Soil Organic Matter Composition and Transformation in a Swamp Soil from Algae of Coastal Continental Antarctica

By Lothar Beyer¹, Birgit Vogt¹, Hans-Peter Blume1¹ and Helmut Erlenkeuser²

Summary: Soil organic matter (SOM) of a swamp soil (US: Lithic Cryofibrist, FAO: Fibri-gelic Histosol) from algae in Antarctica was studied with special emphasis on soil formation processes under extreme climate conditions. An integrated approach of modern analytical methods including wet-chemical analyses and cross polarization magic angle spinning carbon-13 nuclear magnetic resonance spectroscopy (CPMAS ¹³C-NMR) was applied to characterize the SOM composition of a Euic, Shallow Lithic Cryofibrist. The soil morphology suggested a slight humification degree. The wet-chemical litter compound analysis (protein, lipids, polysaccharides, lignin) and CPMAS 13C-NMR confirmed these observation, whereas the classical SOM alkaline extraction (fulvic acid, humic acid, humin) was not suitable to characterize the weak humified organic matter. The data suggest a predominance of proteins within the nitrogen compounds and a slight degradation of carbohydrates as well a selective preservation of alkyl units within the carbon moieties in the deeper soil layers. Despite a time range of nearly 2000 years between the age of youngest and the oldest soil layer only weak transformation processes of SOM were detectable because soil temperatures are low in summer and obviously not high enough to stimulate a further microbial breakdown.

Zusammenfassung: Die organische Bodensubstanz (OBS) eines Moorbodens (US: Lithic Cryofibrist, FAO: Fibri-gelic Histosol) aus Algen in der Antarktis wurde zur Erfassung der Bodenbildungsprozesse unter extremen Klimabedingungen untersucht. Ein integrierter Ansatz mit der Kombination naßchemischer Analysen und "Cross Polarization Magic Angle Spinning Kohlenstoff-13 Kernresonanz-Spektroskopie" (CPMAS ¹³C-NMR) wurde angewendet, um die OBS eines Euic, Shallow Lithic Cryofibrist zu charakterisieren. Die Bodenmorphe zeigte eine schwache Humifizierung. Die naßchemische Streustoffanalyse (Protein, Lipide, Polysaccharide, Lignin) und die CPMAS ¹³C-NMR bestätigten diese Beobachtung. Die klassische, alkalische OBS-Extraktion (Fulvosäure, Huminsäure, Humine) war dagegen nicht zur Charakterisierung des schwach humifizierten Materials geeignet. Die Ergebnisse zeigen die Dominanz der Proteine bei den Stickstoffverbindungen. Darüber hinaus wurde ein schwacher Abbau der Polysaccharide sowie eine selektive Anreicherung von Alkyl-Kohlenstoffverbindungen mit zunehmender Bodentiefe beobachtet. Trotz einer Zeitspanne von nahezu 2000 Jahren zwischen dem Alter der Bodenhorizonte konnte nur ein geringer Umbau der OBS nachgewiesen werden, weil die Temperaturen im Sommer niedrig und offensichtlich nicht hoch genug sind, den mikrobiellen Abbau zu intensivieren.

INTRODUCTION

In the Maritime Antarctic climate region peat soils, mainly formed by debris of mosses and algae (SMITH 1979, 1986, 1990) are wide-spread (CAMPBELL & CLARIDGE 1987), whereas the evidence of organic matter accumulation in the coastal continental Antarctic climate region has been rejected (BOCKHEIM &

Institute of Plant Nutrition and Soil Science, University of Kiel, Olshausenstraße 40, D-24098 Kiel, Germany

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UGOLINI 1990). However, according to Blume et al. (1997) in Wilkes Land, near Casey Station, the mineral soils are associated with patches of Gelic Histosols (US: Cryohemist or Cryofibrist) in small hollows and at hill feet. But up to now the knowledge of peat soils of Coastal Continental Antarctica is still rudimentary. In a previous paper (BEYER et al. 1997a) we discussed the formation and humification processes of a Pergelic Cryohemist (FAO: Terri-gelic Histosol) from mosses in the Casey area as revealed by a combination of classical SOM analyses (Schnitzer & Kahn 1978) and modern instrumental techniques (Schnitzer 1991). The second important organic matter precursors in this landscape are different kind of algae (SMITH 1986, 1990 and references cited in there), which accumulate in small wet relief hollows or glacial water lake sites (Blume & BÖLTER 1993). Therefore in the present paper the SOM composition and formation of a Euic, Shallow Lithic Cryofibrist from algae is presented. An integrated approach using wet-chemical analyses and CPMAS ¹³C-NMR spectroscopy is used in order to improve the understanding of peat formation under the extremely wet and cold conditions in a moraine landscape near a glacier (HEAL & BLOCK 1987).

MATERIALS AND METHODS

Site and soil

The sampling site is located south of the Australian Casey Station, Wilkes Land (66°18'S, 110°32'E) at the coast of East Antarctica (Fig.1). The mean annual temperature is -9.3 °C. Only in January the mean temperature is above the freezingpoint (+0.2 °C). From November to February the sun shines for 5-7 h d⁻¹. The annual precipitation (176 mm) is mostly snow. The Lithic Cryofibrist is located at the end of a slope near a meltwater lake between large moraine rocks. The whole soil consists of dead algae (*Phormidium* sp.). The organic material is uncomposed or weakly decomposed (Fig.2: Hl) and has a greenish-gray color (Tab.1). For most of the year this soil is covered with water or ice. Only in warm summers the first 10-20 cm are above the groundwater table. Under such conditions the redox potentials is low in the H2 (Oe1) and H3 (Oe2) layers (Fig.2: Eh). The pH-value (Tab.1) is similar to those swamps observed generally under temperate climate conditions (WA-CHENDORF et al. 1996). According to the recent Keys to Soil Taxonomy (Soil Survey Staff 1996) the soil is classified as

Institut für Reine und Angewandte Kernphysik, University of Kiel, Olshausenstraße 40, D-24118 Kiel, Germany



Fig. 1: Location of the investigated swamp soil from algae in coastal area of continental Antarctica (Wilkes Land, Casey Station).

Abb. 1: Lage des untersuchten Moorbodens aus Algen in der Küstenregion der kontinentalen Antarktis (Wilkes Land, Casey Station).

hori	zon	depth	Munsell	moisture ^c	pH	gravels
FAOª	US ^b	cm	color		CaCl,	g kg ⁻¹
H1 H2 H3 R	Oi Oe1 Oe2 R	0-1 -8 -18	5GY6/1 5GY5/1 5GY5/1	very wet very wet very wet rock and mor	4.8 4.8 4.8	0 3 24

Tab. 1: Field description of the investigated swamp soil from algae in coastal continental Antarctica.

Tab. 1: Feldbeschreibung des untersuchten Moorbodens aus Algen in der Küstenregion der kontinentalen Antarktis.

"euic", because of the pH level higher than pH 4.5. In addition, because of the shallow solum of 18 cm the Cryofibrist has to be classified on subgroup level as "lithic" and not as "pergelic". Samples were taken during the 1991 Antarctic summer.

Field measurements

According to the recent Keys to Soil Taxonomy (Soil Survey Staff 1996) the swamp soil is an Euic, Shallow, Lithic Cryofibrist or a Fibri-gelic Histosol according to the recent FAO system (FAO 1989). The humification level was estimated according to Post by squeezing the moist soil sample between the fingers (Schlichting et al. 1995): degree 1 indicates no humification and degree 10 complete humification. Redox potentials (Eh) were measured with platinum electrodes (Pfisterer & Gribbohm 1989) after 6 to 12 h installation in the field (Schlichting et al. 1995). The pH values were determined in a 1:2.5 extract using 10 mM CaCl, (Schlichting et al. 1995).

Wet chemical analysis

The wet chemical analysis and measurements were carried out according to Beyer et al. (1993, 1996a) and Schlichting et al. (1995). Air-dried soil samples were milled to a grain size of <200 μm. Total organic carbon (TOC) was measured by dry combustion in a Coulomat 702 (Ströhlein Instruments). Carbonate-C was not detectable. Dry soil samples were heated (600 °C) in an induction furnace under oxygen; CO₂ was trapped in Ba(OH)₂. Soil organic matter was estimated by loss on ignition at 600 °C in a common furnace (Heraeus Instruments). Methoxyl carbon (OCH₃-C) was determined according to the Zeisl-Pregl method. The OCH₃-C in soils was demethylized into me-

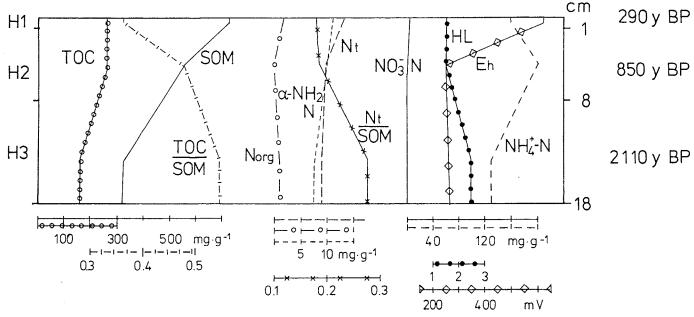


Fig. 2: Properties and age of the investigated swamp soil from algae in coastal continental Antarctica (H1=Oi, H2=Oe1, H3=Oe2, TOC: total organic carbon, SOM: soil organic matter, N_i : total nitrogen, N_{oo} : organic non- α -NH,-N, HL: humification level, Eh: redox potential).

Abb. 2: Eigenschaften und Alter des untersuchten Moorbodens aus Algen in der Küstenregion der kontinentalen Antarktis (TOC: Organischer Kohlenstoff, SOM: Organische Bodensubstanz, N; Gesamtstickstoff, N_{ere}: organischer Nicht-α-NH₂-N, HL: Humifizierungsgrad, Eh: Redoxpotential).

^a FAO (1994); ^b SOIL SURVEY STAFF (1996); ^c in the field during sampling according to SCHLICHTING et al. (1995)

thyl iodide by boiling each sample with hydroiodic acid. Methyl iodide was hydrolized into methanol and silver iodide, which was determined gravimetically. Lignin was estimated as 7 x OCH, content. The extraction of total nitrogen (N) was carried out according to Kjeldahl. The α-NH₂-N was determined after extraction with 10 M HCl and 1 M formic acid (10:1) by using the ninhydrin reaction (triketohydrine hydrate). Proteins were estimated by multiplying α -NH₃-N by 6.25. Lipids, resins and waxes were extracted with ethanol/benzene (1:1). Polysaccharides were extracted in inorganic acids successively. The lipid-free sediment was treated with 0.05 N H₂SO₄ in order to extract the sugar and starch fraction. The remaining sediment was treated with 0.63 N HCl in order to extract hemicellulose. After this treatment cellulose was extracted by using 27 N H₂SO₄. The summarized protein, lipid, polysaccharide and lignin fractions were defined as litter compounds, whereas the remainder to 100 % was defined as non-litter compounds (Fig.3). The humic fractions were extracted according to the classical NaOH procedure. The summarized fulvic acid, humic acid and humin fractions were defined as humic compounds, whereas the remainder to 100 % was defined as non-humic compounds (Fig.4). For further details of the extraction scheme see BEYER et al. (1993, 1996a). Nitrate-N (NO₃-N) and Ammonium-N (NH, +-N) were extracted with 20 mM CaCl₂. Concentrations of the inorganic N fractions in the extracts were determined by flow injection analysis using a Tecator flow injection analyzer (Perstorp Analytical). The organic, non-α-NH, nitrogen (N_{or}) was calculated as N_1 - (α -NH₂-N + inorganic N fractions).

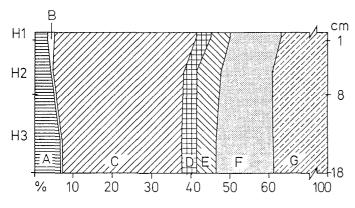


Fig. 3: Litter compound distribution in the SOM (in % of TOC, A: lipids, B: sugar & starch, C: hemicellulose, D: cellulose, E: lignin, F: protein, G: non-litter compounds).

Abb. 3: Streustoffverteilung in der organischen Bodensubstanz (OBS in % vom Gesamtkohlenstoff (C_{org}), A: Lipide, B: Zucker & Stärke, C: Hemizellulose, D: Zellulose, E: Lignin, F: Protein, G: Nicht-Streustoffe).

Carbon-14 measurements

¹⁴C dating was performed on the total organic fraction of the soil samples. Organic carbon was converted to CO₂ by a wet oxidation technique using dichromate/conc. sulfuric acid. The sample gas was purified in different steps by washing and fractionated in destillation processes at cryogenic temperatures from possible contaminants. The gas analyzed for ¹⁴C by β decay counting in the low-level ¹⁴C dating equipments of the lab. ∂¹³C was

determined by stable isotope mass-spectrometry on aliquotes of the sample CO_2 and refers to the international PDB-scale. The ^{14}C -ages were calculated applying the conventional halflife of 5568 a and have been normalized to $\partial^{13}C = -25$ mg g⁻¹ PDB in order to correct for isotope fractionation. The so-called modern reference ^{14}C activity has been determined by means of the international "SRM 4990C Oxalic Acid C-14 Standard". The statistical error (1 sigma = 1s) results from counting statistics and indicates the (unknown) true result of the measurement to lay in the 1s region with a probability of 68 %: H1: age ± 35 , H2: age ± 40 , H3: age ± 50 .

Cross polarization magic angle spinning carbon-13 nuclear magnetic resonance spectroscopy (CPMAS ¹³C-NMR)

The CPMAS ¹³C-NMR spectra were obtained at 2.3 Tesla (25.2 MHz) in a Bruker MSL 100 spectrometer equipped with a commercial 7 mm CPMAS probe at a rotation frequency of 4 kHz. A contact time of 1 ms was used. Due to short T_{IH} in SOM (FRÜND & LÜDEMANN 1989) a recycle delay of 0.3 s was chosen. The ¹³C chemical shifts are reported relative to tetramethylsilane (TMS = 0 ppm). The spectra were subdivided into the following ranges for carbon species in SOM and integrated: 0-46 ppm (alkyl-C), 46-60 ppm (methoxyl-C), 60-80 ppm (hydroxyl-C), 80-110 ppm (acetal-C), 110-140 ppm (aromat-C), 140-160 ppm (phenol-C) and 160-210 ppm (carboxyl-C) (Wilson 1987). It has been shown that under the experimental conditions used the relative signal area can be quantitatively related to the C concentration (FRÜND & LÜDEMANN 1989).

RESULTS AND INTERPRETATION

Selected soil properties and the soil age are shown in Fig. 2. Total organic carbon (TOC) concentrations and losses on ignition as a criterion for SOM content decreased with increasing profile depth. However, TOC was increasingly enriched in the organic matter of deeper horizons (TOC/SOM). The N_ι concentrations decreased in deeper soil horizons. The nitrogen was mainly determined as α-NH₂-N. Other N compounds were of minor importance. The decreasing N_ι/SOM ratio indicate as well as the TOC/SOM ratio a modification in the chemical SOM status (SCHNITZER & KHAN 1978). The high NH₄+ concentrations in comparison to nitrate-N corresponded to low redox potentials, which restricted the nitrification process (BARTLETT & JAMES 1994). The age of the three soil horizons range from 290 years (H1/Oi layer) to 2110 years (H3/Oe2 layer).

Figure 3 confirms the field observation, which indicated little humification in the soil profile (Fig. 2: Hl). However, the slight decrease of polysaccharides (PS), especially hemicellulose, and the increase of lipids suggest initial organic matter decomposition by PS degradation and a relative lipid enrichment (BEYER 1995). On the other hand the occurrence of humic compounds suggests high amounts of humified matter (Fig. 4). This is in contrast to the low humification level in all soil layers (Fig. 2)

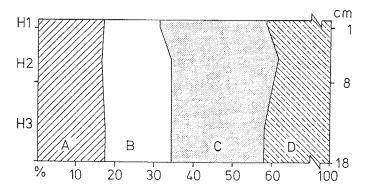


Fig. 4: Humic compound distribution in the SOM (in % of TOC, A: fulvic acids, B: humic acids, C: humins, D: non-humic compounds).

Abb. 4: Huminstoffverteilung in der OBS (in % von C_{org} , A: Fulvosäuren, B: Huminsäuren, C: Humine, D: Nicht-Huminstoffe).

and confirms the assumption of BEYER et al. (1996b, 1997b) that the classical SOM alkaline extraction is not appropriate to characterize weakly-humified matter. Therefore in the present study additionally the non-destructive ¹³C NMR approach was used.

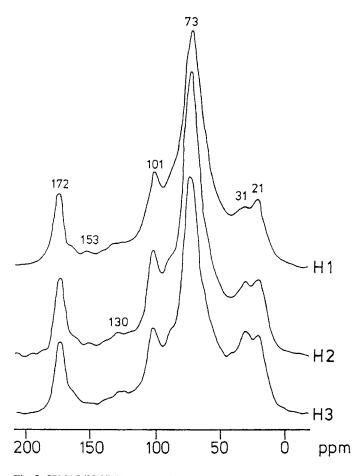


Fig. 5: CPMAS ¹³C-NMR spectra of horizons of the swamp soil from algae in coastal continental Antarctica.

Abb. 5: CPMAS ¹³C-NMR Spektren von Horizonten des Moorbodens aus Algen in den Küstenregionen der kontinentalen Antarktis.

In Figure 5 the ¹³C-NMR spectra of the three soil horizons from the Lithic Cryofibrist are compared. The peak at 21 ppm derives from the methyl group of carbohydrates and the peak at 31 ppm is typical for alkyl-CH, units, whereas 73 ppm and probably 101 ppm derive from polysaccharides (Breitmeier & Voelter 1990). In addition the carboxyl peak at 172 ppm was considerable. However, the profile showed no significant depth function within the soil layers. Only the quantitative NMR estimation suggests a slight chemical modification in the deeper soil layers (Fig. 6). The NMR data confirmed the slight decrease of polysaccharides and increase of lipids observed with the wet chemical litter analysis (Fig. 3) with a similar behaviour of acetal and alkyl carbon moieties (Fig. 6). In contrast to well-known NMR data of peat soils in temperate climate regions (e.g. Kros-SHAVN et al. 1991, WACHENDORF et al. 1996, 1997) in the investigated Lithic Cryofibrist aromat and phenol carbon units are of minor inportance. The pattern of the NMR spectra looks very similar to those documented for organic litter material (BEYER et al. 1996b). This confirms the very slight humification degree in the Antarctic swamp.

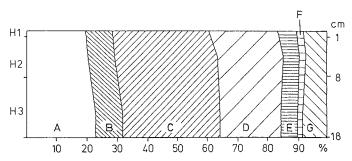


Fig. 6: Estimated quantitative SOM composition according to the integrated data of CPMAS ¹³C-NMR spectroscopy (A: alkyl-C, B: methoxyl-C, C: hydroxyl-C, D: acetal-C, E: aromat-C, F: phenol-C, G: carboxyl-C).

Abb. 6: Abgeschätzte quantitative OBS Zusammensetzung nach integrierten Daten der CPMAS ¹³C-NMR Spektroskopie (A: Alkyl-C, B: Methoxyl-C, C: Hydroxyl-C, D: Acetal-C, E: Aromat-C, F: Phenol-C, G: Corboxyl-C).

DISCUSSION

In the Euic, Shallow Lithic Cryofibrist from algae the greenish-gray soil color in all horizons and the slight humification degree (Blume et al. 1996), which was estimated in the field, was in correspondence to the results of the wet-chemical litter analysis and NMR spectroscopy. Both methods indicated very weak transformation processes in this soil, whereas the classical SOM alkaline extraction was not suitable to characterize the little-humified matter (Beyer et al. 1996b). The data suggests a slight degradation of cabohydrates and the selective preservation of alkyl moieties in the deeper soil layers. Obviously a time span from about 2000 years is not enough for an intensive chemical modification of the parent algal material (SMITH1990). The organic N was mostly bound in amino compounds.

These results suggest, that proteins are of major importance, which are easily available as food resource for the microorganisms under temperate climate circumstances (WACHENDORF et

al. 1996), but not in Antarctica. Obviously the carboxyl carbon derived from fatty acids and proteins (Breitmaier & Voelter 1989, Wilson 1990), because a significant humification was not visible. The alkyl units probably derived from ether-linked nalkyl chains (Gelin et al. 1993). These highly non-hydrolyzable biomacromolecules are located in the outer cell wall of several species of algae (e.g. Largeau et al. 1990) and have a high preservation potential in soils and sediments (Gelin et al. 1993). However, the very slight increase of alkyl carbon units in the Lithic Cryofibrist does not reflect the considerable selective preservation as described by Gelin et al. (1993), Largeau et al. (1990) and NIP et al. (1986). Due to the proximity of a meltwater lake, the whole soil profile is very moist and consequently the thermal capacity of this swamp soil is high. The solar energy input into Antarctica is not sufficient to increase soil temperature significantly in such wet soils (BEYER et al. 1998). Therefore, transformation processes of organic matter are not detectable (Blume et al. 1997). In contrast, in water saturated soils under temperate climate conditions the initial SOM can be slowly transformed, because soil temperatures in summer are high enough to stimulate microbial decomposition processes (Vis-HNIAC 1993).

CONCLUSION

A wet Antarctic swamp soil from algae is characterized by the predominance of proteins within the nitrogen compounds and a slight degradation of carbohydrates in the SOM. A selective preservation of alkyl moieties in the deeper soil layers is suggested. During nearly 2000 years little transformation processes of SOM were detectable because soil temperatures in summer are low and obviously were not high enough to stimulate a further microbial break-down.

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