	0.02	0.01	0.07	0.07	1.36	4.39	3.55
	2		0.04	0.03	0.04			0.15			3.51		
	3		0.01	0.02	0.01			0.05			8.29		
<i>SG-DS-1008</i>	1	sediment	0.01	0.01	0.02	0.03	0	0.22	0.08	0.12	10.56	3.62	6.01
	2		0	0.01	0.03			0			0.08		
	3		0	0.01	0.03			<i>0.01</i>			0.23		
SG-MX-1008	1	sediment	0	0.01	0.02	0.02	0.01	0	0	0	0.26	0.21	0.19
	2		0	0.01	0.03			0			0		
	3		0	0.01	0.02			0.01			0.37		
SG-OM-0808	1	soil	0.03	0.08	0.03	0.04	0.02	0	0	0	0	0.03	0.04
	2		0.04	0.28	0.03			0			0.03		
	3		0	0.10	0.06			0			0.07		
<i>SG-M2-0808</i>	1	soil	0	0.10	0.02	0.04	0.02	<i>0</i>	0	0.03	0.11	0.39	0.44
	2		0.04	0.37	0.03			0.01			0.17		
	3		0.04	0.14	0.06			0.05			0.90		
<i>SG-VP-0808</i>	1	soil	0	0.38	0.03	0.03	0	0.16	0.13	0.11	6.31	4.57	3.81
	2		0.06	0.43	0.03			<i>0.22</i>			7.20		
	3		0.02	0.15	0.03			0.01			0.20		
SG-RB-0808	1	soil	0.01	0.15	0.03	0.04	0.01	0.01	0.04	0.05	0.21	0.95	1.38
	2		0.04	0.32	0.04			0.09			2.55		

	3		0.08	0.37	0.05			0			0.10		
<b>Isfallsglaciären</b>													
IG-NS-1208	1	sediment	0.01	0.06	0	0.01	0.02	0.02	0.05	0.05		3.00	3.00
	2		0.01	0.22	0.04			0.11			3.00		
	3		0	0.08	0			0.03					
IG-SS-1208	1	sediment	0	0.04	0.02	0.02	0.01	0.02	0.02	0	0.80	1.97	1.17
	2		0.03	0.04	0.01			0.02			1.96		
	3		0	0.06	0.01			0.03			3.14		
IG-EL-1208	1	sediment	0.03	0.04	0.02	0.02	0.01	0.01	0.01	0	0.63	0.50	0.25
	2		0	0.02	0.03			0.01			0.21		
	3		0	0.02	0.01			0.01			0.65		
IG-DL-1108	1	sediment	0.03	0.02	0.01	0.02	0.02	0.04	0.03	0.01	2.81	4.69	6.46
	2		0	0.06	0.03			0.03			0.96		
	3		0.03	0.04	0.00			0.02			14.29		
	3				0.03			0.02			0.72		
IG-DR-1108	1	sediment	0.01	0.06	0	0	0.01	0.02	0.02	0		1.90	0.92
	2		0	0.04	0.01			0.02			1.25		
	3		0.01	0.03	0.01			0.02			2.55		
IG-LITMOR-1808	1	soil	0	0.02	0	0	0.01	0.01	0.03	0.02		4.55	2.74
	2		0.03	0.07	0.01			0.02			2.61		
	3		0.01	0.16	0.01			0.05			6.49		
IG-LM-1208	1	soil	8.00	0.05	0	0.01	0.01	0.02	0.02	0.01		1.30	1.15
	2		0	0.06	0.02			0.01			0.48		
	3		0.05	0.04	0.01			0.02			2.11		
IG-OM-1808	1	soil	0.01	0.09	0	0.02	0.02	0.04	0.06	0.03		4.66	4.34
	2		0.02	0.21	0.04			0.06			1.59		
	3		0	0.11	0.01			0.10			7.73		
<b>Tarfalajaure transect</b>													
<b>Sydöstra Kaskatjökkoglaciären</b>													
CB-1408	1	sediment	0	0.04	0.01	0.01	0.01	0.02	0.02	0.01	1.42	2.85	2.01
	2		0.01	0.09	0.01			0.04			4.27		
	3		0.01	0.04	0			0.02					

<b>Kebnepakteglaciären</b>													
KG-BI-1308		sediment	0.04	0.02	0.08	0.08		0.61	0.61		7.44	7.44	
<b>KG-MW-1308</b>	<b>1</b>	sediment	0.01	0.03	0.01	0.01	0.01	<b>0.01</b>	0.01	0.01	1.00	1.57	1.53
	<b>2</b>		0	0.04	0.01			<b>0.02</b>			3.31		
	<b>3</b>		0.02	0.03	0.03			<b>0.01</b>			0.41		
<b>Tarfalajaure and Tarfalajäkk</b>													
<b>T-L-1308</b>	<b>1</b>	soil	0.11	1.42	0.08	0.05	0.03	<b>0.65</b>	0.44	0.18	7.76	10.37	5.50
	<b>2</b>		0.04	0.67	0.02			<b>0.36</b>			16.68		
	<b>3</b>		0.08	1.03	0.05			<b>0.31</b>			6.67		
<b>TL-RB-1408</b>	<b>1</b>	soil	0.12	1.40	0.07	0.06	0.01	<b>0.73</b>	0.65	0.14	10.30	10.19	1.62
	<b>2</b>		0.11	1.49	0.06			<b>0.73</b>			11.75		
	<b>3</b>		0.06	0.79	0.06			<b>0.49</b>			8.51		
<b>MS-TL-1408</b>	<b>1</b>	sediment	0.04	0.52	0.03	0.03	0.01	<b>0.24</b>	0.29	0.21	8.20	9.24	4.52
	<b>2</b>		0.07	1.12	0.04			<b>0.51</b>			14.19		
	<b>3</b>		0.05	0.27	0.02			<b>0.11</b>			5.33		
MS+IG-1908	1	soil	0.25	3.56	0.06	0.06	0.01	1.00	1.08	0.09	16.67	18.40	4.39
	2		0.32	4.46	0.05			1.17			23.40		
	3		0.28	3.72	0.07			1.06			15.14		
ML-1908	1	soil	0.09	1.12	0.03	0.05	0.02	0.42	1.06	0.84	14.00	20.57	7.48
	2		0.17	3.02	0.07			2.01			28.71		
	3		0.11	1.53	0.04			0.76			19.00		
MS-1008	1	sediment	0.10	1.05	0.04	0.02	0.01	0.16	0.09	0.06	4.40	4.40	3.24
	2		0.05	0.48	0.03			0.03			1.16		
	3		0.09	1.50	0.01			0.07			7.64		
<b>ICELAND</b>													
<b>Svínafellsjökull</b>													
SV-Toplce-3105	1	sediment	0.07	0.29	0.03	0.03	0.01	0.02	0.09	0.12	0.68	2.18	2.67
	2		0.06	0.07	0.04			0.23			5.26		
	3		0.04	0.26	0.02			0.01			0.60		
SV-BI-2705		sediment	0.04	0.21	0.05	0.05		0.05	0.05		0.94	0.94	
SV-BI-3105	1	sediment	0.02	0.21	0.03	0.04	0.03	0.01	0.27	0.46	0.20	3.86	6.14
	2		0.08	0.20	0.07			0.81			10.95		

	3		0.07	0.19	0.02			0.01			0.44		
SV-PL-2905	1	sediment	0	0.04	0.03	0.02	0.01	0.01	0.01	0	0.23	0.62	0.41
	2		0.01	0.18	0.02			0.01			0.57		
	2				0.01			0.01			1.06		
	3		0	0.18	0			0.01					
SV-PL-2109	1	sediment	0.02	0.12	0.03	0.03	0.01	0.04	0.04	0.01	1.33	1.33	0.33
	2		0.02	0.08	0.04			0.04			1.00		
	3		0.02	0.15	0.03			0.05			1.67		
SV-R-3005	1	sediment	0	0.05	0.02	0.03	0.01	0.01	0	0	0.34	0.17	0.17
	2		0	0.09	0.02			0			0.16		
	3		0	0.04	0.05			0			0		
SV-R-2109	1	soil	0.03	0.61	0.03	0.03	0.01	0.23	0.28	0.14	7.67	8.03	2.56
	2		0.04	0.41	0.03			0.17			5.67		
	3		0.04	0.72	0.04			0.43			10.75		
SV-YM-2009	1	sediment	0.01	0.06	0.03	0.03	0.01	0.01	0.01	0.00	0.33	0.39	0.10
	2		0.01	0.07	0.03			0.01			0.33		
	3		0.01	0.07	0.02			0.01			0.50		
<b>SV-LM-2905</b>	<b>1</b>	soil	0.06	0.96	0.09	0.06	0.03	<b>0.40</b>	0.54	0.19	4.48	9.41	4.28
	<b>2</b>		0.03	1.11	0.06			<b>0.75</b>			11.67		
	<b>3</b>		0.07	0.80	0.04			<b>0.47</b>			12.08		
SV-LM-2109	1	soil	0.09	2.00	0.07	0.06	0.01	1.01	0.75	0.25	14.43	13.08	2.49
	2		0.06	1.29	0.05			0.51			10.20		
	3		0.07	1.15	0.05			0.73			14.60		
<b>SV-LIAM-3005</b>	<b>1</b>	soil	0.40	5.27	0.37	0.44	0.19	<b>4.22</b>	5.24	2.68	11.41	11.66	0.77
	<b>2</b>		0.56	6.94	0.66			<b>8.28</b>			12.53		
	<b>3</b>		0.34	4.18	0.29			<b>3.22</b>			11.04		
SV-LIAM-2109	1	soil	0.03	0.15	0.02	0.02	0.01	0.05	0.03	0.02	2.50	1.39	0.98
	2		0.02	0.10	0.03			0.02			0.67		
	3		0.02	0.11	0.02			0.02			1.00		
<b>SV-2.5K-3105</b>	<b>1</b>	soil	0.15	2.79	0.16	0.17	0.07	<b>1.92</b>	2.97	1.80	11.76	16.81	4.39
	<b>2</b>		0.20	3.85	0.13			<b>2.79</b>			20.82		
	<b>3</b>		0.13	2.51	0.11			<b>1.59</b>			14.53		

	<b>4</b>		0.27	6.00	0.28			<b>5.56</b>			20.12		
SV-2.5K-2109	1	soil	0.16	2.82	0.07	0.10	0.04	1.06	2.02	0.90	15.14	20.41	3.64
	2		0.25	5.52	0.07			1.57			22.43		
	3		0.24	4.99	0.15			3.13			20.87		
	4		0.30	6.50	0.10			2.32			23.20		
<b>Virkisjökull</b>													
VK-SIS-0206	1	sediment	0.04	0.14	0.04	0.03	0.01	0.01	0.06	0.05	0.29	2.97	3.22
	2		0.02	0.06	0.01			0.09			7.37		
	2				0.03			0.11			3.35		
	3		0.02	0.10	0.03			0.02			0.88		
VK-SI-0206		sediment	0.03	0.13	0.02	0.02		0.06	0.06		3.97	3.97	
VK-BI-0206		sediment	0.05	0.03	0.03	0.03		0.08	0.08		2.44	2.44	
VK-US-2605	1	sediment	0	0.05	0	0.01	0.02	0	0	0		0.20	0.24
	2		0.01	0.07	0.01			0			0.37		
	3		0	0.04	0.03			0			0.03		
VK-US-1909	1	sediment	0.01	0.07	0.03	0.03	0.01	0.01	0.01	0.00	0.33	0.31	0.05
	2		0.01	0.03	0.03			0.01			0.33		
	3		0.02	0.04	0.04			0.01			0.25		
VK-PL-2605	1	sediment	0	0.04	0.03	0.027	0	0	0.003	0	0.15	0.10	0.05
	2		0.01	0.03	0.03			0			0.06		
	3		0	0.04	0.03			0			0.08		
VK-PL-1909	1	sediment	0.01	0.04	0.02	0.02	0.01	0.01	0.01	0.00	0.50	0.67	0.31
	2		0.01	0.03	0.02			0.01			0.50		
	2				0			0.01					
	2				0.01			0.01			1.00		
	2				0.01			0.01			1.00		
	3		0.01	0.12	0.03			0.01			0.33		
VK-DS-2705	1	sediment	0.01	0.03	0.02	0.03	0.01	0	0.01	0	0.20	0.27	0.12
	2		0	0.02	0.03			0.01			0.19		
	3		0.04	0.02	0.02			0.01			0.41		
VK-DS-2009	1	sediment	0.02	0.05	0.03	0.03	0.01	0.02	0.03	0.01	0.67	1.00	0.33
	2		0.01	0.06	0.03			0.04			1.33		

	3		0.01	0.04	0.02			0.02			1.00		
<b>VK-YM-0206</b>	<b>1</b>	soil	0.04	0.18	0.02	0.03	0.01	<b>0.09</b>	0.07	0.02	4.14	2.64	1.45
	2		0.01	0.16	0.03			0.07			2.53		
	3		0	0.08	0.04			0.05			1.25		
VK-YM-1909	1	soil	0.02	0.06	0.01	0.02	0.01	0.03	0.02	0.01	3.00	1.44	1.39
	2		0.01	0.06	0.02			0.02			1.00		
	3		0.01	0.03	0.03			0.01			0.33		
VK-M2-1909	1	soil	0.02	0.13	0.03	0.02	0.01	0.08	0.10	0.02	2.67	4.39	1.67
	2		0.03	0.22	0.02			0.09			4.50		
	3		0.03	0.18	0.02			0.12			6.00		
<b>VK-OM-0206</b>	1	soil	0	0.18	0.03	0.05	0.01	0.09	0.58	0.36	2.71	9.99	5.26
	<b>2</b>		0.07	1.08	0.06			<b>0.62</b>			10.32		
	2				0.05			0.64			11.73		
	3		0.10	1.91	0.06			0.97			15.19		
VK-OM-1909	1	soil	0.06	1.12	0.06	0.05	0.01	0.69	0.75	0.07	11.50	14.72	5.01
	2		0.06	1.24	0.06			0.73			12.17		
	3		0.10	2.05	0.04			0.82			20.50		
<b>VK-RM-2709</b>	<b>1</b>	soil	0.22	6.35	0.10	0.11	0.05	<b>3.34</b>	4.71	3.07	33.40	39.44	7.88
	2		0.28	7.77	0.07			2.56			36.57		
	3		0.27	8.05	0.17			8.22			48.35		
<b>Kviarjökull</b>													
KV-PL-2409	1	sediment	0.02	0.08	0.04	0.02	0.01	0.04	0.02	0.01	1.00	1.11	0.46
	2		0.02	0.04	0.03			0.02			0.67		
	3		0.02	0.03	0.01			0.02			2.00		
	3				0.01			0.01			1.00		
	3				0.01			0.01			1.00		
	3				0.01			0.01			1.00		
KV-LM-2409	1	soil	0.03	0.17	0.04	0.04	0	0.06	0.05	0.01	1.50	1.25	0.25
	2		0.03	0.21	0.04			0.05			1.25		
	3		0.02	0.09	0.04			0.04			1.00		
KV-R-2409	1	soil	0.04	0.28	0.04	0.03	0.01	0.13	0.14	0.01	3.25	5.92	2.32
	2		0.04	0.38	0.02			0.14			7.00		

	3		0.04	0.39	0.02			0.15			7.50		
<b>KV-M2-2409</b>	<b>1</b>	soil	0.04	0.49	0.02	0.03	0.01	<b>0.17</b>	0.18	0.02	8.50	7.75	3.19
	2		0.06	0.60	0.02			0.21			10.50		
	3		0.05	0.44	0.04			0.17			4.25		
<b>KV-M3-2409</b>	1	soil	0.12	1.95	0.05	0.05	0.01	0.45	0.59	0.12	9.00	12.19	4.05
	2		0.07	1.48	0.04			0.67			16.75		
	<b>3</b>		0.10	1.34	0.06			<b>0.65</b>			10.83		
<b>Fjallsjökull</b>													
FJ-PL-2309	1	sediment	0.02	0.03	0.02	0.02	0.01	0.03	0.02	0.01	1.50	1.11	0.67
	2		0.01	0.05	0.03			0.01			0.33		
	3		0.02	0.07	0.02			0.03			1.50		
FJ-R-2309	1	sediment	0.01	0.06	0.02	0.02	0.01	0.02	0.04	0.04	1.00	1.94	2.24
	2		0.01	0.02	0.03			0.01			0.33		
	3		0.02	0.25	0.02			0.09			4.50		
<b>FJ-LM-2309</b>	1	soil	0.03	0.34	0.03	0.03	0.01	0.13	0.14	0.06	4.33	5.17	1.30
	<b>2</b>		0.04	0.48	0.03			<b>0.20</b>			6.67		
	3		0.03	0.22	0.02			0.09			4.50		
<b>FJ-M2-2309</b>	<b>1</b>	soil	0.08	1.68	0.07	0.06	0.04	<b>1.10</b>	0.86	0.59	15.71	13.02	3.55
	2		0.11	2.11	0.09			1.29			14.33		
	3		0.04	0.64	0.02			0.18			9.00		
<b>FJ-M3-2309</b>	1	soil	0.14	3.58	0.08	0.07	0.01	1.75	1.44	0.43	21.88	19.32	3.13
	2		0.07	1.15	0.06			0.95			15.83		
	<b>3</b>		0.11	2.38	0.08			<b>1.62</b>			20.25		
<b>Skaftafellsjökull</b>													
SK-PL-2209	1	sediment	0.01	0.05	0.02	0.02	0	0.02	0.02	0	1.00	1.00	0
	2		0.01	0.04	0.02			0.02			1.00		
	3		0.01	0.03	0.02			0.02			1.00		
SK-R-2209	1	sediment	0.01	0.03	0.02	0.02	0	0.02	0.01	0.01	1.00	0.67	0.29
	2		0.02	0.02	0.02			0.01			0.50		
	3		0.01	0.05	0.02			0.01			0.50		
SK-TM-2209	1	soil	0.02	0.03	0.01	0.02	0.01	0.02	0.02	0.01	2.00	1.33	0.76
	2		0.02	0.04	0.02			0.03			1.50		

	3		0.01	0.02	0.02			0.01			0.50		
SK-M2-2209	1	soil	0.06	1.12	0.05	0.05	0.01	0.67	0.57	0.10	13.40	12.13	1.21
	2		0.08	1.13	0.05			0.55			11.00		
	3		0.07	1.16	0.04			0.48			12.00		
<b>Skeiðarárjökull</b>													
SD-LM-2609	1	soil	0.02	0.08	0.02	0.03	0.01	0.02	0.03	0.01	1.00	1.00	0
	2		0.02	0.11	0.04			0.04			1.00		
	3		0.02	0.08	0.03			0.03			1.00		
SD-M2-2609	1	soil	0.06	0.71	0.03	0.02	0.01	0.26	0.20	0.06	8.67	10.89	2.78
	2		0.06	0.82	0.02			0.20			10.00		
	3		0.05	0.41	0.01			0.14			14.00		
<b>GREENLAND</b>													
<b>AHR-0508</b>	<b>1</b>	soil	0.34	4.82	0.14	0.12	0.07	<b>2.33</b>	2.33	1.52	16.64	19.43	2.48
	2		0.51	7.35	0.04			0.81			20.25		
	3		0.14	1.84	0.18			3.85			21.39		
G-1408	1	sediment	0.01	0.03	0.01	0.01	0.01	0.02	0.02	0.02	2.00	1.63	1.11
	1				0.01			0.03			3.00		
	1				0.01			0.01			1.00		
	1				0			0.01					
	2		0.01	0.03	0.02			0.01			0.50		
	3		0.02	0.05	0			0.05					
<b>G-1408</b>	1	soil	0.08	0.84	0.01	0.04	0.02	0	0.30	0.15	0	7.21	3.61
	1				0.04			0.37			9.25		
	1				0.05			0.38			7.60		
	1				0.05			0.40			8.00		
	2		0.07	0.58	0.03			0.29			9.67		
	<b>3</b>		0.10	0.95	0.04			<b>0.35</b>			8.75		
<b>Store Sø Lake</b>													
SS-D-0508	1	sediment	0.03	0.26	0.02	0.02	0.01	0.07	0.14	0.07	3.50	6.22	3.75
	2		0.03	0.34	0.02			0.21			10.50		
	3		0.03	0.29	0.03			0.14			4.67		
<b>SS-D-0508</b>	1	soil	0.17	2.01	0.07	0.09	0.02	1.06	1.27	0.26	15.14	13.74	1.67

	2		0.22	2.73	0.11			1.56			14.18		
	3		0.14	1.60	0.10			1.19			11.90		
<b>SS-LI-1408</b>	1	soil	0.60	11.77	0.40	0.23	0.15	8.96	4.88	3.56	22.40	20.54	1.64
	2		0.35	5.54	0.17			3.28			19.29		
	3		0.33	3.97	0.12			2.39			19.92		
SS-L-1008	1	sediment	0.16	1.83	0.06	0.08	0.07	0.91	1.33	1.38	15.17	14.56	3.79
	2		0.40	7.51	0.16			2.88			18.00		
	3		0.05	0.38	0.02			0.21			10.50		
<b>SS-L-1008</b>	1	soil	0.16	1.52	0.08	0.10	0.07	1.01	1.28	1.04	12.63	12.04	1.82
	2		0.10	0.84	0.04			0.40			10.00		
	3		0.25	3.01	0.18			2.43			13.50		
SS-L-1308	1	sediment	0.02	0.11	0.01	0.03	0.02	0.07	0.23	0.16	7.00	7.58	1.94
	2		0.05	0.54	0.04			0.24			6.00		
	3		0.07	0.80	0.04			0.39			9.75		
<b>SS-L-1308</b>	1	soil	0.33	4.71	0.14	0.11	0.04	2.41	1.92	0.46	17.21	18.50	2.41
	2		0.26	4.02	0.07			1.49			21.29		
	3		0.22	3.51	0.11			1.87			17.00		
<b>SS-O-1008</b>	1	soil	0.19	3.89	0.11	0.09	0.02	2.52	1.76	0.66	22.91	19.98	3.09
	2		0.15	2.10	0.08			1.34			16.75		
	3		0.17	2.69	0.07			1.42			20.29		
<b>Zackenbergriver</b>													
ZR-SS-1508	1	sediment	0.06	0.55	0.04	0.03	0.01	0.41	0.34	0.06	10.25	11.08	4.06
	2		0.07	0.67	0.02			0.31			15.50		
	3		0.06	0.65	0.04			0.30			7.50		
<b>ZR-SS-1508</b>	1	soil	0.12	1.17	0.05	0.05	0.02	0.64	0.56	0.07	12.80	12.82	4.17
	2		0.11	1.18	0.03			0.51			17.00		
	3		0.09	0.93	0.06			0.52			8.67		
ZR-BL-0108	1	sediment	0.01	0.04	0.03	0.02	0.01	0.02	0.01	0.01	0.67	0.73	0.25
	2		0.01	0.04	0.04			0.02			0.50		
	3		0.01	0.04	0.02			0.01			0.50		
	3				0			0.01					
	3				0.01			0.01			1.00		

	3				0.01			0.01			1.00		
<b>ZR-BL-0108</b>	1	soil	0.41	5.87	0.26	0.22	0.04	3.74	3.05	0.60	14.38	13.88	1.34
	2		0.29	3.69	0.18			2.68			14.89		
	3		0.45	5.26	0.22			<b>2.72</b>			12.36		
ZR-LM-1608	1	sediment	0.02	0.17	0.02	0.02	0.02	0.13	0.09	0.04	6.50	4.50	2.29
	2		0.01	0.12	0.01			0.05			5.00		
	3		0.02	0.13	0.04			0.08			2.00		
<b>ZR-LM-1608</b>	1	soil	0.25	3.36	0.15	0.17	0.03	1.91	2.17	0.31	12.73	13.09	0.73
	2		0.30	3.81	0.20			2.52			12.60		
	3		0.27	3.82	0.15			<b>2.09</b>			13.93		
ZR-AC-1608	1	sediment	0.03	0.73	0.04	0.03	0.01	0.73	0.51	0.19	18.25	16.17	5.20
	1		0.02	0.44	0.02			0.40			20.00		
	3		0.03	0.37	0.04			0.41			10.25		
<b>ZR-AC-1608</b>	1	soil	0.07	1.02	0.04	0.04	0.01	<b>0.53</b>	0.45	0.07	13.25	12.53	2.26
	2		0.06	0.81	0.04			0.40			10.00		
	3		0.07	1.02	0.03			0.43			14.33		
ZR-B-0808	1	sediment	0.02	0.10	0.02	0.02	0.01	0.13	0.21	0.07	6.50	9.11	3.07
	2		0.03	0.33	0.02			0.25			12.50		
	3		0.01	0.14	0.03			0.25			8.33		
<b>ZR-B-0808</b>	1	soil	0.27	3.78	0.09	0.10	0.05	<b>1.54</b>	1.55	0.78	17.11	15.21	2.07
	2		0.21	2.87	0.15			2.33			15.53		
	3		0.13	1.74	0.06			0.78			13.00		
ZR-S-0208	1	sediment	0.01	0.08	0.03	0.02	0.01	0.03	0.04	0.01	1.00	1.83	0.76
	2		0.01	0.08	0.02			0.05			2.50		
	3		0.01	0.12	0.02			0.04			2.00		
<b>ZR-S-0208</b>	1	soil	0.12	1.62	0.09	0.08	0.02	<b>0.98</b>	0.89	0.11	10.89	11.70	1.01
	2		0.12	1.62	0.06			0.77			12.83		
	3		0.10	1.34	0.08			0.91			11.38		

Appendix 14. Mean (mg/kg) and standard deviation (mg/kg) of replicates (mostly triplicates) of analysed metal concentrations across sampling sites in Sweden, Iceland and Greenland.

Sample	Type	Ca	Cr	Mn	Co	Ni	Cu	Zn	Al	P	S	As	Pb	Na	K	Mg	Fe	Cd
<b>SWEDEN</b>																		
<b>Tarfalajaure transect</b>																		
CB-1408	soil	1845 ±638	8.46± 2.27	66± 32	3.14± 0.78	3.36± 0.35	11.61 ±0.97	3.41± 0.74	2656± 848	120± 6	4.86± 3.73	0	0.15± 0.04	271± 81	274± 52	1102 ±303	4935± 1675	0.41± 0.13
KG-BI-1308	sed	2773	10.52	89	3.39	3.47	11.66	3.39	3085	123	12.10	0	0.61	299	139	1363	4625	0.28
KG-MW-1308	sed	1363 ±251	5.41± 0.73	44± 11	2.11± 0.28	2.4± 0.17	9.11± 0.88	2.21± 0.3	1742± 314	106± 5	36.92± 17.78	0	0.18± 0.01	187± 35	192± 24	839± 113	3253± 464	0.27± 0.04
T-L-1308	soil	2350 ±244	7.9± 0.82	84± 17	3.52± 0.08	4.93± 0.31	17.76 ±1.64	3.29± 0.29	2962± 305	135± 9	15± 15.29	0	0.63± 0.09	252± 19	173± 8	1275 ±130	4721± 577	0.4± 0.04
TL-RB-1408	soil	2092 ±361	11.11 ±1.4	66± 17	3.09± 0.26	4.28± 0.11	13.4± 0.2	3.93± 0.28	3213± 472	144± 5	13.12± 8.77	0	0.63± 0.03	354± 55	186± 16	1137 ±146	4840± 795	0.42± 0.06
MS-TL-1408	sed	2262 ±284	8.55± 1.15	83± 12	2.89± 0.48	2.66± 0.58	9.33± 2.47	3.84± 0.93	3028± 460	136± 18	6.62± 8.84	0	0.27± 0.09	292± 47	262± 101	1185 ±200	4963± 707	0.41± 0.05
MS+IG-1908	soil	2328 ±533	10.15 ±1.64	91± 27	2.65± 0.48	2.58± 0.3	7.65± 0.75	3.79± 0.32	3266± 608	118± 12	45.53± 2.04	0	1.16± 0.31	298± 56	205± 59	1244 ±221	5456± 1071	0.41± 0.07
ML-1908	soil	3346 ±552	13.88 ±1.97	106 ±16	3.95± 0.59	4.42± 0.66	16.08 ±2.82	5.44± 0.72	4436± 619	153± 14	19.77± 12.19	0	0.86± 0.19	464± 92	295± 63	1831 ±323	6765± 1024	0.51± 0.07
MS-1008	sed	2332 ±342	8.42± 1.13	64± 11	3.23± 0.05	3.6± 0.44	10.34 ±0.86	2.78± 0.3	2412± 435	128± 3	6.67± 11.56	0	0.14± 0.25	263± 56	130± 21	1161 ±144	3537± 426	0.21± 0.03
<b>Storglaciären</b>																		
SG-BI-0808	sed	2095	1.38	106	5.71	1.63	7.06	10.83	2518	154	0	0	0.36	401	92	1098	8875	0.50
SG-R-0808	sed	4162 ±622	5.94± 5.27	173 ±36	5.89± 0.79	3.06± 1.11	14.97 ±2	11.4± 5.82	3983± 225	218± 29	146.27± 103.49	0.14± 0.16	0.15± 0.1	494± 92	231± 64	1740 ±128	9354± 1997	0.54± 0.1
SG-SS-1008	sed	3444 ±516	11.34 ±1.43	103 ±16	3.69± 0.55	3.46± 0.4	10.06 ±0.96	3.32± 0.35	3426± 298	125± 10	2.47± 3.49	0	0.12± 0.04	369± 13	159± 1	1544 ±199	4932± 624	0.31± 0.04
SG-DS-1008	sed	2394 ±190	8.72± 0.72	76± 13	3.15± 0.27	2.77± 0.38	7.75± 2.1	2.5± 0.5	2499± 237	103± 30	19.46± 23.76	0	0.12± 0.21	237± 79	113± 20	1073 ±144	3808± 292	0.23± 0.01

SG-MX-1008	sed	2167 ±522	7.25± 1.67	77± 24	2.98± 0.71	2.17± 0.15	6.18± 0.51	1.85± 0.31	2121± 523	116± 7	20.33± 3.81	0	0	156± 44	78±1 4	861± 194	3477± 945	0.2± 0.06
SG-OM-0808	soil	1993 ±347	5.1±3 .88	82± 37	3± 0.63	2± 1.25	5.33± 2.51	4.6± 4.36	2367± 769	130± 51	24.01± 22.02	0	0.02± 0.03	186± 72	223± 196	948± 201	4138± 1834	0.25± 0.11
SG-M2-0808	soil	5129 ±958	3.61± 1.15	192 ±2	6.31± 0.27	2.57± 0.39	13.42 ±0.34	14.01± 0.9	4847± 1352	203± 43	161.88± 100.67	0.07± 0.09	0.45± 0.48	1008 ±624	258± 77	1997 ±714	10734 ±720	0.62± 0.05
SG-VP-0808	soil	3177 ±105	10.3± 0.71	103 ±22	3.75± 0.33	3.04± 0.72	7.99± 2.74	2.91± 0.52	3095± 36	115± 6	8.4± 14.55	0	0.14± 0.13	268± 63	125± 29	1357 ±150	4721± 179	0.28± 0.01
SG-RB-0808	soil	4878 ± 2245	1.98± 0.5	210 ±8	6.13± 1.87	1.5± 0.59	5.97± 2.72	13.64± 0.35	4758± 1844	240± 15	267.38± 188.34	0.19± 0.18	0.41± 0.28	1338 ±942	423± 166	2107 ±937	10854 ±2647	0.63± 0.14
<b>Isfallsglaciären</b>																		
IG-NS-1208	sed	2363 ±817	11.41 ±3.03	78± 34	2.71± 0.63	2.82± 0.39	9.2± 1.4	2.59± 0.61	2900± 987	114± 3	4.33± 2.92	0	0.33± 0.05	342± 101	223± 46	1106 ±353	4617± 1609	0.39± 0.13
IG-SS-1208	sed	2707 ±459	9.25± 2.81	59± 51	3.04± 0.65	3.07± 0.86	9.81± 1.19	2.7± 0.62	3096± 736	134± 15	38.33± 2.35	0	0.33± 0.17	166± 120	115± 81	1274 ±333	4401± 241	0.25± 0.22
IG-EL-1208	sed	2897 ±210	11.84 ±0.75	90± 5	3.23± 0.3	3.22± 0.27	8.53± 0.7	2.85± 0.13	3400± 230	106± 5	3.4±3.12	0	0.07± 0.02	423± 31	204± 19	1221 ±107	4989± 335	0.43± 0.03
IG-DL-1108	sed	2543 ±796	9.52± 2.54	80± 33	3.35± 0.49	3.51± 0.46	10.21 ±0.09	2.66± 0.63	2713± 877	109± 7	5.67± 7.28	0	0.07± 0.05	255± 83	110± 23	1123 ±321	4011± 1268	0.24± 0.08
IG-DR-1108	sed	2550 ±311	9.94± 1.79	77± 13	3.52± 0.28	3.79± 0.19	10.82 ±0.71	2.7± 0.21	2699± 320	110± 3	0	0	0± 0.01	262± 24	110± 9	1146 ±116	4038± 528	0.24± 0.03
IG-LITMOR-1808	soil	1957 ±681	7.91± 1.83	51± 24	2.57± 0.45	3.34± 0.14	9.61± 0.17	2.5± 0.28	2197± 720	102± 3	4.71± 4.46	0	0.09± 0.04	292± 93	146± 40	885± 299	3178± 1075	0.27± 0.09
IG-LM-1208	soil	2039 ±229	8.52± 0.9	55± 8	2.7± 0.18	3.26± 0.3	9.31± 1.51	2.29± 0.08	2291± 225	101± 9	3.45± 5.98	0	0.11± 0.04	298± 20	179± 17	940± 98	3388± 409	0.29± 0.03
IG-OM-1808	soil	1920 ±400	7.43± 0.87	58± 15	2.46± 0.33	3.19± 0.38	10.95 ±4.44	2.28± 0.31	2231± 414	94±3	11.23± 18.02	0	0.11± 0.02	250± 41	134± 10	864± 155	3288± 652	0.28± 0.05
<b>ICELAND</b>																		
<b>Svínafellsjökull</b>																		
SV-Toplce-3105	sed	2333 ±651	7.93± 2.4	85± 29	3.06± 0.56	2.68± 0.63	7.56± 3.41	2.8± 0.52	2466± 696	114± 14	9.57± 8.36	0	0.2± 0.33	184± 98	139± 10	1055 ±291	3974± 1067	0.24± 0.07

SV-BI-2705	sed	2553	1.33	119	5.32	1.48	6.27	10.33	2923	175	6.48	0	0.33	513	118	1165	8163	0.47
SV-BI-3105	sed	3298 ±86	10.97 ±1.12	111 ±24	3.87± 0.08	3.38± 0.94	10.43 ±3.77	3.21± 0.81	3460± 151	116± 12	7.98± 8.87	0	0.29± 0.51	330± 87	136± 32	1430 ±154	5164± 212	0.31± 0.01
SV-PL-2905	sed	3331 ±426	2.43± 0.3	149 ±5	4.84± 0.46	2.03± 0.05	11.58 ±1.42	12.89± 0.5	2865± 112	195± 16	54.1± 31.1	0	0.27± 0.1	409± 30	182± 21	1378 ±99	9491± 328	0.64± 0.13
SV-PL-2109	sed	2953 ±334	2.4± 0.23	164 ±16	4.34± 0.37	1.96± 0.27	12.5± 0.47	12.91± 0.63	3166± 386	180± 25	28.36± 12.29	0	0.3± 0.05	471± 87	230± 32	1593 ±71	11305 ±689	0.63± 0.06
SV-YM-2009	sed	3213 ±179	1.8± 0.17	131 ±7	4.28± 0.12	2.04± 0.27	8.68± 0.33	11.82± 0.46	3060± 148	185± 7	113.79± 94.6	0	0.26± 0.01	522± 69	184± 18	1410 ±56	9049± 651	0.65± 0.03
SV-R-3005	sed	2908 ±336	2.82± 0.19	160 ±18	5.98± 0.31	2.73± 0.05	12.75 ±0.35	14.78± 1.35	2644± 305	238± 14	73.25± 29.75	0	0.21± 0.02	279± 65	120± 40	1660 ±44	11204 ±415	0.58± 0.02
SV-R-2109	soil	2973 ±203	2.98± 0.43	159 ±22	4.7± 0.17	2.23± 0.15	12.7± 0.8	14.36± 0.98	3230± 393	186± 11	65.62± 3.39	0	0.35± 0.03	478± 37	190± 28	1489 ±101	11207 ±1423	0.76± 0.02
SV-LM-2905	soil	3371 ±808	2.66± 0.4	156 ±20	5.08± 1.08	2.25± 0.34	10.45 ±0.46	12.4± 0.25	3702± 729	183± 26	33.11± 16.98	0	0.25± 0.06	711± 290	239± 59	1713 ±372	9841± 862	0.67± 0.1
SV-LM-2109	soil	3097 ±147	2.87± 0.19	152 ±15	4.75± 0.12	2.19± 0.15	11.03 ±2.07	13.01± 0.6	3608± 22	172± 3	33.8± 5.04	0	0.55± 0.18	593± 50	220± 22	1587 ±57	10506 ±819	0.74± 0.01
SV-LIAM-3005	soil	3445 ±391	2.79± 0.38	175 ±16	4.79± 0.46	2± 0.15	9.65± 0.46	13.12± 0.65	3600± 155	236± 16	112.3± 23.63	0.04± 0.05	0.52± 0.18	646± 61	282± 18	1497 ±90	9358± 434	0.58± 0.09
SV-LIAM-2109	soil	3141 ±143	2.49± 0.01	145 ±5	4.65± 0.16	2.19± 0.06	13.54 ±0.42	13.52± 0.34	2993± 198	191± 23	63.89± 24.52	0	0.31± 0.01	485± 64	156± 16	1365 ±29	11069 ±224	0.7± 0.01
SV-2.5K-3105	soil	4334 ±591	2.58± 0.25	179 ±21	6.72± 1.11	2.21± 0.16	8.79± 0.65	13.87± 0.7	4790± 530	209± 19	91.99± 19.67	0.08± 0.06	1.01± 0.05	1107 ±176	302± 40	2035 ±191	11116 ±920	0.67± 0.07
SV-2.5K-2109	soil	3827 ±271	2.45± 0.1	164 ±11	5.4± 0.28	2.08± 0.06	7.27± 0.47	13.29± 0.41	4375± 204	198± 9	89.53± 10.03	0	1.08± 0.15	919± 86	293± 30	1943 ±137	11349 ±849	0.75± 0.04
<i>Virkisjökull</i>																		
VK-SIS-0206	sed	2201 ±211	8.17± 0.8	77± 18	3.05± 0.37	2.84± 0.26	8.72± 2.71	2.32± 0.44	2293± 302	100± 14	13.72± 7.8	0	0.06± 0.1	177± 18	124± 57	992± 110	3625± 626	0.22± 0.04
VK-SI-0206	sed	2080	8.47	63	2.71	2.98	7.55	2.30	2130	101	2.71	0	0.07	179	116	1007	3351	0.20
VK-BI-0206	sed	3409	1.64	140	5.48	1.63	7.31	11.31	4100	167	8.11	0	0.49	873	196	1424	9246	0.54
VK-US-2605	sed	2717 ±373	1.1± 0.29	134 ±17	4.62± 0.51	1.2± 0.3	4.86± 0.89	11.23± 0.53	2992± 568	206± 15	56.55± 35.71	0	0.18± 0.14	506± 86	234± 32	1079 ±97	9054± 756	0.62± 0.15

VK-US-1909	sed	2883 ±539	1.41± 0.44	140 ±6	4.45± 0.29	1.39± 0.16	5.62± 0.24	11.79± 1.32	3027± 618	197± 13	61.36± 37.25	0	0.24± 0.07	477± 57	256± 44	1199 ±76	9680± 511	0.64± 0.02
VK-PL-2605	sed	3140 ±182	1.13± 0.1	137 ±6	5.16± 0.5	1.34± 0.13	5.52± 0.61	10.8± 0.56	3651± 209	183± 12	56.77± 25.93	0	0.03± 0.01	592± 64	221± 13	1152 ±95	8658± 461	0.48± 0.02
VK-PL-1909	sed	2314 ±693	1.14± 0.17	120 ±15	4.31± 0.21	1.44± 0.08	5.56± 0.73	12.2± 1.43	2377± 960	205± 8	5.06± 2.82	0	0.28± 0.05	415± 172	205± 38	1123 ±99	9263± 744	0.63± 0.02
VK-DS-2705	sed	1922 ±193	1.44± 0.87	121 ±36	4.29± 0.6	1.4± 0.48	5.45± 0.25	11.97± 1.71	1921± 291	194± 11	4.29± 4.46	0	0.28± 0.01	385± 49	203± 20	1055 ±170	8948± 1995	0.66± 0.12
VK-DS-2009	sed	1712 ±313	2.18± 1.96	127 ±48	4.44± 0.74	1.78± 0.57	5.49± 0.32	12.64± 2.48	1662± 372	206± 16	14.08± 24.28	0	0.33± 0.05	307± 81	179± 11	1163 ±188	9669± 2887	0.65± 0.12
VK-YM-0206	soil	2836 ±563	1.35± 0.26	124 ±14	5.06± 0.6	1.52± 0.1	6.78± 0.66	11± 0.61	3237± 652	181± 11	7.48± 6.56	0	0.12± 0.08	617± 182	211± 46	1281 ±140	8786± 579	0.54± 0.11
VK-YM-1909	soil	3847 ±650	1.33± 0.18	125 ±18	4.71± 0.3	1.9± 0.1	8.94± 0.96	10.47± 0.68	4654± 827	166± 9	3.32± 2.97	0	0.19± 0.2	792± 184	229± 41	1469 ±95	9719± 1205	0.68± 0.05
VK-M2-1909	soil	2207 ±213	1.44± 0.34	126 ±8	4.27± 0.29	1.37± 0.09	6.37± 0.19	10.98± 0.86	2808± 328	169± 13	1.47± 1.39	0	0.44± 0.03	477± 65	190± 24	1140 ±53	9271± 481	0.62± 0.03
VK-OM-0206	soil	2956 ±75	1.28± 0.15	122 ±5	5.22± 0.66	1.73± 0.15	7.61± 1.32	11.05± 0.18	3735± 153	174± 9	6.47± 5.61	0	0.45± 0.35	580± 84	165± 42	1267 ±21	8866± 272	0.56± 0.11
VK-OM-1909	soil	2124 ±254	1.11± 0.32	118 ±9	3.76± 0.2	1.41± 0.48	5.52± 1.08	9.87± 0.31	2907± 239	147± 16	13.86± 2.82	0	0.51± 0.19	456± 33	193± 19	1124 ±128	8366± 91	0.57± 0.01
VK-RM-2709	soil	3076 ±288	2.43± 0.2	95± 83	4.74± 0.31	1.97± 0.47	6.57± 0.37	11.7± 0.83	4116± 387	173± 17	54.65± 11.78	0.04± 0.06	1.29± 0.3	525± 439	182± 139	1488 ±106	9958± 371	0.45± 0.39
<b>Kviarjökull</b>																		
KV-PL-2409	sed	1673 ±214	1.53± 0.23	95± 12	3.22± 0.37	1.87± 0.25	5.39± 0.74	9.44± 0.79	2306± 325	87±1 0	0	0	0.32± 0.03	447± 61	234± 19	1035 ±152	6148± 892	0.43± 0.06
KV-R-2409	soil	2225 ±176	2.31± 0.55	138 ±19	4.25± 0.33	2.41± 0.29	7.97± 0.28	12.26± 1.00	2925± 217	146± 16	3.82± 6.61	0	0.43± 0.16	523± 50	251± 23	1462 ±102	9381± 823	0.62± 0.05
KV-LM-2409	soil	2587 ±46	2.23± 0.25	120 ±2	4.12± 0.1	2.73± 0.28	5.38± 0.17	9.83± 0.64	3557± 133	126± 14	3.64± 2.07	0	0.33± 0.14	634± 48	336± 6	1752 ±59	8117± 311	0.56± 0.01
KV-M2-2409	soil	2336 ±294	2.22± 0.19	111 ±8	4.05± 0.26	2.55± 0.16	6.3± 0.55	10.24± 0.15	3056± 403	126± 1	7.42± 4.95	0	0.33± 0.03	559± 98	266± 29	1618 ±119	7800± 428	0.53± 0.03
KV-M3-2409	soil	2635 ±364	2.38± 0.28	121 ±5	4.22± 0.34	2.62± 0.35	6.94± 0.39	11.28± 0.61	3698± 355	109± 6	18.34± 1.23	0	1.87± 0.55	741± 96	267± 22	1637 ±247	8286± 557	0.59± 0.03
<b>Fjallsjökull</b>																		

FJ-PL-2309	sed	2352 ±227	2.36± 0.48	145 ±13	5.46± 0.25	2.52± 0.23	11.36 ±0.85	14.01± 0.84	2972± 217	199± 23	71.22± 25.33	0	0.35± 0.02	293± 62	111± 16	2104 ±132	12805 ±1484	0.83± 0.01
FJ-R-2309	sed	1596 ±199	2.16± 0.47	113 ±18	4.25± 0.34	2.08± 0.17	7.54± 0.28	11.15± 0.64	1571± 231	177± 8	92.68± 17.72	0	0.31± 0.01	242± 57	102± 17	1359 ±109	9097± 784	0.6± 0.04
FJ-LM-2309	soil	2569 ±324	2.15± 0.21	147 ±3	5.11± 0.2	2.01± 0.05	9.6± 0.53	13.51± 0.73	3097± 260	173± 16	54.94± 37.42	0	0.53± 0.02	439± 70	146± 30	1749 ±54	10891 ±314	0.79± 0.01
FJ-M2-2309	soil	3598 ±160 8	2.71± 0.72	144 ±9	5.18± 0.41	2.3± 0.2	9.99± 0.46	12.89± 1.77	3863± 1151	170± 27	39.02± 21.64	0	1.08± 0.64	890± 562	210± 106	1937 ±484	10836 ±1135	0.75± 0.04
FJ-M3-2309	soil	3392 ±901	2.61± 0.79	138 ±12	5.15± 0.11	2.16± 0.21	9.28± 0.03	13.49± 0.91	3941± 1022	171± 16	34.9± 18.36	0	2.3± 0.81	841± 288	215± 62	1816 ±312	10710 ±570	0.76± 0.01
<b>Skaftafellsjökull</b>																		
SK-PL-2209	sed	4566 ±634	3.98± 0.32	159 ±8	5.44± 0.25	2.99± 0.12	12.91 ±1.18	12.32± 0.29	4821± 624	191± 2	24.32± 7.6	0	0.23± 0.08	976± 148	266± 58	2160 ±88	11346 ±215	0.75± 0.03
SK-R-2209	sed	2738 ±325	4.93± 0.85	134 ±18	5.38± 0.49	3.46± 0.52	11.07 ±0.79	13.23± 1.45	2540± 381	207± 11	34.48± 4.72	0	0.32± 0.02	466± 76	117± 6	1768 ±167	12410 ±1482	0.78± 0.05
SK-TM-2209	soil	3936 ±457	3.68± 0.26	153 ±10	5.44± 0.29	2.77± 0.04	11.69 ±0.23	12.57± 0.38	4187± 545	213± 3	10.21± 4.84	0	0.17± 0.03	856± 156	184± 24	1920 ±93	11325 ±542	0.73± 0.03
SK-M2-2209	soil	3763 ±285	3.19± 0.48	135 ±14	4.93± 0.39	2.85± 0.29	10.92 ±1.06	11.58± 0.43	4156± 405	176± 13	24.55± 0.57	0	0.22± 0.03	832± 74	266± 32	2119 ±167	10521 ±1077	0.74± 0.02
<b>Skeiðarárjökull</b>																		
SD-LM-2609	soil	8575 ±1085	10.97 ±0.95	182 ±17	5.51± 0.12	4.68± 0.24	18.09 ±1.67	9.44± 0.14	7468± 827	89±1	58.47± 3.59	0	0	1930 ±227	301± 35	4539 ±459	12447 ±1418	0.75± 0.01
SD-M2-2609	soil	7654 ±813	9.35± 0.59	178 ±14	6.01± 0.41	4.96± 0.15	17.15 ±1.33	10.91± 0.44	7213± 679	101± 8	61.29± 5.2	0	0.24± 0.04	1773 ±189	288± 30	4154 ±271	12653 ±1168	0.84± 0.06
<b>GREENLAND</b>																		
G-1408	sed	1829 ±565	3.19± 1.07	54± 14	1.8± 0.38	1.72± 0.38	3.57± 0.41	6.13± 1.47	1909± 516	259± 117	55.55± 28.38	0.36± 0.03	0.69± 0.13	59± 13	357± 119	1151 ±323	4329± 990	0.32± 0.07
SS-D-0508	sed	1844 ±463	3.86± 0.48	61± 14	1.94± 0.13	1.91± 0.06	5.59± 3.68	6.88± 0.42	2108± 305	289± 19	14.95± 7.54	0.26± 0.06	0.72± 0.01	65± 24	414± 49	1311 ±105	4475± 368	0.33± 0.03
SS-L-1008	sed	1327 ±293	3.27± 1.09	45± 11	1.55± 0.4	1.58± 0.45	2.39± 0.76	5.92± 1.79	1778± 482	170± 7	28.07± 25.61	0.13± 0.09	0.87± 0.3	69± 15	240± 85	1025 ±290	3339± 634	0.26± 0.05

SS-L-1308	sed	989± 235	2.45± 0.57	30± 4	1.14± 0.16	1.32± 0.26	2.04± 0.5	4.33± 0.58	1220± 178	142± 11	8.01± 6.84	0.12± 0.01	0.63± 0.1	46±7	190± 57	786± 127	2551± 414	0.19± 0.03
ZR-SS-1508	sed	1150 ±273	3.6± 0.19	45± 7	1.66± 0.09	1.8± 0.17	3.4± 0.5	5.75± 0.42	1691± 143	158± 13	20.17± 9.56	0.12± 0.04	0.71± 0.04	68± 16	272± 28	988± 77	3450± 334	0.26± 0.02
ZR-BL-0108	sed	1011 ±146	1.63± 0.49	31± 6	1.04± 0.18	0.9± 0.13	1.48± 0.21	3.28± 0.32	1050± 58	87±2 4	0.89± 1.54	0.16± 0.19	0.52± 0.02	51±1	139± 17	566± 42	4063± 1967	0.28± 0.12
ZR-LM-1608	sed	620± 22	2.46± 0.21	27± 2	1.08± 0.08	1.61± 0.12	2.32± 0.18	12.12± 10.74	956± 274	64±1 4	334.08± 95.18	0.38± 0.14	0.52± 0.03	56± 12	173± 48	450± 64	2220± 174	0.42± 0.29
ZR-AC-1608	sed	970± 56	7.62± 1.39	68± 3	2.89± 0.33	4.17± 0.71	7.04± 0.92	11.27± 1.35	3983± 261	100± 11	232.17± 12.22	1.23± 0.07	1.09± 0.17	115± 14	669± 160	1251 ±268	6793± 1016	0.52± 0.07
ZR-B-0808	sed	1051 ±423	2.39± 0.68	36± 12	1.4± 0.31	1.59± 0.36	3.99± 0.89	4.38± 0.86	1381± 306	81±1 6	87.94± 26.39	0.22± 0.16	0.47± 0.12	104± 33	206± 44	581± 90	2783± 764	0.22± 0.05
ZR-S-0208	sed	1120 ±90	2.48± 0.26	42± 4	1.56± 0.16	1.61± 0.14	3.7± 0.28	5.64± 2.6	1269± 62	138± 32	148.08± 32.35	0.31± 0.03	0.5± 0.04	99± 11	176± 11	557± 25	3391± 469	0.29± 0.08
G-1408	soil	2376 ±71	7.94± 0.46	120 ±4	3.74± 0.15	3.57± 0.19	6.1± 0.33	13.14± 0.52	4477± 270	278± 19	18.25± 6.83	0.11± 0.03	1.11± 0.02	106± 5	640± 35	2849 ±208	8844± 527	0.62± 0.03
AHR-0508	soil	1522 ±161	5.17± 1.42	60± 10	1.92± 0.18	2.13± 0.32	3.1± 0.34	7±0.19	2410± 337	213± 23	94.46± 50.59	0.25± 0.06	0.74± 0.08	82± 14	302± 93	1226 ±105	4249± 402	0.32± 0.03
SS-D-0508	soil	1896 ±182	5.2± 1.03	71± 13	2.21± 0.25	2.35± 0.28	3.82± 0.56	8.57± 0.81	2610± 413	297± 14	46.64± 12.82	0.32± 0.12	0.85± 0.03	75± 20	467± 112	1519 ±181	4893± 652	0.36± 0.05
SS-LI-1408	soil	2282 ±493	8.19± 1.9	90± 32	3.15± 0.83	3.62± 0.77	7.39± 1.91	11.98± 2.83	3851± 983	219± 12	90.86± 23.18	0.29± 0.12	1.3± 0.11	110± 34	655± 251	2316 ±741	6951± 1858	0.51± 0.12
SS-L-1008	soil	1282 ±207	2.75± 0.75	45± 10	1.41± 0.25	1.22± 0.27	2.07± 0.65	4.67± 0.86	1588± 355	160± 7	51.69± 31.99	0.08± 0.03	0.83± 0.1	81± 16	236± 58	845± 171	3181± 590	0.24± 0.04
SS-L-1308	soil	979± 409	3.66± 0.6	36± 13	1.48± 0.36	1.63± 0.19	2.41± 0.07	5.19± 0.85	1679± 278	133± 42	48.42± 13.8	0.09± 0.08	0.66± 0.07	66± 21	174± 74	928± 182	3322± 576	0.25± 0.04
SS-O-1008	soil	1372 ±155	5.95± 0.83	64± 7	2.44± 0.33	2.67± 0.28	5.92± 1.27	8.62± 0.97	2882± 385	165± 12	59.43± 6.8	0.14± 0.05	0.96± 0.02	79±7	496± 119	1643 ±228	5214± 444	0.39± 0.03
ZR-SS-1508	soil	1310 ±181	5.33± 0.47	56± 6	2.16± 0.14	2.51± 0.15	5.03± 0.43	6.78± 0.33	2158± 217	189± 20	52.57± 16.38	0.19± 0.06	0.8± 0.11	80± 13	352± 37	1259 ±86	4459± 372	0.33± 0.03
ZR-BL-0108	soil	1556 ±260	9.73± 0.51	102 ±14	3.06± 0.26	3.81± 0.26	6.07± 0.26	13.65± 1.73	4153± 49	179± 15	136.74± 10.38	0.43± 0.02	0.99± 0.07	109± 14	853± 108	1602 ±72	6561± 174	0.5± 0.02
ZR-LM-1608	soil	1355 ±255	8.89± 1.11	83± 10	3.28± 0.29	4.48± 0.54	10.07 ±1.74	14.24± 0.78	4704± 1470	120± 5	132.53± 32.08	1.17± 0.13	1.31± 0.16	151± 50	713± 112	1468 ±128	8020± 816	0.61± 0.06

ZR-AC-1608	soil	986± 259	4.57± 1.2	50± 1	1.97± 0.35	2.45± 0.7	4.24± 1.25	6.85± 1.57	2407± 648	143± 42	28.51± 5.20	0.64± 0.17	0.79± 0.13	81±8	293± 26	848± 124	4591± 894	0.35± 0.06
ZR-B-0808	soil	1084 ±85	6.91± 0.25	87± 8	3.07± 0.13	3.84± 0.09	7.98± 0.12	15.6± 0.48	3531± 226	120± 13	92.62±3 6.13	1.32± 0.16	1.12± 0.02	137± 12	533± 54	1139 ±39	7051± 459	0.56± 0.02
ZR-S-0208	soil	1115 ±36	7.68± 0.32	79± 4	3.01± 0.04	4.16± 0.05	8.88± 0.03	11.12± 0.6	4093± 131	116± 11	112.76± 20.45	1.15± 0.08	1.12± 0.06	165± 8	578± 30	1258 ±38	6994± 107	0.52± 0.01

Appendix 15. Concentration of BHPs and GDGTs ( $\mu\text{g/g}$  TOC) in soil and sediment samples from the Tarfala valley, Sweden. BDL – below detection limit.

Compounds		m/z	KG-MW-1308	T-L-1308	TL-RB-1408	MS-TL-1408	SG-R-0808	SG-DS-1008	SG-VP-0808	SG-M2-0808
<b>Bacteriohopanepolyols</b>										
BHT	Anhydro-BHT	613	BDL	12	11	7	BDL	BDL	BDL	BDL
	BHT	655	1624	2761	2697	2667	11	BDL	3679	84
	BHT isomer	655-II	16	104	137	133	BDL	BDL	135	BDL
	Me-BHT	669	33	360	812	760	BDL	BDL	714	BDL
	BHPentol	713	17	34	56	53	BDL	BDL	64	BDL
Amino	unsat aminotriol	712	BDL	BDL	6	1	BDL	BDL	BDL	BDL
	35-aminotriol	714	217	218	283	731	2	BDL	913	BDL
	Me-35-aminotriol	728	BDL	17	16	47	BDL	BDL	24	BDL
	35-aminotetrol	772	BDL	8	5	18	BDL	BDL	10	BDL
	Me-35-aminopentol	844	11	BDL	BDL	BDL	6	BDL	BDL	BDL
Sugar	Tetra	943+985	79	97	177	294	BDL	BDL	521	BDL
	Penta	1001+1043	BDL	11	32	37	6	BDL	46	BDL
	Hexa	1059	BDL	BDL	2	BDL	BDL	BDL	BDL	BDL
	Me-Hexa	1073	BDL	BDL	5	BDL	BDL	BDL	BDL	BDL
Amino Sugar	unsat Tetra	1042+1000	BDL	16	33	40	BDL	BDL	52	BDL
	BHT cyclitol ether	1002+1044	217	117	256	546	4	BDL	616	BDL
	BHT glucosamine	1002	189	132	361	274	BDL	BDL	668	BDL
	Me-Tetra	1016+1058	205	42	40	72	BDL	BDL	58	BDL
	Penta	1102+1060	BDL	24	46	69	BDL	BDL	91	BDL
	Me-Penta	1074+1116	BDL	8	8	14	BDL	BDL	7	BDL
	Hexa	1160+1118	BDL	10	20	22	BDL	BDL	16	BDL
Me-Hexa	1174+1132	56	18	11	35	BDL	BDL	13	BDL	
Soil	Adenosylhopane	746+ 788+ 830	219	634	687	807	1	BDL	861	BDL
	2Me-adenosylhopane	760+ 802+ 844	BDL	58	34	73	BDL	BDL	47	BDL
	Adenosylhopane Type 2	761	BDL	27	25	40	BDL	BDL	14	BDL
	2Me-adenosylhopane Type 2	775	BDL	21	12	32	BDL	BDL	3	BDL
	Adenosylhopane Type 3	802	BDL	184	229	498	BDL	BDL	123	BDL
	2Me-adenosylhopane Type 3	816	BDL	39	70	113	BDL	BDL	30	BDL
$\Sigma$ BHPs			2883	4952	6071	7383	30	BDL	8705	84
<b>Glycerol dialkyl glycerol tetraethers</b>										
br-GDGT Ia		1022	BDL	52	69	114	BDL	BDL	90	BDL

br-GDGT IIa	1036	BDL	69	169	202	BDL	BDL	221	BDL
br-GDGT IIIa	1050	BDL	36	266	100	BDL	BDL	155	BDL
br-GDGT Ib	1020	BDL	BDL	1	BDL	BDL	BDL	BDL	BDL
br-GDGT IIb	1034	BDL	BDL	2	BDL	BDL	BDL	BDL	BDL
br-GDGT Ic	1018	BDL	1	6	7	BDL	BDL	6	BDL
br-GDGT IIc	1048	BDL	1	11	BDL	BDL	BDL	6	BDL
br-GDGT IIIc	1046	BDL	BDL	1	BDL	BDL	BDL	BDL	BDL
Σ br-GDGTs		BDL	159	525	423	BDL	BDL	478	BDL
Crenarchaeol (cren)	1292	BDL	5	BDL	21	BDL	BDL	BDL	BDL
Σ GDGTs		BDL	164	525	444	BDL	BDL	478	BDL

Appendix 16. Concentration of BHPs and GDGTs ( $\mu\text{g/g}$  TOC) in soil and sediment samples from Svínafellsjökull, Virkisjökull, Kviarjökull and Fjallsjökull glaciers in Iceland. BDL – below detection limit.

Compounds		m/z	SV-PL-2905	SV-LIAMI-3005	SV-2.5K-3105	VK-YMI-0206	VK-OMI-0206	VK-RMI-2709	KV-M2-2409	KV-M3-2409	FJ-LM-2309	FJ-M2-2309	FJ-M3-2309
<b>Bacteriohopanepolyols</b>													
BHT	Anhydro-BHT	613	BDL	BDL	BDL	BDL	BDL	6	17	19		1	3
	BHT	655	103 3	470	610	2071	1895	1323	1981	2903	618	787	691
	BHT isomer	655-II	57	18	31	103	83	37	75	82	30	21	18
	Me-BHT	669	258	86	180	557	376	492	320	619	149	287	200
	BHPentol	713	20	8	17	35	44	43	51	83	20	24	23
	BHHexol	771	BDL	BDL	BDL	BDL	BDL	23	15	32	10	BDL	BDL
Amino	35-aminotriol	714	200	42	40	306	194	24	90	113	32	19	25
	Me-35-aminotriol	728	8	2	4	BDL	17	BDL	6	BDL	6	BDL	2
	35-aminotetrol	772	5	7	14	BDL	46	47	BDL	27	BDL	12	41
	Me-35-aminotetrol	786	BDL	1	1	BDL	BDL	BDL	BDL	BDL	BDL	BDL	3
Sugar	Unsaturated Tetra	941+ 943	BDL	BDL	BDL	BDL	BDL	54	BDL	55	BDL	12	20
	Tetra	943+ 985	65	4	16	207	86	19	27	86	BDL	15	14
	Penta	1001+ 1043	14	1	3	19	24	7	BDL	19	BDL	BDL	2
	Me-Penta	1015	BDL	BDL	BDL	BDL	BDL	21	BDL	200	BDL	BDL	25
Amino Sugar	Unsaturated Tetra	1042+ 1000	15	1	4	21	23	5	10	13	BDL	BDL	2
	BHT cyclitol ether	1002+ 1044	220	11	24	251	79	10	43	57	15	47	26
	BHT glucosamine	1002	123	15	28	332	164	15	94	91	28	33	31
	Me-Tetra	1016+ 1058	34	3	8	66	27	6	20	23	8	8	10
	Penta	1102+ 1060	39	3	9	45	37	4	12	15	5	4	4
	Me-Penta	1074+ 1116	15	4	1	16	7	BDL	5	8	5	BDL	BDL

	Hexa	1160+ 1118	19	2	4	18	14	5	BDL	8	1	4	3
	Me-Hexa	1174+ 1132	10	1	3	21	6	2	BDL	10	BDL	3	3
Soil	Adenosylhopane	746+ 788+ 830	279	183	181	325	317	120	241	202	65	121	54
	2Me-adenosylhopane	760+ 802+ 844	21	14	23	16	33	20	26	24	9	17	10
	Adenosylhopane Type 2	761	7	5	14	8	14	2	7	7	BDL	3	1%
	2Me-adenosylhopane Type 2	775	2	2	10	BDL	8	4	4	9	BDL	BDL	BDL
	Adenosylhopane Type 3	802	76	57	126	35	276	46	109	78	41	40	23
	2Me-adenosylhopane Type 3	816	12	4	18	7	26	7	15	13	11	8	5
Σ BHPs			253 2	944	1369	4459	3796	2342	3168	4796	1053	146 6	123 8
<b><i>Glycerol dialkyl glycerol tetraethers</i></b>													
	br-GDGT Ia	1022	22	27	53	21	62	9	36	17	20	13	15
	br-GDGT IIa	1036	50	65	108	62	119	18	71	32	48	18	26
	br-GDGT IIIa	1050	19	23	44	30	74	7	26	10	21	5	9
	br-GDGT Ib	1020	BDL	12	8	BDL	BDL						
	br-GDGT IIb	1034	3	5	12	BDL	BDL						
	br-GDGT IIIb	1048	BDL	0	BDL	BDL							
	br-GDGT Ic	1018	BDL	2	4	BDL	3	0	BDL	BDL	BDL	BDL	BDL
	br-GDGT IIc	1048	BDL	5	1	BDL	BDL						
	br-GDGT IIIc	1046	BDL	0	BDL	BDL							
	Σ br-GDGTs		95	139	229	113	258	35	133	59	89	36	51
	Crenarchaeol (cren)	1292	BDL	1	BDL	BDL							
	Σ GDGTs		95	140	229	113	258	35	133	59	89	36	51

Appendix 17. Concentration of BHPs and GDGTs ( $\mu\text{g/g}$  TOC) in soil and sediment samples from Zackenberg valley, Greenland. BDL – below detection limit.

Compounds		m/z	AHR-0508	G-1408	SS-D-0508	SS-L1-1408	SS-L-1008	SS-L-1308	SS-O-1008	ZR-SS-1508	ZR-BL-0108	ZR-LM-1608	ZR-AC-1608	ZR-B-0808	ZR-S-0208
<b><i>Bacteriohopanepolyols</i></b>															
BHT	Anhydro-BHT	613	BDL	BDL	10	23	13	21	12	17	10	5	8	BDL	8
	BHT	655	3788	1192	2592	3212	3939	3287	5304	4034	1726	2169	1821	2826	2240
	BHT isomer	655-II	131	68	107	121	188	114	244	125	123	157	105	282	81
	Me-BHT	669	845	274	662	1108	799	606	1317	533	365	453	172	638	464
	BHPentol	713	138	BDL	BDL	111	93	93	108	86	42	59	87	55	46
	Me-BHPentol	727	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	23	BDL	BDL	BDL
	BHHexol	771	BDL	BDL	BDL	BDL	46	81	BDL	47	43	25	BDL	BDL	BDL
Amino	Unsaturated aminotriol	712	BDL	BDL	BDL	BDL	BDL	BDL	BDL	47	BDL	BDL	BDL	BDL	BDL
	35-aminotriol	714	187	231	215	124	331	210	391	457	47	87	274	135	107
	Me-35-aminotriol	728	BDL	18	17	4	24	21	8	28		7	11	BDL	BDL
	35-aminotetrol	772	132	35	72	61	13	99	114	37	76	117	13	125	25
	Me-35-aminotetrol	786	40	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	75	BDL
Sugar	Unsaturated Tetra	941+943	BDL	5	BDL	41	74	74	129	36	BDL	BDL	BDL	BDL	BDL
	Tetra	943+985	46	21	102	154	82	74	130	55	41	27	14	33	84
	Penta	1001+1043	13	BDL	15	6	16	BDL	BDL	30	5	9	18	BDL	20
	Me-Penta	1015	50	BDL	44	54	94	168	404	80	BDL	BDL	BDL	8	26
	Hexa	1059	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	5
AminoSugar	unsat Tetra	1042+1000	BDL	5	31	15	15	18		31	5	9	22	13	23
	BHT cyclitol ether	1002+1044	31	28	62	47	73	50	119	121	26	28	59	48	61
	BHT glucosamine	1002	74	53	75	65	105	49	179	222	29	106	153	119	115
	Me-Tetra	1016+1058	19	17	19	28	21	16	36	50	12	28	32	18	27

	Penta	1102+1060	19	11	28	15	34	27	55	41	BDL	15	45	24	31
	Me-Penta	1074+1116	BDL	BDL	BDL	6	BDL								
	Hexa	1160+1118	4	BDL	12	11	6	14	17	9	5	BDL	5	4	3
	Me-Hexa	1174+1132	3	1	9	15	6	11	10	7	14	BDL	BDL	BDL	BDL
Soil	Adenosylhopane	746+788+830	364	274	485	261	426	301	309	296	737	153	170	525	317
	2Me-adenosylhopane	760+802+844	39	43	18	27	76	36	21	21	94	32	40	75	19
	Adenosylhopane Type 2	761	12	11	11	11	15	13	8	18	29	9	10	15	14
	2Me-adenosylhopane Type 2	775	8	10	5	4	20	6	2	7	15	4	16	7	2
	Adenosylhopane Type 3	802	217	173	277	294	377	299	485	172	237	130	70	266	222
	2Me-adenosylhopane Type 3	816	16	29	41	26	63	32	26	23	17	20	12	26	54
Σ BHPs			6176	2499	4909	5844	6949	5720	9428	6630	3721	3649	3157	5317	3994
<b><i>Glycerol dialkyl glycerol tetraethers</i></b>															
br-GDGT Ia		1022	58	21	39	14	96	61	68	55	8	46	22	28	25
br-GDGT IIa		1036	75	42	70	26	173	65	92	80	26	104	27	79	58
br-GDGT IIIa		1050	17	14	18	10	45	14	28	15	15	30	6	31	20
br-GDGT Ib		1020	BDL	1	3	BDL	6	BDL							
br-GDGT IIb		1034	BDL	1	4	BDL	BDL	BDL							
br-GDGT Ic		1018	2	BDL	2	1	6	3	4	BDL	1	1	BDL	2	1
br-GDGT IIc		1048	BDL	BDL	BDL	1	4	1	2	BDL	0	BDL	BDL	1	1
br-GDGT IIIc		1046	BDL	BDL	BDL	0	BDL	BDL	BDL	BDL	3	BDL	BDL	2	1
Σ br-GDGTs			153	77	128	53	325	143	193	150	56	189	54	149	105
Crenarchaeol (cren)		1292	BDL	12	BDL	BDL	BDL	BDL	BDL	BDL	33	BDL	BDL	49	BDL
Σ GDGTs			153	89	128	53	325	143	193	150	90	189	54	199	105

Appendix 18. DNA concentration measured using QuBit 2.0 (ng/ml) of soil and sediment samples collected in Sweden, Iceland and Greenland. BDL – below detection limit.

Sample	Type	DNA concentration (QuBit 2.0), ng/ml
<b>SWEDEN</b>		
<b>Tarfalajaure transect</b>		
KG-MW-1308_2	sediment	BDL
T-L-1308_1	soil	7.45
T-L-1308_2	soil	4.09
T-L-1308_3	soil	7.7
TL-RB-1408_1	soil	30.4
TL-RB-1408_2	soil	18.7
TL-RB-1408_3	soil	27.5
MS-TL-1408_1	soil	2.72
MS-TL-1408_2	soil	28.8
MS-TL-1408_3	soil	5.49
<b>Storglaciären</b>		
SG-DS-1008_3	sediment	BDL
SG-VP-0808_1	soil	8.97
SG-VP-0808_2	soil	7.72
<b>ICELAND</b>		
<b>Svínafellsjökull</b>		
SV-PL-2905_1	soil	9.16
SV-PL-2905_3	soil	15.8
SV-LM-2109_1	soil	19.2
SV-LM-2109_2	soil	19.4
SV-R-2109_1	soil	9.18
SV-R-2109_3	soil	5.99
SV-LIAM-3005_1	soil	42.8
SV-LIAM-3005_2	soil	59.2
SV-LIAM-3005_3	soil	27.9
SV-LIAM-2109_1	soil	2.12
SV-LIAM-2109_3	soil	1.06
SV-2.5K-3105_2	soil	25.2
SV-2.5K-3105_3	soil	17.7
SV-2.5K-3105_4	soil	36.6
SV-2.5K-2109_2	soil	27.8
SV-2.5K-2109_4	soil	30.8
<b>Virkisjökull</b>		
VK-YM-0206_1	soil	1.07
VK-YM-0206_2	soil	3.6
VK-YM-0206_3	soil	4.69
VK-YM-1909_2	soil	1.2
VK-M2-1909_2	soil	5.38
VK-M2-1909_3	soil	3.05

VK-OM-0206_1	soil	11.1
VK-OM-0206_2	soil	11.6
VK-OM-0206_3	soil	4.41
VK-OM-1909_2	soil	18.6
VK-RM-2709_2	soil	39.5
VK-RM-2709_3	soil	17.4
<b>Kviarjökull</b>		
KV-R-2409_1	soil	12.3
KV-R-2409_3	soil	6.09
KV-LM-2409_1	soil	8.84
KV-LM-2409_2	soil	3.18
KV-M2-2409_1	soil	6.88
KV-M2-2409_2	soil	12.3
KV-M3-2409_1	soil	4.82
KV-M3-2409_2	soil	10.7
<b>Fjallsjökull</b>		
FJ-LM-2309_1	soil	6.25
FJ-LM-2309_2	soil	11.4
FJ-M2-2309_1	soil	28.8
FJ-M3-2309_1	soil	18.7
FJ-M3-2309_2	soil	10.4
<b>Skaftafellsjökull</b>		
SK-TM-2209_2	soil	BDL
SK-M2-2209_2	soil	13.9
SK-M2-2209_3	soil	14.1
<b>Skeiðarárjökull</b>		
SD-LM-2609_1	soil	2.36
SD-LM-2609_2	soil	1.06
SD-M2-2609_1	soil	31.2
SD-M2-2609_2	soil	13.8
<b>GREENLAND</b>		
G-1408_3	sediment	BDL
SS-D-0508_1	sediment	2.95
SS-D-0508_2	sediment	0.96
SS-L-1008_2	sediment	32.5
SS-L-1308_3	sediment	11.9
ZR-SS-1508_2	sediment	8.52
ZR-BL-0108_1	sediment	BDL
ZR-LM-1608_1	sediment	BDL
ZR-AC-1608_1	sediment	BDL
ZR-B-0808_2	sediment	BDL
ZR-S-0208_3	sediment	BDL
SS-O-1008_1	soil	25.5
SS-O-1008_3	soil	24.8

AHR-0508_3	soil	30.7
G-1408_1	soil	13.8
G-1408_2	soil	14.3
G-1408_3	soil	29.9
SS-D-0508_1	soil	37.1
SS-D-0508_2	soil	19.2
SS-LI-1408_1	soil	30.5
SS-LI-1408_2	soil	34.6
SS-LI-1408_3	soil	51.2
SS-L-1008_3	soil	61.7
SS-L-1308_1	soil	33.8
ZR-SS-1508_2	soil	9.93
ZR-BL-0108_1	soil	46.4
ZR-BL-0108_3	soil	42
ZR-LM-1608_2	soil	31.4
ZR-LM-1608_3	soil	40.6
ZR-AC-1608_1	soil	16
ZR-AC-1608_3	soil	15.2
ZR-B-0808_1	soil	33.3
ZR-B-0808_2	soil	34.6
ZR-S-0208_1	soil	18.9
ZR-S-0208_2	soil	11.1