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Abstract: Some previous studies showed that the formation of several deep dark humus rich topsoils in Northern Europe was strongly influenced by the application of different organic materials by anthropogenic activities in former times. Such topsoils classified as plaggic Anthrosols also occurred in the Jæren region in SW Norway. However, source material and formation time of these Plaggic Anthrosols have not yet been clarified. Close to this region we found further humus-rich topsoils in the Karmøy municipality (2 sites at main island of Karmøy and 1 site at Feøy). These soils show a thick humus-rich topsoil up to 30 cm, and their formation cannot only be explained by natural conditions. We analyzed the molecular signature of the soil organic matter (SOM) by benzene polycarboxylic acids (BPCA), non-targeted bulk SOM mass spectrometry, □34S and 14C AMS dating in order to determine source materials and the age of the SOM. The black carbon (BC) contents of the plaggic soils in Jæren (mean 3.4 g kg-1) deliver clear evidence for inputs of combustion residues from ancient fire management and/or from settlements. The C XANES and Py-FIMS-spectra reveal relative enrichments of aromatic C and heterocyclic N compounds in the plaggic soils corresponding to the BC contents. In contrast, the humus-rich topsoils in Karmøy seem to be unaffected by fire management due to the low BC contents (mean 0.6 g kg-1) and the relative low portions of aromatic C and heterocyclic N compounds from C-XANES and Py-FIMS. The □34S isotope signature of the SOM ranged from 10.6 to 15.2 ‰ in the soils at the islands and 10.0 to 13.5 ‰ in Jæren, corresponding to the Anthrosols in the Baltic Sea region (Median: □34S = 11.5 ‰) and suggest an input of marine biomass (□34S of seaweed = 20 ‰). The AMS 14C dating and complementary archaeological literature implied that the soils in Jæren and Karmøy have been formed between the Roman Iron Age (500 BC to AD 500) and the Viking Age (AD 800 to AD 1,000). Our results provide strong evidence for an anthropo-pedogenesis of the humus-rich topsoils in Karmøy and indicate parallels to the plaggic soils in Jæren as well as to Anthrosols in the Baltic Sea region. Therefore, we propose to classify the humus-rich topsoils in Karmøy as Anthrosols.

Humus-rich topsoils in SW Norway – Molecular and isotopic signatures of soil organic matter as indicators for anthropo-pedogenesis

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Abstract

Some previous studies showed that the formation of several deep dark humus-rich topsoils in Northern Europe was strongly influenced by the application of different organic materials by anthropogenic activities in former times. Such topsoils classified as plaggic Anthrosols also occurred in the Jæren region in SW Norway. However, source material and formation time of these Plaggic Anthrosols have not yet been clarified. Close to this region we found further humus-rich topsoils in the Karmøy municipality (2 sites at main island of Karmøy and 1 site at Feøy). These soils show a thick humus-rich topsoil up to 30 cm, and their formation cannot only be explained by natural conditions. We analyzed the molecular signature of the soil organic matter (SOM) by benzene polycarboxylic acids (BPCA), non-targeted bulk SOM mass spectrometry, $\delta^{34}S$ and ^{14}C AMS dating in order to determine source materials and the age of the SOM. The black carbon (BC) contents of the plaggic soils in Jæren (mean 3.4 g kg^{-1}) deliver clear evidence for inputs of combustion residues from ancient fire management and/or from settlements. The C-XANES and Py-FIMS-spectra reveal relative enrichments of aromatic C and heterocyclic N compounds in the plaggic soils corresponding to the BC contents. In contrast, the humus-rich topsoils in Karmøy seem to be unaffected by fire management due to the low BC contents (mean 0.6 g kg^{-1}) and the relative low portions of aromatic C and heterocyclic N compounds from C-XANES and Py-FIMS. The $\delta^{34}S$ isotope signature of the SOM ranged from 10.6 to 15.2 ‰ in the soils at the islands and 10.0 to 13.5 ‰ in Jæren, corresponding to the Anthrosols in the Baltic Sea region (Median: $\delta^{34}S$ = 11.5 ‰) and suggest an input of marine biomass ($\delta^{34}S$ of seaweed = 20 ‰). The AMS ^{14}C dating and complementary archaeological literature implied that the soils in Jæren and Karmøy have been formed between the Roman Iron Age (500 BC to AD

500) and the Viking Age (AD 800 to AD 1,000). Our results provide strong evidence for an anthropo-pedogenesis of the humus-rich topsoils in Karmøy and indicate parallels to the plaggic soils in Jæren as well as to Anthrosols in the Baltic Sea region. Therefore, we propose to classify the humus-rich topsoils in Karmøy as Anthrosols.

Keywords:

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1. Introduction

Recent investigations show that development of humus-rich topsoils around the Baltic Sea were strongly influenced by the application of different organic materials (e.g. animal manure, ashes, turf, organic waste) and, thus, soils were classified as Anthrosols (Acksel et al., 2017). Humus-rich topsoils were also found in Southwest Norway and classified as Plaggic Anthrosols (Schnepel et al., 2014). The high P contents of these soils (up to 2,924.3 mg kg^{-1}) and archaeological data (Kvamme, 1982; Myhre, 1985; Opedal, 1994; Sølvberg, 1976) indicate a strong anthropogenic influence in the Viking Age, which points to a fundament soil amendment practiced earlier than those forming most Plaggic Anthrosols in Northwest Germany (Schnepel et al., 2014). The anthropogenic character of these soils corresponds to various authors who reported that different materials such as ashes, turf, organic waste and mineral soil material were used for soil amendment in Norway (Kvamme, 1982; Myhre, 2000; Rønneseth, 1974; Sølvberg, 1976). A combination of different materials – consisting of a wide range of possible inputs e.g. seaweed, sea sand and turfs after usage for bedding cattle – formed up to 75 cm deep humus-rich topsoils at the Orkneys (Scotland) (Davidson and Simpson 1984). The soil organic matter (SOM) source, recent SOM quality and the soil age (by ^{14}C AMS dating) of the Norwegian Plaggic Anthrosols have not yet been clarified. Furthermore, we recently found other humus-rich topsoils in the Karmøy municipality on the islands of Karmøy and Feøy, located slightly northwest of Jæren. These soils show thick humus-rich topsoils up to 30 cm and their formation cannot be explained by natural conditions. Therefore, the question arises if the formation of the humus-rich topsoils in Karmøy also has been formed by earlier agricultural practice, e.g. plaggen management, such as the plaggic soils in Jæren.

Indicators for anthropogenic influence are high contents of soil organic matter (SOM) and phosphorus (P). Additionally, high contents of black carbon (BC) often characterize soils originating from human activity. These mainly condensed aromatic structures generally originate from biomass combustion (Kleber et al., 2003; Rodionov et al., 2006; Schmidt et al., 1999). Thus, the BC in the soil can originate from the incomplete combustion of fossil fuels (e.g. coal, oil) and/or from vegetation fires (Brodowski et al., 2007; Goldberg, 1985). For example, Acksel et al. (2016, 2017) identified large contents aromatic compounds by the determination of benzene polycarboxylic acids (BPCA), by pyrolysis-field ionization mass spectrometry (Py-FIMS) and synchrotron based X-ray absorption near-edge fine structure (XANES) spectroscopy at the carbon (C) and nitrogen (N) *K*-edges (C- and N-XANES). These were relatively enriched in the characteristic biogenically mixed “hortic” horizons of the Anthrosols in the Baltic region (18% BC of C_{org}). Acksel et al. (2016, 2017) hypothesized that BC-enrichments in these soils resulted from human activities like slash and burn or the disposal of settlement residues, because natural fires were relatively rare in Central Europe in contrast to typical steppe landscapes (Tinner et al., 1999). Humus-rich soils in SW Norway have not yet been characterized by the above methods.

Another possibility to trace the source of SOM is to measure the stable isotope composition, because the isotope distribution pattern of soils differ in natural systems (Schoenau and Bettany, 1989), mainly affected by vegetation (Freney and Williams, 1983; Krouse, 1991). For example, S isotope ratios in native and cultivated Chernozems in Canada reflected the δ^{34}S abundance of their predominant vegetation, grasses, herbs and field crops (Schoenau and Bettany, 1989). Acksel et al. (2017) detected high δ^{34}S-values in deeper-lying horizons of Anthrosols on various islands in the Baltic Sea region (+13.5‰), corresponding to high $\delta^{34}S$-values in seaweed (+20‰). This suggested that seaweed was incorporated into the soils by humans because any direct marine influence (precipitation and sea spray) could be excluded. Therefore, it is possible that marine biomass was used as soil amendment in a wide range of coastal regions in Northern Europe which is assumed, e.g., in Norway for plaggen management (Austad et al., 2001; Sølvberg, 1976).

Besides the investigation of the SOM source the determination of soil age is another important issue. An indication for an ancient agricultural land use in Norway is the heathland expansion between 4,000 BC to AD 200 as a result of deforestation by human fire management (Prøsch-Danielsen and Simonsen, 2000). However, other authors reported that the described soil amendments were applied later from AD 400 to AD 600 (Kvamme, 1982; Myhre, 1985; Sølvberg, 1976). Therefore, it can be assumed that the soils at Karmøy and Feøy were also influenced by manuring practices at this time. However, archaeological artifacts have not been found in these soils and, thus, the time of formation could not be estimated in that way. A possibility for determining a maximum age of soils is the AMS ^{14}C dating of the humin fraction (Pessenda et al., 2001; Scharpenseel et al., 1986). The humin fraction can reveal an approximated maximum age of SOM due to removal of any recent organic materials with fulvic and humic acids. AMS ^{14}C datings of soils showed that the humin fraction was older by factor 1.2 to 1.7 than the corresponding age of the bulk SOM (Pessenda et al., 2001; Scharpenseel et al., 1986). The ^{14}C dating of the humin fraction of Baltic Anthrosols yielded ages that were about 500 years older than the bulk SOM (Acksel et al., 2017). The ^{14}C ages, and, thus, time periods of SOM formation in the humus-rich topsoils at Karmøy and Feøy and of the Plaggic Anthrosols at in Jæren are not known.

The diversity of organic material applied and indications of rather early amelioration practices (Kvamme, 1982; Myhre, 2000; Rønneseth, 1974; Sølvberg, 1976) make Norway and especially SW Norway an interesting area to study Anthrosols. However, Anthrosols in SW Norway have been studied to a limited degree compared to the Baltic Anthrosols. To our knowledge only Schnepel et al. (2014) have published data from Anthrosols in this region. Therefore, the present study further analyzes samples from Jæren (SW Norway) collected by Schnepel et al. (2014) and additional samples collected from humus-rich topsoils at the islands of Karmøy and Feøy in Karmøy municipality (SW Norway).

The objectives of the present study were (i) to examine the SOM sources of the soils at Karmøy, Feøy and in Jæren by qualitative and quantitative BC analyses, non-targeted bulk SOM mass spectrometry and sulphur isotopic measurements, (ii) to estimate the age of SOM by AMS ^{14}C dating in order to find out evidence for an anthropogenic influence on the soil formation at Karmøy and Feøy, and (iii) to propose the consequences for soil classification according to the WRB system (IUSS Working Group WRB, 2014).

2 Materials and Methods

2.1 Study areas and soil sampling

Soil samples were collected at the islands of Karmøy (59°15′N, 5°15′E, 178 m²) and Feøy (59°23′ N, 5°9′ E, 1.3 km²) in Rogaland County in South West Norway (Fig. 1). Both islands are located north of the city of Stavanger and lie in an area with maritime climate. For the 1961–1990 normal, the mean annual temperature was 7.4ºC and the mean annual precipitation 1,180 mm (Sola observation station, close to Stavanger airport; Norwegian Meteorological Institute). Highest temperatures occur in August (14.4°C) and lowest in February (0.6°C).

The underlying bedrock of the islands is Caledonian nappe rocks (Roffeis and Corfu, 2014), consisting dominantly of gabbro and diorites (Norges Geologiske Undersøkelse, 2017). Areas mainly along the western coast in the southern part of Karmøy are covered with marine sediments while other parts of the island are covered with till (Norges Geologiske Undersøkelse, 2017). Investigations revealed that the uppermost layer of the glacigenic material consists of clayey till from the Late Weichselian glaciation (younger than 25 ka) (Andersen et al., 1981, 1983). The southern part of the island of Karmøy became ice free before 16 ka (Gump et al., 2017).

Karmøy and the areas around have a long land use history. First settlements are from the Paleolithic (12,000–5,500 uncal. BP) and, although there are no archeological findings of the first agriculturalists, it can be assumed that agricultural practices were carried out since about 5,500 uncal. BP (Hernæs, 1997). Karmøy and Feøy belong to the agricultural region 'Coast from South Norway to Nordland [county in North Norway]' (Puschmann et al., 2004). This very diverse agricultural region is dominated by grasslands used for livestock fodder production (Puschmann et al., 2004). Currently, about 25% of the municipality of Karmøy (including Feøy) are classified as agricultural land, and about 17% of the area are classified as forests and about 5% as wetlands (Statistics Norway, 2017).

The samples of the humus-rich topsoils were collected from profiles at Sandhåland (S) and at Hillesland (Hi) at the island of Karmøy and at the island of Feøy (F). From each profile samples were taken from the upper (0–30 cm) and the underlying (30–45 cm) horizons. The German soil description system (Ad-hoc-AG Boden, 2005) has been used to classify the horizons. The parent materials of the sites differ. While the underlying bedrocks at Sandhåland and Hillesland are covered with marine sediments and till, respectively, at least more than 50% of the gabbro at Feøy lacks any covering material (Norges Geologiske Undersøkelse, 2017). The soil sampling was performed in October and we found no groundwater. Local wells are very deep (> 100 m) and therefore, we have no information about the groundwater level eventually affecting the SOM dynamics. However, color photograph of profile F indicates common abundance of mottles (5-15%) and the grey color of profile S indicates rather reductive conditions at 30 to 70 cm, which call for some restrictions in the decomposition of primary organic matter.

For Sandhåland historical records show that the site has been used for cultivation at least since the 1700s (Lundberg, 2008). However, since the establishment of the farm may go back to the older Iron Age (i.e. the period before AD 600) and the oldest documented farm buildings were located close to the site (Lundberg, 2008), a cultivation starting much earlier than the 1700s is likely. As part of the farming system at least since the 1800s and until the 1960s, the fields at Sandhåland were fertilized with deposited marine biomass that was collected mostly during autumn and winter and left to decompose until the spring (Lundberg, 2008). In addition, peat mixed with organic materials, as for example, heather and fish residues was used to fertilize the fields of the farm (Lundberg, 2008). Hillesland may have been populated during the same time period as Sandhåland; written documentation of the farm starts in the 1700s (Lundberg and Handegård, 1996). A manuring technique documented for at least the second part of the 1700s is the production of a kind of 'compost' based on, for example, waste from slaughtering and fish cleaning in barrels or vats (Lundberg and Handegård, 1996). In the 1800s marine biomass, peat, mould, heather, etc. were used to produce compost (Lundberg and Handegård, 1996). Feøy has a long history of use for livestock grazing. The island has been free of forest since at least 2,000 years, when the native forest had been cleared for grazing land (Lundberg, 1998). When the samples were collected, the sites at Sandhåland and Hillesland were used as grasslands, while the site at Feøy was used for sheep grazing. The available area for agricultural usage is restricted due to the surrounding rock outcrops and the soil area of the humus-rich topsoils may be estimated at 49.4 km^2 at Karmøy (main area: 230 km^2) and 0.16 km^2 at Feøy (main area: 1.3 km^2).

Additionally, we have included soil samples of four profiles of Plaggic Anthrosols from SW Norway published by Schnepel et al. (2014) because the quality and source of the SOM and the formation time of these Anthrosols have not yet been investigated. These soil samples were collected from the upper (0-30 cm) and the underlying (30-60 cm) horizons from the profiles PE1 (Njærheim), PE2 (Byberg), PE3 (Heigre), and PE4 (Årsvol). For a detailed description of the investigation sites see Schnepel et al. (2014).

Figure 1

2.2 Sample pretreatment and basic soil characteristics

For chemical analyses, the samples were air-dried (60 °C) and sieved < 2 mm. After ball-milling for 10 min, finely ground subsamples were used for determinations of total C- and N-contents (VARIO EL analyzer; Elementar Analysensysteme GmbH, Hanau, Germany). To measure total P-contents (P_t) photometrically (Shimadzu UV-Mini–1240), a soil extract was tinged with a vanadate-molybdate reagent after combustion at 800ºC and acid hydrolysis with HNO_3 (Hubbe et al., 2007; Schnepel et al., 2014). Exchangeable cations were analyzed using atomic absorption spectroscopy (AAS, Varian SpectrAA 300) after extracting 10 g soil thrice with 25 mL 0.1 M $SrCl_2$ (pH 8.2) in a column procedure. To determine cation exchange capacity (CEC), the soil columns were further treated with 3 portions of 25 mL 0.1 M $MgCl_2$, and the obtained Sr was measured using AAS (Varian SpectrAA 300) (Schnepel et al., 2014). Physical soil characteristics (texture, bulk density) and pH were determined according to standard procedures (Blume et al., 2011). The measurements of total P, exchangeable cations, CEC and physical soil parameters were carried out by Carolin Schnepel (IBU, Soil Science, University of Oldenburg). Soil color of <2 mm-samples was determined using a Konica Minolta CR410 Chroma Meter (28217 Bremen, Germany). The applied measurement technology and system (L*a*b) for determining soil color have been described in detail, e.g. by Acksel et al. (2016).

2.3 Quantification of benzene polycarboxylic acids (BPCAs)

The BC content was quantified by the $BPCA_S$ method (Glaser et al., 1998, Brodowski et al., 2005, Kappenberg et al., 2016). This method has been applied previously to the Anthrosol profiles at the islands of Poel, Fehmarn and Sjaelland in the Baltic Sea region (Acksel et al., 2016, 2017).

2.4 Pyrolysis-field ionization mass spectrometry (Py-FIMS)

About 5 mg of finely ground sample was pyrolyzed in the ion source (emitter: 4.7 kV, counter electrode −5.5 kV) of a double-focusing Finnigan MAT 95 mass spectrometer. The samples were heated in high vacuum of 10^{-4} Pa from 100 °C to 700 °C in temperature steps of 10 °C per magnetic scan. Between the scans the emitter was flash heated to 1,500 °C to prevent the accumulation of residues. About 60 spectra were recorded for the *m/z* range 15 to 900 for each three replicates. Marker signals (*m/z*) were assigned to relevant compound classes of SOM as described in Leinweber et al. (2009) and references therein. Volatilized matter (VM, in % w/w) was calculated by mass loss of the samples due to pyrolysis.

2.5 Carbon K-edge X-ray absorption near edge fine structure (XANES) spectroscopy

Samples were prepared by drop coating as described in detail by Kruse et al. (2011). Carbon and nitrogen *K*-edge spectra were collected at the High Resolution Spherical Grating Monochromator (SGM) beamline of the Canadian Light Source synchrotron, Saskatoon, SK, Canada in the fast scan-mode. Thirty scans were measured and averaged for each soil sample to obtain representative spectra. The normalization of the spectra was carried out using the Athena software (Demeter 0.9.25, Bruce Ravel). The C *K*-edge spectra were energy calibrated using the C → π* C=O transition of glutaric acid at 288.6 eV (Kim et al., 2003). The main spectral features were assigned by comparison with published spectra of reference compounds (Kruse et al., 2011). The curve fitting program fityk (version 0.9.8, verified 23 June 2011; Wojdyr, 2010) was used for a quantitative analysis of the different functional groups within a single spectrum. The spectra were fitted with a background arc tangent function and a series of Lorentzian curves (Gillespie et al., 2011). The peak height of the Lorentzian curve was used to determine the content of the individual chemical component. The peak height of all 1 s → π* features in each spectrum was set at 100 % to enable relative quantitative comparisons among spectra. Features of the C-XANES spectra were assigned to quinone, C=C in protonated/alkylated aromatics, N substituted aromatics, aliphatics, amide, carboxylic and carbohydrate functions.

2.6 Isotope signatures $\delta^{34}S$

For measurements of the isotopic composition samples were selected from the upper (0-30 cm) and the underlying (30-45 cm) horizons of the humus-rich topsoils at Karmøy and Feøy, and from of the upper (0-30 cm) and underlying (30-60 cm) horizons from the Plaggic Anthrosols in Jæren (Schnepel et al. 2014). The δ^{34}S was measured by Agroisolab (Agroisolab GmbH, D-52428 Jülich, Germany) using an elemental analyzer coupled to IRMS (EA-IRMS). Stable S isotope ratios were calibrated with the Canyon Diablo Troilit (CDT) standard. The isotope values are listed in parts per thousand (‰):

$$\delta^{34}S\ (‰) = \frac{{}^{34}S/{}^{32}S_{sample}}{{}^{34}S/{}^{32}S_{standart}} - 1 \times 1000$$

2.7 AMS ^{14}C dating of humin fractions of the underlying horizons

The humin fraction was separated from soil sieved < 2 mm by extracting 25 g soil of the underlying horizons with 500 mL 0.1 M NaOH, shaking overnight, centrifuging at 1560 · g for 15 min, discarding the supernatant (fulvic and humic acids) and freeze-drying the extraction residue (Kahn and Sowden, 1971). For AMS ^{14}C dating the humin fractions were selected from samples of the underlying horizons (30-45 cm) of the humus-rich topsoils at Karmøy and Feøy, as well as of the underlying horizons (30-60cm) from the Plaggic Anthrosols in Jæren (Schnepel et al., 2014). The ^{14}C age was determined with Accelerator Mass Spectrometry (AMS) in the radiocarbon dating lab at Max Planck Institute for Biogeochemistry (Jena, Germany). The ^{14}C ages were calculated with the $^{14}C/^{12}C$ ratio of the oxalic acid standard and the $^{14}C/^{12}C$ ratio of the samples has been corrected to a $\delta^{13}C$ value of -25‰. The International Radiocarbon Dating Standard is N.I.S.T (National Institute of Standards and Technology; Gaithersburg, Maryland, USA) Oxalic Acid I ($C_2H_2O_4$). The conversion of the ^{14}C age in calendar and calibrated dates was done with OxCal 4.2 using the IntCal13 calibration curve (Bronk Ramsey, 2009; Reimer, 2013).

2.8 Data calculations and statistical treatment

All data were statistically evaluated and tested by the statistics software “R” (ver. 3.0.2, R Core Team, 2013). Differences in basic soil parameters and Py-FIMS data between the horizons were tested for significance (*$P < 0.05$, **$P < 0.01$, ***$P < 0.001$) by the student *t*-test and differences between the soil profiles were tested by one-way ANOVA (F-test; $P < 0.05$). The partial least squares (PLS) regressions for differences between the chemical compositions of the soils were computed for the portions of compound classes from Py-FIMS and XANES.

3 Results

3.1 Basic soil properties of the humus-rich topsoils at Karmøy

The humus-rich topsoil at Feøy (F) shows a very pronounced Axp-horizon up to 30 cm thickness (x = biogenically mixed, p = cultivated), a transitional Ax+ilCv- horizon (i = siliceous, l = loose material, v = weathered) and a following parent material ilCv-horizon (Fig. 2). The Axp-horizon was rich in humus, had a high bioturbation and was strongly rooted. The humus content decreased in the following Ax+ilCv-horizon with increasing sand content. The parent ilCv-horizon was relatively enriched in sand, stones and weathered igneous rock fragments. The profile showed a strong root penetration down to the parent ilCv-horizon. The soil profile of Sandhåland (S) had also an Axp-horizon, which was rich in humus with a strong root penetration and a high bioturbation. The following transitional tm(e)Ax+tm(e)lGo-horizon (tm = tidal marine, e = 2 to 75 % w/w carbonate) was rich in fine sand and had many clearly visible crushed molluscan shells. This horizon showed many dark, humus-rich spots, resulting from a high bioturbation whereby the humus material from the Axp- had been transported downwards into the underlying tm(e)Ax+tm(e)lGo-horizon. The basis of the profile revealed a tmGo horizon, which primarily consisted of sand and stones.

Figure 2

The pH, C_{org}-, N_t- and P-contents of the profiles at Hillesland (Hi) and at Feøy (F) were significantly different from the Plaggic Anthrosols in Jæren (Schnepel et al. 2014) (Tab. 1). These profiles had lower pH (4.6), higher C_{org}-contents up to 65 g kg^{-1}, N-contents (1.86 to 4.14 g kg^{-1}), base saturations (26 to 27 %) and higher P-contents up to 3,484.1 mg kg^{-1} compared to the Plaggic Anthrosols in Jæren. In contrast, the profile at Sandhåland (S) was slightly alkaline (7.4 to 7.6) and showed a high $CaCO_3$-content up to 299.3 g kg^{-1}. This profile also differed in respect to a low C_{org}- (1.5 %) and N-content (0.079 %) and C-stock (7 kg m^{-2}), very high base saturations (100 %) and lower P-contents of 407.4 to 764.7 mg kg^{-1} compared to the profiles at Hi and at F and the soils in Jæren. The contents and the sum of the exchangeable cations of all humus-rich topsoils in Karmøy were relatively similar, dominated by Ca^{2+} (10.8 to 24.5 $cmol_c$ kg^{-1}) and were clearly higher than in the Plaggic Anthrosols. The cations exchange capacity was higher in the profiles of Hi and F (46.9 to 83.5 $cmol_c$ kg^{-1}) in comparison to the profile S (11.5 to 25.9 $cmol_c$ kg^{-1}) and to the Plaggic Anthrosols in Jæren (21.2 to 22.9 $cmol_c$ kg^{-1}).

Table 1

The bulk densities of the soils in Karmøy were significantly different to the Plaggic Anthrosols (around 1 g cm^{-3}) except for profile S (Tab. 2). The texture of the profiles Hi and F were characterized by high contents of sand (42 to 53 %) and silt (39 to 45 %). In general, the texture of the Plaggic Anthrosols (Schnepel et al., 2014) was significantly different ($P<0.05$) from soils in Karmøy. The profile S consisted mainly of sand (up to 96 %), which was dominated by the medium sand fraction (0.2 to < 0.63 mm, 72 %). The L*-values (0 = black, 100 = white) of the profiles Hi and F and of the Plaggic Anthrosols were low in comparison to the profile S corresponding to the C_{org}-contents. Similar to the L*-values, the values of the Munsell color also indicate high contents of dark pigments.

Table 2

3.2. Pyrolysis-field ionization mass spectrometry of the soils in Karmøy and Jæren

The proportions of volatile matter (VM) of the soils in Karmøy (in average: 14.2 %) were similar to the soils in Jæren (in average: 13.4) and decreased with soil depth (Tab. 3). The total ion intensity (TII) of the profiles of Hi and F (229.0 to 372.4 × 10^6 counts mg^{-1}) were relatively similar to the soils in Jæren (261.7 to 346.7 × 10^6 counts mg^{-1}) and the average was 5.2 times larger compared to the profile S (33.6 to 82.1 × 10^6 counts mg^{-1}). In general, the SOM compound classes of all soils were dominated by phenols and lignin monomers, lignin dimers, alkylaromatics, lipids and sterols. The underlying horizons of the profiles Hi and F had significantly larger proportions of phenols and lignin monomers (factor 1.3), lignin dimers (factor 1.3), lipids (factor 1.3), alkylaromatics (factor 1.5) and peptides (factor 1.1) and lower proportions of carbohydrates (factor 0.9), heterocyclic N (factor 0.9), sterols (factor 0.7), suberin (factor 0.4) and fatty acids (factor 0.5) than the upper horizons. In contrast, the underlying horizon of profile S showed significantly lower portions in all compound classes than the upper horizon, except for lipids. The horizons of the profiles in Jæren had a relatively similar distribution of compound classes with slightly higher portions of lignin dimers (factor 1.1; $P < 0.001$), lipids (factor 1.2; $P < 0.01$) and alkylaromatics (factor 1.2; $P < 0.01$), and lower portions of heterocyclic N (factor 0.9; $P < 0.05$), sterols (factor 0.9), suberin (factor 0.7; $P < 0.05$) and fatty acids (factor 0.6; $P < 0.01$) in the underlying horizons.

These differences in the portions of compound classes between the upper and underlying horizons of the soils in Jæren were similar to the soils in Karmøy. However, the distribution of the compound classes of the soils in Karmøy was significantly different to the soils in Jæren. Specifically, the portions of carbohydrates, phenols and lignin monomers, heterocyclic N and peptides of both horizons of the profile Hi and F in Karmøy were significantly lower compared to the horizons of soils in Jæren. The largest similarities among the soils of Karmøy and Jæren were the portions of sterols, suberin and fatty acids in the underlying horizons. In contrast, the profile S has similar portions of phenols and lignin monomers, lipids, alkylaromatics, heterocyclic N and peptides, but significantly lower portions of carbohydrates and lignin dimers in comparison to the soils at Jæren. Overall, the proportions of the humin fractions as isolated for ^{14}C age determination and especially the thermograms of organic matter volatilization in Py-FIMS (not shown) indicate that the biomolecules do not occur isolated as such but mostly incorporated large proportions of humic substances.

Table 3

The plot of the Partial Least Squares discriminant analysis (PLS-DA) of the relative abundance of 10 important compound classes from Py-FIMS explained about 78 % of differences among the soils. The first and second components of the PLS-DA showed a strict separation of the chemical SOM composition between the soils in Karmøy (lipids, suberin and sterols) and in Jæren (carbohydrates, phenols and lignin monomers, lignin dimers, alkyl aromatics, heterocyclic N and peptides) except of the E horizon of profile PE2. The E horizons of the Plaggic Anthrosols in Jæren were dominated by carbohydrates, phenols and lignin monomers, heterocyclic N and peptides and were separated from their A horizons by fatty acids. The underlying horizons of the soils in Karmøy were clearly separated from their upper horizons in sterols.

Figure 3

3.3. Carbon K-edge XANES

The C *K*-edge XANES spectra of the soils in Karmøy and Jæren had two dominant features from C 1 s → π* transition at 285.0 eV (b) and 288.7 eV (j) (Fig. 4). The soil profile of S showed additional distinct features at 290.1 eV, 295.3 eV, 298.2 eV, 300.6 eV and 301.8 eV (l). Furthermore, all spectra of the soils revealed additional small features at 284.0 eV (a), 285.6 eV (c), 285.9 eV (d), 286.2 eV (e), 286.5 eV (f), 286.8 eV (g), 287.2 eV (h) 288.0 eV (i) and 289.5 eV (k). The feature (a) was assigned to the C 1 s → π * transition in C=C in quinone (Francis and Hitchcock, 1992), feature (b) to C=C in protonated/alkylated alkenes and aromatics (Cody et al., 1998; Dhez et al., 2003) and (c) to aromatic C bound to nitriles (Hitchcock and Mancini, 1994). The feature (d) was assigned to aromatic C bound to aldehydes and/or C bound in pyridine (Cooney and Urquhart, 2004; Dhez et al., 2003) and feature (e) to carbonyl C in aldehydes and/or C bound in pyrrole (Hitchcock and Mancini, 1994; Urquhart and Ade, 2002). The feature (f) was assigned to aromatic C bound in urea, carbonyl in aldehydes, ketones, aliphatic C in nitriles and/or C bound in pyrroles, NCHNH in imidazoles and purines (Boese et al., 1997; Cooney and Urquhart, 2004; Dhez et al., 2003; Hitchcock and Mancini, 1994; Samuel et al., 2006; Urquhart and Ade, 2002) and (g) to aromatic C bound in carbamate, amine O-bound ester, C bound in NCHNH in imidazoles and purines and carbonyl C in ketones (Boese et al., 1997; Cooney and Urquhart, 2004; Dhez et al., 2003; Hitchcock and Mancini, 1994; Samuel et al., 2006; Urquhart and Ade, 2002). The feature (h) was assigned to aromatic C bound in hydroxyl and ether and aliphatic C in CH, CH_2, CH_3 (Dhez et al., 2003; Hitchcock and Ishii, 1987; Hitchcock et al., 1986; Solomon et al., 2009) and (i) to CONH and HNCONH in thymine, guanosine, uracil, aliphatic C in CH, CH_2, CH_3, carbonyl C in amides and acetate (Dhez et al., 2003; Hitchcock and Mancini, 1994; Hitchcock and Ishii, 1987; Hitchcock et al., 1986; Samuel et al., 2006; Urquhart and Ade, 2002). Feature (j) was assigned to CONH and HNCONH in thymine, guanosine, uracil and carbonyl in aliphatic COOH (Gordon et al., 2003; Hansson et al., 2004; Samuel et al., 2006; Urquhart and Ade, 2002) and (k) to aliphatic C in C-OH and urea (Dhez et al., 2003; Ishii and Hitchcook, 1988; Urquhart and Ade, 2002). The feature (l) consisting of four broad absorption peaks was assigned to carbonate (Dhez et al., 2003; Urquhart and Ade, 2002).

The soils at Hi and at F had relatively similar portions of feature (a) 4.5 to 5.6 % and (b) 10.4 to 12.2 % in comparison to the soils in Jæren ((a): 5 % and (b): 11.1 %). In the soils in Jæren the portions of feature (c) revealed some enrichment in aromatic compounds by factor 1.3, (d) by factor 1.6 and (e) by factor 1.3 compared to the soils at Hi and at F. The portions of features (f) around 3 %, (g) 3 %, (h) 7 %, (i) 20 %, (j) 17 % and (k) 19 % of the soils at Hi and at F were similar to the soils in Jæren. The profile of S at Karmøy had the lowest portions of features (a) to (j) and the highest portions of feature (k) up to 24.9 % and (l) up to 21.1 % than all other soils.

Figure 4

The plot of the Partial Least Squares discriminant analysis (PLS-DA) of the portions of peak heights from the normalized stacked carbon *K*-edge XANES spectra explained about 83 % of differences between the soils. The first component of the PLS-DA showed a strict separation of the chemical composition between the soils in Karmøy (aliphatic C) and in Jæren (aromatic C, C bound to heterocyclic N). Furthermore, the upper horizons of the soils in Jæren were clearly separated from the underlying E horizons by the second compound except for the E-horizon of profile PE2 and the Ap-horizon of PE3. The PLS-DA showed no differences between the upper and the underlying horizons of the soils in Karmøy. Spectra of the Plaggic Anthrosols in Jæren were dominated by features c, d, e, f, g and b, and of the soils in Karmøy by features h, a, I, j and k.

Figure 5

3.4. Benzene polycarboxylic acids (BPCAs) of the soils in Karmøy and Jæren

The BC contents of the humus-rich topsoils at Karmøy were relatively low, ranging from <0.1 to 0.7 in the upper and 0.1 to 1.4 g C kg^{-1} soil in the underlying horizons (Fig. 6). The BC contents of the plaggic soils in Jæren ranged from 2.0 to 6.0 in the upper and 0.2 to 5.5 g C kg^{-1} soil in the underlying horizon and were significantly higher (P<0.05) in comparison to the soils in Karmøy.

Figure 6

3.5. Isotope signatures $\delta^{34}S$ of the soils in Karmøy and Jæren

The $\delta^{34}S$ values of the humus-rich topsoils in Karmøy ranged from 10.6 to 14.1‰ in the upper and from 12.3 to 15.2‰ in the underlying horizons, similar to the $\delta^{34}S$–values of the marine material ranging from 9.5 to 21‰ (Fig. 7). The soils in Jæren had $\delta^{34}S$ values in the upper (10.0 to 11.1‰) and the underlying horizons (11.9 to 13.5‰), similar to the soils in Karmøy.

Figure 7

3.6. AMS ^{14}C dating of the humin fraction of soils in Karmøy and Jæren

Four AMS^{14}C radiocarbon ages date the humin fraction of two soils in Karmøy (Hi and S) and of two soils Jæren (PE2 and PE3) to the Viking Age (~ AD 800 to about AD 1,000). The AMS^{14}C age of AD 430 to AD 580 points at an earlier formation of the organic matter in PE1 in Jæren. There is an AMS^{14}C age for each one soil in Karmøy (F) and Jæren (PE2) dating to the period AD 1,220 to AD 1,300.

Table 4

4. Discussion

4.1 Basic soil characteristics as indicators for soil formation in Karmøy

The P-contents of the soils in Karmøy (up to 3,484 mg kg^{-1}) (Tab. 1) were extraordinarily high compared to other natural and anthropogenic European soils. For instance, P-contents of natural soils in temperate climates range from 200 to 800 mg kg^{-1} but are < 100 mg P kg^{-1} in sandy soils (e.g. Podzols, Ultisols) (Scheffer and Schachtschabel, 2010). Examples for soils with high P-contents are the Plaggic Anthrosols in NW Germany which range between 50 to 1,700 mg P kg^{-1} (Giani et al., 2013, Blume and Leinweber, 2004). Other Anthrosols had P-contents from 215 to

1,034 mg P kg^{-1} (Wiedner et al., 2014), 262 to 1,000 mg P kg^{-1} (Lauer et al., 2013) in Germany, and from 394 to 768 mg P kg^{-1} in Scotland (Simpson, 1997). Schnepel et al. (2014) detected P-contents of up to 2,924 mg P kg^{-1} in plaggic soils (Tab. 1). The even higher P-contents measured for the soils in Karmøy clearly indicate an early strong manuring or fertilization (Holliday and Gartner, 2007). Especially sheep faeces (pasture grazing, vegetation dominated by ryegrass and white clover) contain 1.7 g P kg^{-1} of fresh matter (8.0 g P kg^{-1} of dry matter) which is much more than in cattle faeces with around 0.561 g P kg^{-1} of fresh matter (5.5 g P kg^{-1} of dry matter) (McDowell and Stewart, 2005). Since this region has a long history of use for sheep, cattle and horses grazing (Lundberg, 1998) it is possible that the high P-contents of the soils are also caused by animal faeces.

Another indication for large organic matter inputs are the high C_{org}-contents of the profiles Hi and F (up to 65 g kg^{-1} soil) (Tab. 1) which were similar to and/or even higher than in other anthropogenic soils. For example, C_{org}-contents in a Pretic Anthrosol in Eastern Germany ranged from 10.8 to 21.7 g kg^{-1} (Wiedner et al., 2014), in Hortic, Mollic and Umbric Anthrosols in the Baltic Sea region from 5.2 to 22 g kg^{-1} (Acksel et al., 2016, 2017), in anthropogenic pits of Neolithic settlements in Bavaria/Germany from 4.9 to 41 g kg^{-1} (Schmid et al., 2002), in Plaggic Anthrosols in NW Germany from 1.5 to 29 g kg^{-1} (Blume and Leinweber, 2004; Giani et al., 2014), in soils from European North Russia from 14.9 to 37.1 g kg^{-1} (Hubbe et al., 2007) and in Jæren, SW Norway, from 26.3 to 51.6 g kg^{-1} (Schnepel et al., 2014), in anthropogenic soils formed by a process analogous to plaggic soils at Tofts Ness, Sanday, Orkney from 10 to 62 g kg^{-1} (Bull et al., 1999), and in Terra Preta in the Brazilian Amazon region from 22 to 45 g kg^{-1} (Glaser et al., 2000). Furthermore, it can be assumed that the profiles of Hi and F were subjected to intense inputs of stable organic matter and/or the SOM was protected from rapid decomposition. Some influence of the soil moisture regime (slightly stagnic properties visible by mottling) and stabilization by clay- and silt-minerals is indicated by the larger C_{org} concentration in Hi and F compared to the more coarsely textured profile S. Therefore, the combination of high inputs of organic matter and various stabilization processes likely resulted in the large C-stocks. The profiles have large C-stocks (17 to 23 kg m^{-2}) despite a horizon thickness of only 45 cm compared to the plaggic Anthrosols at Jæren which had a C-stock of 21 kg m^{-2} at 60 cm horizon thickness. The calculated C-stocks of Baltic Anthrosols ranged from 7 to 18 kg m^{-2} with an average horizon thickness of 70 cm (Acksel et al., 2016, 2017), which indicated a lower input and/or higher decomposition compared to the soils at Hi and at F. The C_{org}-content and C-stock of profile S were relatively low compared to the profiles of Hi and F. Despite the smaller horizon thickness and the sandy texture, the comparably large C_{org}-contents and C-stocks of profiles of Hi and F indicate a larger C accumulation and/or slower mineralization than in the profile S and in the Baltic Anthrosols described by (Acksel et al., 2016, 2017).

The relatively high N-contents of 1.86 to 4.14 g kg^{-1} in the soils at Hi and at F were similar to various Anthrosols (Acksel et al., 2016, 2017; Wiedner et al., 2014) and indicate a strong fertilization because N-contents of A-horizons from various mineral soils in moderate humid climate ranged from 1 to 2 g kg^{-1} (Scheffer and Schachtschabel, 2010). An indication for the source of the SOM in the profiles Hi and F is the C/N-ratio compared to the plaggic soils at in Jæren. C/N-ratios of soils can be strongly influenced by different N-contents of organic amendments. Kalinina et al. (2009) explained low C/N-ratios (10 to 20) in Russian Plaggic Anthrosol by the application of fen peat because of its high N-contents (2.5 to 4.4 %: Göttlich, 1990) compared to the use of N-poor heather material (Swift et al., 1979) that formed Plaggic Anthrosols at Oldenburg (NW Germany), which had higher C/N-ratios (18 to 25) (Giani et al., 2013). Therefore, the comparably low C/N ratios in the soils at Hi and at F could be caused by the input of N-rich organic material, e.g. fish residues and composted manure, in comparison to the soils in Jæren. On the other hand, the sites at Hillesland, Feøy and Sandhåland were used as grazing land for more than 2,000 years (Lundberg, 1998) and this calls for the abundance of at least some legume species (e.g. white clover) in the grasslands. However, we believe that especially the spreading of excrement/manure was responsible for the current high N-contents as the grazing of animal (sheep and cattle) and the total P contents indicate.

The cation exchange capacities (CEC) of the soils in Karmøy (11 – 80 $\mathrm{cmol_c\ kg^{-1}}$) were higher than in humus-rich topsoils from Baltic Anthrosols (15 – 27 $\mathrm{cmol_c\ kg^{-1}}$) (Acksel et al., 2016, 2017), Pretic Anthrosol in Lower Saxony, Germany (4 – 15 $\mathrm{cmol_c\ kg^{-1}}$) (Wiedner et al., 2014) and/or in Terra Preta (7 – 40 $\mathrm{cmol_c\ kg^{-1}}$) (Glaser et al., 2000). The high CEC and contents of plant available nutrients of the soils in Karmøy (Ca, Mg, Na, and K) indicate a high content of decomposed humic materials.

The natural conditions cannot explain these high C-stocks due to the displacement of the natural vegetation by the heathland expansion between 4,000 BC and AD 200 by human fire management for the reclamation of agricultural land (Prøsch-Danielsen and Simonsen, 2000). Equally, the topsoil at Sandhåland is located on an ancient beach, probably with sparse natural vegetation. There are reports that different materials such as peat, heather, seaweed, organic waste, ashes, sand and the mixtures of the above different materials were used as soil amendments in Northern European coastal regions, all resulting in humus-rich topsoils (Acksel et al., 2017;

Davidson and Simpson, 1984; Kvamme, 1982; Myhre, 2000; Rønneseth, 1974; Schnepel et al., 2014; Sølvberg, 1976). Therefore, the extraordinarily high P-contents, high C-stocks, N-contents and the horizon thickness of the soils in Karmøy plausibly can be explained by previous large inputs of organic matter by human activities.

4.2 Identification of the SOM sources of the soils in Karmøy and Jæren

4.2.1 Aromatic compounds as indicator for combustion residues

The BC contents of the soils in Jæren (mean: 3.4 g BC kg^{-1}) revealed no significant differences ($P<0.05$) to various Anthrosols (mean: 2.9 g BC kg^{-1}) and humus-rich natural soils (mean: 3.3 g BC kg^{-1}) (Fig. 6). The BC contents of the soils in Karmøy (0.6 g BC kg^{-1}) were similar to humus-poor soils (1.0 g BC kg^{-1}, average value from Gerlach et al., 2006, 2012; Schmidt et al., 1999; Wiedner et al., 2014). In contrast, the BC contents were significant lower ($P<0.05$) than in various humus-rich soils (Anthrosols, Chernozems, Kastanozems) (Acksel et al., 2016, 2017; Gerlach et al., 2006, 2012; Glaser et al., 1998; Glaser and Amelung, 2003; Lauer et al., 2014; Rodionov et al., 2010; Schmid et al., 2002; Schmidt et al., 1999; Wiedner et al., 2014) and the soils in Jæren (Fig. 6).

Differences in the chemical SOM composition between the soils in Karmøy and Jæren are clearly derived from the PLSs of compound class-proportions from the non-targeted analyses Py-FIMS (Fig. 3) and C-XANES (Fig. 5). Complementary to the higher BC contents of the soils in Jæren, we also derived higher portions of C bound to heterocyclic N compounds by C-XANES (factor 2 for peak (d) and factor 1.5 for peak (e)) and heterocyclic N by Py-FIMS (factor 1.5) than in the soils in Karmøy. The enrichments of C bound to heterocyclic N with increasing BC were also confirmed by the significant positive correlation between the portions of peak (d) from C-XANES and the portions of BC in these soils (BC % of C_{org} = 8.765 x - 16.23; r^2 = 0.75***).

Aromatic C compounds and heterocyclic N (pyrroles, indoles and/or pyridines) can originate from charred materials (Kiersch et al., 2012a, 2012b). Acksel et al. (2016) detected high portions of aromatic C and heterocyclic N with the complementary methods XANES and Py-FIMS along with high BC-proportions in Anthrosols from the Baltic Sea region. These analytical data provided compelling evidence for the input of charred material in these soils which likely also holds true for the soils in Jæren.

Various authors reported that high BC contents in some soils from Europe outside from steppe landscapes originate from human activities like slash and burn or the disposal of settlement residues, supported by patchy distributions over a wide range of BC contents in close coincidence with Neolithic settlement sites. High BC contents were analyzed in pit fillings (mean: 1.6 g BC kg^{-1}) from prehistoric settlement areas in Central and Western Germany (Lauer et al., 2014), in pits (mean: 1.6 g BC kg^{-1}) in the Lower Rhine Basin (Gerlach et al., 2006) and on larger spatial scale in Hortic Anthrosols (mean: 1.8 g BC kg^{-1}) at islands in the Baltic Sea region (Acksel et al., 2016, 2017), in a Hortic Anthrosol (mean: 3.1 g BC kg^{-1}) in Lower Saxony, Germany (Wiedner et al., 2014) and in pits of neolithic settlements (mean: 3.5 g BC kg^{-1}) from Bavaria, Germany (Schmid et al., 2002). Equally, the high BC content of the soils at Jæren provide clear evidence for the input of combustion residues. These may originate from deforestation as indicated by the heathland establishment in SW Norway which was hypothesized from a high influx of charred particles combined with anthropogenic indicator pollen such as *Plantago lanceolata*, *Potentilla-type* and *Lotus-type* (Kaland, 1986; Øvstedal, 1985; Sundve. 1977) or from settlements.

Prøsch-Danielsen and Simonsen (2000) reported that the period of heathland expansion (4,000 BC to AD 200), resulting from deforestation by human fire management for the reclamation of agricultural land, was mainly completed by the end of the Bronze Age. It can be assumed that the plaggic management in general began between AD 1,250 and AD 800 (Simpson, 1993) which is around 1,000 years later than the proven fire management from the Bronze Age. Furthermore, most of the BC was found in the upper horizon of the soils at Jæren and indicate a later continued or recent input of combustion residues.

The input of combustion residues is supported by various publications which show that many materials, e.g. ashes, were applied to Norwegian soils (Kaland, 1987; Myhre, 1985, 2000; Sølvberg, 1976). Therefore, the main BC in the soils at Jæren seems to originate from the input of settlement residues rather than from deforestation management. Apart from this, the similarities to BC contents in fire-unaffected soils (Fig. 6), the very low BC contents and the results from XANES as well as Py-FIMS of the soils in Karmøy indicated no influence of fire and/or input of combustion residues to these soils. Therefore, it can be assumed that fire did not play a significant role for the formation of the humus-rich topsoils in Karmøy, which is different from the soils in Jæren.

4.2.2 Sulphur isotope composition as indicator for marine or terrestrial material

δ^{34}S–values from Karmøy and Jæren (up to 15‰) were significantly different to typical continental soils (Chukhrov et al., 1979; Eriksen, 1996; Krouse and Tabatabai, 1986; Krouse, 1980; Novák et al., 2003), terrestrial plants (Freney and Williams, 1983; Krouse et al., 1991; Kusakabe et al., 1976; Schoenau and Bettany, 1989) and fertilizers (Mizota and Sasaki, 1996), that all have δ^{34}S–values near zero (terrestrial mean). The enriched δ^{34}S–values of the soils in Karmøy and Jæren thus indicate a marine influence. The close location to the coast is one potential explanation of the enrichment. The S-isotopic composition of soils and plants in coastal areas can be affected by the marine environment, which is highly enriched in δ^{34}S (Chukhrov et al., 1979). The isotopic composition of marine atmosphere is +11‰ δ^{34}S for the central atlantic ocean (Gravenhorst, 1978) and -3.8 to 4.3‰ δ^{34}S were measured in the marine atmosphere from Brittany France (Nguyen and Cortecci, unpublished data). Values of 5 – 8‰ were reported for atmospheric inputs and coastal-near soils in Skogaby, Sweden (Novák et al. 2003). Seawater and/or sea spray had δ^{34}S-values around +17.7 to +21‰ (Böttcher et al., 2007; Chukhrov et al., 1979; Rees, 1978; Thode et al., 1961). However, other authors found lower δ^{34}S-values in ocean water; e.g. the North Sea had δ^{34}S-values of +14.7‰ (Östlund 1959). The δ^{34}S-value of rain from the Atlantic Ocean ranged from +12.1 to +15.0‰ (Chukhrov et al., 1979) and from Sweden ranged from +3.4‰ (Köping), +1.8‰ (Huddinge) and +2.6‰ (Flahult) (Östlund 1959). The atmosphere and fertilizer especially influenced the inorganic sulphur fraction (sulfate) in soils (Heilmann, 2012) which accounts only 5 % of the total sulphur content in agriculture soils (Eriksen, 2009; Kertesz et al., 2004).

Therefore, it can be assumed that the ^{34}S/^{32}S isotope composition of the soils will be less influenced by the sulphur deposition from the atmosphere and seaspray of the Atlantic coast but rather by vegetation and organic inputs. A further aspect is that the most irreversible reactions favour the ^{32}S isotope (Tscherkez and Tea, 2013) and, therefore, the plants and soils can be decreased in ^{34}S compared to the marine and/or terrestrial atmosphere. For example, δ^{34}S-values of native and cultivated soils in Canada, e.g. in Chernozem (Ah: −1.4‰, grass: −2.7‰; Ap: −4.0‰, wheat straw: −1.7‰), Gleysol (Ahe: −7.3‰, grass: −5.4‰; Ap: −5.2‰, persicaria: −2.1‰) and

Luvisol (Ae: +1.7‰, leaves, grass, moss: +3.2‰; Ap: +1.1‰, barley straw: +2.5‰) were less than the $\delta^{34}S$ abundance of their atmosphere ($\delta^{34}S$ = +6.0‰) (Schoenau and Bettany, 1989). Isotopic composition of peat bog on the Shetland Isles, UK shows that the input of S by surface water to the peat have a maritime-dominated isotopic composition close to $\delta^{34}S$ = +20‰ but the uptake of S by vegetation introduces a -10‰ shift in $\delta^{34}S$ from these input values and is explained by the isotopic fractionation, since plants preferentially incorporate ^{32}S (Bartlett et al., 2005). Besides the atlantic influence the soils in SW Norway also can be influenced by precipitation coming from the mainland which contributed to lower $\delta^{34}S$-values that at the Shetland island. Therefore, the $\delta^{34}S$-values (up to 15‰) of the hums-rich topsoils should be very lower than the $\delta^{34}S$-values of the atmospheric inputs (10.0 to 20.0‰) due to the isotopic fractionation. However, the isotopic composition of the hums-rich topsoils (+10.0 to +15.2‰) is very similar to the atmosphere, precipitation and sea spray. A further aspect is that the $\delta^{34}S$-values of the hums-rich topsoils should be decreased with the distance from the coast to inland. Mizota and Sasaki (1996) plotted the $\delta^{34}S$-values of native soils from Japan as a function of the distance from the coast showing a decrease of $\delta^{34}S$ towards the inland (0 to 1 km = +10 to 18‰; 1 to 2 km = 10 to 13‰; about 2 km = 0 to 10‰). Wakshal and Nielsen (1982) reported similar decreased $\delta^{34}S$-values in rainwater from the Mediterranean Coast to the Jordan Rift Valley and Golan Heights, Israel (0.5 km = 9.5‰; 10.6 km = 6.8‰; 16.3 km = 5.0‰; 32 km = 5.6‰). However, the soils of Karmøy and Jæren show no decreased $\delta^{34}S$ values with the distance from the coast to inland (S = 0.1 km = 12.8-15.2‰; F = 0.35 km = 12.5-13.5‰; Hi = 1.5 km = 10.6-12.3‰; PE2 = 2 km = 10.8-11.9‰; PE4 = 2.4 km = 11.0-13.5‰; PE3 = 5 km = 10.0-13.4‰; PE1 = 8 km = 10.3-12.0‰). Equally, Acksel et al. (2017) showed that various topsoils at the islands of

Fehmarn and Poel in the Baltic Sea region were not enriched in δ^{34}S–values, although located as close as 1 km to the coast. Therefore, it can be concluded that the enrichment of δ^{34}S in the humus-rich topsoils in Norway should be caused by other sources than by the marine atmosphere.

Various authors reported that seaweed or other marine biomass was used as soil amendment in coastal regions (Ireland, Orkney Islands) as an alternative to animal manure (Chapman, 1970; Conry, 1971; Frazer, 1807; Simpson, 1993). The extraordinarily high δ^{34}S-values in these soils can be explained by the fact that seaweed and other marine plants contain high concentrations of organic S compounds as a result of elevated S uptake from seawater (Jaulneau et al., 2010) and are enriched in δ^{34}S (+20‰) compared to terrestrial plants. Therefore, the input of marine biomass to soils can result in higher δ^{34}S-values. Acksel et al. (2017) detected high δ^{34}S-values in deeper-lying horizons of Anthrosols at various islands in the Baltic Sea region (+13‰) corresponding to high δ^{34}S-values in seaweed (+20‰) that is regularly deposited at the beech during storm events, thus providing evidence that marine biomass, likely seaweed, was incorporated into the soils by human activities. Lundberg (2008) reported that the ancient farmers at Sandhåland (S) collected marine biomass (*laminaria spp.*) from the beach during autumn and winter and placed this material in large heaps for decomposition to spread it on the fields in the following spring, because fresh marine biomass would damage cultivated plants. For the soils in Jæren, which were formed by plaggen management (Schnepel et al., 2014), it can be assumed that dried marine biomass was used as alternative bedding material in stables before being incorporating into arable soils, comparable to typical plaggen management with other bedding materials in northern Germany (Blume and Leinweber, 2004). This is in agreement with various authors, who reported that the input of mixtures of sea sand, seaweed, peat and/or animal dung was responsible, among others, for the formation of Anthrosols in Ireland and Scotland (Chapman, 1970; Conry, 1971; Frazer, 1807; Simpson, 1993).

The question arises, how many tons of seaweed may have been transported to the fields to attain such high C_{org} contents and $\delta^{34}S$-values in these soils. A possibility to estimate/calculate the biomass input is the application of a simple exponential model. The turnover of carbon can be calculate after Jenkinson and Johnston (1977) with the equation: $C_r = C_e + (C_o – C_e) \times e^{-rt}$ (C_e (t ha^{-1}) = total C content of the soil when equilibrium has been reached, C_o (t ha^{-1}) = the initial C content of the soil, e = the Euler constant, t = time period in years, r = decomposition rate ($year^{-1}$) and C_r (t ha^{-1}) = the carbon content of the soil at the respective time). To calculate the seaweed input in these soils we assume an average decomposition rate of the organic matter with r = -0.02 $year^{-1}$. This value was calculated for a soil where roots and stubble had been ploughed in (Kortleven, 1963). According to this calculation we estimate an input of dried seaweed around 14 t ha^{-1} and/or 46 t ha^{-1} fresh seaweed per year in Hillesland, 16 and/or 52 t ha^{-1} at Feøy, 4 t ha^{-1} and/or 13 t ha^{-1} at Sandhåland and 13 t ha^{-1} and/or 43 t ha^{-1} at Jæren for about 1,000 years of cultivation. The higher calculated input of biomass in the soils at Hillesland and Feøy can be explained by the limited available area for agriculture usage due to the surrounding rock outcrops or the soils were additionally fertilized with other organic amendments (In the 1800s marine biomass, peat, mould, heather, etc. was used to produce compost; Lundberg and Handegård, 1996). Since the soil of Sandhaland is situated directly on the beach it is possible that these soils have been fertilized with seaweed/marine biomass more than the other soils. On the other hand, the low calculated input of 4 t ha^{-1} (dried) and/or 13 t ha^{-1} (fresh) of profile S originates from the low C_{org} which in turn may haven been caused by a comparably stronger mineralization (indicated by the sandy texture). Therefore, it can be assumed that the profile S was manured with the same amounts of organic amendments (especially with seaweed/marine biomass) as the other profiles (13 t ha^{-1} (dried) and/or 52 t ha^{-1} (fresh)). Overall, these inputs of marine biomass (seaweed and/or algae) by human activities plausibly explain the enrichment of δ^{34}S in soils in Karmøy and Jæren. This means that the ancient farmers transported marine biomass over relatively large distances from the beach to their agricultural land in the mainland.

4.3 The timing of soil formation in Karmøy and Jæren

The AMS^{14}C radiocarbon ages date the humin fraction of two soils in Jæren (PE2 and PE3) to the Viking Age (AD 800 to AD 1,000), which was the assumed time of formation for plaggen soils by Schnepel et al. (2014). It is possible, however, that the soils are older than the measured radiocarbon age because the humin fraction may still contain some recent organic matter despite the removal of fulvic and humic acids (Scharpenseel et al., 2014). A possible contamination with recent organic carbon inputs and the turnover of SOM complicates the dating of these humus-rich ancient soils. Radiocarbon ages of organic matter should be considered therefore as minimum ages related to the onset of soil formation (Gilet-Blein et al., 1980). Scharpenseel et al. (1968) added around 1,000 years to the measured ^{14}C age of the bulk SOM from mollic horizons in order to estimate the absolute age of the SOM. Equally, Acksel et al. (2017) hypothesized a formation of Anthrosols earlier than AMS^{14}C ages of humin fractions in the Baltic Sea region that corresponded to the beginning of agricultural land use as supported by settlement history. According to Lundberg (2008) a farm at Sandhåland (S) at Karmøy in Norway may have its origin in the older Iron Age, i.e. the period before AD 600. A further indication for an ancient agricultural land use and the simultaneous formation of the soils in Karmøy and Jæren is the heathland expansion between 4,000 BC to 200 BC (Prøsch-Danielsen and Simonsen, 2000). Therefore, if we accept that the soils at Karmøy and Jæren are older than the measured AMS^{14}C age and compare this with the reports of Lundberg (2008), Prøsch-Danielsen and Simonsen (2000), the formation of these soils may have begun during the Roman Iron Age period between 500 BC to AD 500.

4.4 Consequences for the classification of the soils in Karmøy

The results of this study indicated a strong anthropogenic influence on the formation of the humus-rich topsoils in Karmøy. In the WRB (IUSS Working Group WRB, 2014), soils created by human activities are classified as Anthrosols and were subdivided according to diagnostic horizons in Hortic, Pretic, Terric and/or Plaggic Anthrosols (Tab. 5). According to the properties of these diagnostic horizons, the upper horizon (0-30 cm) of the profile S can be classified as hortic horizon (SOC > 1%, extractable P_2O_5 > 100 mg kg^{-1}, base saturation > 50%, thickness > 20 cm, colour value < 3 (moist)). The analytical characteristics of the profiles Hi and F also only fulfill the diagnostic criteria of hortic horizons with the exception of the low base saturation (<50%), which is an important criterion for distinguishing horizons in the WRB. Therefore, the horizons of both profiles (Hi and F) cannot be assigned to one of the diagnostic horizons of Anthrosols, which are key to their classification as Anthrosols. Other humus-rich horizons are e.g. mollic, umbric and chernic horizons for the distinction of natural soils do not allow an assignment to Anthrosols according to the WRB. Nevertheless, we previously classified various humus-rich topsoils in the Baltic Sea region as Anthrosols despite lacking the diagnostic criteria of a hortic, pretic, terric or plaggic horizon due to compelling evidence for an anthropo-pedogenesis (Acksel et al. 2017). Therefore, we propose to classify these soils as Umbric and/or Mollic Anthrosols and to consider the source material of SOM enrichments as a further diagnostic criterion for Anthrosols. Equally, the soils at Hi and F rather meet the diagnostic criteria of an umbric horizon (SOC > 0.6%, base saturation < 50%, thickness > 20 cm, colour value < 5 (dry)). Although the umbric horizon is not a qualifier for an Anthrosol, we propose to classify the soils at Hi and F as Umbric Anthrosols due to the soil properties and the strong anthropogenic indicators (P-contents, δ^{34}S-values). These problems in Anthrosol classification call for new diagnostics horizons because more and more humus-rich topsoils were discovered that have been formed by human activities but do not comply with the current diagnostic criteria of the WRB for Anthrosols.

Table 5

5. Conclusions

Our results from Py-FIMS, C-XANES, BPCA, δ^{34}S and AMS ^{14}C-datings provide strong evidence for an anthropo-pedogenesis of humus-rich topsoils in Karmøy and indicate parallels to the plaggic soils in Jæren and to some Anthrosols in the Baltic Sea region. The AMS ^{14}C dating and the complementary archaeological literature implied that the soils in Jæren and Karmøy have been formed between the Roman Iron Age (500 BC to AD 500) and the Viking Age (about AD 800 to AD 1,000). The BC contents of the soils at Jæren deliver clear evidence for inputs of combustion residues from ancient fire management and/or from settlements. We assume that fire did not play a significant role for the formation of the humus-rich topsoils in Karmøy. The δ^{34}S data support the hypothesis that marine biomass was incorporated into the soils in Karmøy and in Jæren, corresponding to Anthrosols in the Baltic Sea region.

Since the soils at in Jæren have been formed by plaggen management (Schnepel et al., 2012) we can assume that dried marine biomass such as seaweed or kelp also has been used as alternative bedding material in stables before having been incorporating into these soils in analogy to sods, straw and/or turf. Based on the obvious anthropogenic formation of the humus-rich topsoils in Karmøy, we propose to classify the soils at Hillesland and at Feøy as Umbric Anthrosols and at Sandhåland as Hortic Anthrosol. To reconstruct which other organic materials had been applied to the soils, biomarkers for animal and/or human faeces as well as other organic material (e.g. peat, plant) should be identified and determined to shed more light on the manure material the soils received.

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References

Acksel, A., Amelung, W., Kühn, P., Gehrt, E., Regier, T., Leinweber, P., 2016. Soil organic matter characteristics as indicator of Chernozem genesis in the Baltic Sea region. Geoderma Reg. 7, 187–200. doi:10.1016/j.geodrs.2016.04.001

Acksel, A., Kappenberg, A., Kühn, P., Leinweber, P., 2017. Human activity formed deep, dark topsoils around the Baltic Sea. Geoderma Reg. 10, 93–101. http://dx.doi.org/10.1016/j.geodrs.2017.05.005

Andersen, B.G., Sejrup, H.P. and Kirkhus, Ø., 1983. Eemian and Weichselian deposits at Bø on Karmøy, SW Norway. A preliminary report. Norsk geologisk Undersøkelse 380, 189-201.

Andersen, B.G., Nydal, R., Wangen, O.P. and Østmo, S.R., 1981. Weichselian before 15,000 years B.P. at Jæren–Karmøy in southwestern Norway. Boreas 10, 297-314.

Austad, I., Øye, I., Andersen, R., Auestad, I., Domaas, S.-T., Gjertland, B., Hauge, L., Koren, E., Kvamme, M., Norderhaug, A., Timberlid, A., 2001. Den Tradisjonelle Vestlandsgården Som Kulturbiologisk System, in Skar, B.: Kulturminner og miljø. Forskning i et grenseland mellom natur og kultur. Norsk Institutt for Kulturminneforskning, Oslo, Norway, pp. 135–205.

Bartlett, R., Bottrell, S., and Coulson, J., 2005. Behaviour of sulphur during diagenesis of a maritime ombrotrophic peat from Yell, Shetland Islands, UK. Applied geochemistry, 20(8), 1597-1605.

Bird, M.I., Cali, J. a, 1998. A million-year record of fire in sub-Saharan Africa. Nature

394, 767–769. doi:10.1038/29507

Blume, H.P., Leinweber, P., 2004. Plaggen soils: Landscape history, properties, and classification. J. Plant Nutr. Soil Sci. 167, 319–327. doi:10.1002/jpln.200420905

Blume, H.-P., Stahr, K., Leinweber, P., 2011. Bodenkundliches Praktikum. Eine Einführung in pedologisches Arbeiten für Ökologen, Land- und Forstwirte, Geo- und Umweltwissenschaftler. 3. Auflage, Spektrum Akademischer Verlag, Heidelberg. ISBN: 978-3-8274-2733-5

Boese, J., Osanna, A., Jacobsen, C., Kirz, J., 1997. Carbon edge XANES spectroscopy of amino acids and peptides. J. Electron. Spectrosc. Relat. Phenom. 85(1), 9-15.

Böttcher, M.E., Brumsack, H.-J., Dürselen, C.-D., 2007. The isotopic composition of modern seawater sulfate: I. Coastal waters with special regard to the North Sea. J. Mar. Syst. 67, 73–82. doi:10.1016/j.jmarsys.2006.09.006

Brodowski, S., Rodionov, a., Haumaier, L., Glaser, B., Amelung, W., 2005. Revised black carbon assessment using benzene polycarboxylic acids. Org. Geochem. 36, 1299–1310. doi:10.1016/j.orggeochem.2005.03.011

Brodowski, S., Amelung, W., Haumaier, L., Zech, W., 2007. Black carbon contribution to stable humus in German arable soils. Geoderma 139, 220–228. doi:10.1016/j.geoderma.2007.02.004

Bronk Ramsey, C., 2009. Bayesian Analysis of Radiocarbon Dates. Radiocarbon 51, 337–360.

Bull, I. D., Simpson, I. A., Dockrill, S. J., Evershed, R. P., 1999. Organic geochemical evidence for the origin of ancient anthropogenic soil deposits at Tofts Ness, Sanday, Orkney. Org Geochem. 30(7), 535-556.

Chapman, V.J., 1970. Seaweeds and their uses, 2nd edn., Methuen, London, 304.

Chukhrov, F. V., Ermilova, L.P., Churikov, V.S., Nosik, L.P., 1979. The isotopic composition of plant sulfur. Org. Geochem. 2, 69–75. doi:10.1016/0146-6380(80)90022-4

Cody, G.D., Ade, H., Wirick, S., Mitchell, G.D., Davis, A., 1998. Determination of chemical-structural changes in vitrinite accompanying luminescence alteration using C-NEXAFS analysis. Org. Geochem. 28, 441–455. doi:10.1016/S0146-6380(98)00010-2

Conry, M. J., 1971. Irish plaggen soils—their distribution, origin and properties. Eur. J. Soil Sci. 22, 401–416.

Cooney, R. R., and Urquhart, S. G., 2004. Chemical trends in the near-edge X-ray absorption fine structure of monosubstituted and para-bisubstituted benzenes. J. of Phys. Chem. B 108(47), 18185-18191.

Davidson, D.A., Simpson, I.A., 1984. The formation of deep topsoils in Orkney. Earth Surf. Process. Landforms 9, 75–81. doi:10.1002/esp.3290090108

Dhez, O., Ade, H., Urquhart, S.G., 2003. Calibrated NEXAFS spectra of some common polymers. J. Electron. Spectrosc. Relat. Phenom. 128, 85–96. doi:10.1016/S0368-2048(02)00237-2

Eriksen, J., 2009. Soil sulfur cycling in temperate agricultural systems, Doktorthesis, Faculty of agricultural Sciences, Aarhus University, 15 – 21.

Eriksen, J., 1996. Incorporation of S into soil organic matter in the field as determined by the natural abundance of stable S isotopes. Biol. Fertil. Soils 22, 149–155.Francis, J.T., Hitchcock, a P., 1992. Inner-shell spectroscopy of para-benzoquinone, hydroquinone, and phenol - distinguishing quinoid and benzenoid structures. J. Phys. Chem. 96, 6598–6610. doi:10.1021/j100195a018.

Frazer, R., 1807. Statistical Survey of the County of Wexford. Dublin.

Freney, J.R. and Williams, C.H., 1983. The sulfur cycle in soil. In: M.B. Ivanov and J.R. Freney (ed), Glob. Biogeochem. Sulfur Cycle, No. 19, 139–201. John Wiley and Sons, New York.

Gerlach, R., Fischer, P., Eckmeier, E., Hilgers, A., 2012. Buried dark soil horizons and archaeological features in the Neolithic settlement region of the Lower Rhine area, NW Germany: Formation, geochemistry and chronostratigraphy. Quat. Int. 265, 191–204. doi:10.1016/j.quaint.2011.10.007

Gerlach, R., Baumewerd-Schmidt, H., van den Borg, K., Eckmeier, E., Schmidt, M.W.I., 2006. Prehistoric alteration of soil in the Lower Rhine Basin, Northwest Germany-archaeological, ^{14}C and geochemical evidence. Geoderma 136, 38–50. doi:10.1016/j.geoderma.2006.01.011

Giani, L., Makowsky, L., Mueller, K., 2014. Plaggic Anthrosol: Soil of the Year 2013 in Germany: An overview on its formation, distribution, classification, soil function and threats. J. Plant Nutr. Soil Sci. 177(3), 320-329.

Gilet-Blein, N., Marien, G., Evin, J., 1980. Unreliability of 14 C dates from organic matter of soils. Radiocarbon, 22(3), 919-929.

Gillespie, A.W., Walley, F.L., Farrell, R.E., Leinweber, P., Eckhardt, K.-U., Regier, T.Z., Blyth, R.I.R., 2011. XANES and pyrolysis-FIMS evidence of organic matter composition in a hummocky landscape. Soil Sci. Soc. Am. J. 75, 1741. doi:10.2136/sssaj2010.0279

Glaser, B. and Amelung, W., 2003. Pyrogenic carbon in native grassland soils along a climosequence in North America. Global Biogeochem. Cycles 17, 1–8. doi:10.1029/2002GB002019

Glaser, B., Balashov, E., Haumaier, L., Guggenberger, G., Zech, W., 2000. Black carbon in density fractions of anthropogenic soils\rof the Brazilian Amazon region.

Org. Geochem. 31, 669–678. doi:10.1016/S0146-6380(00)00044-9

Glaser, B., Haumaier, L., Guggenberger, G., Zech, W., 1998. Black carbon in soils: The use of benzenecarboxylic acids as specific markers. Org. Geochem. 29, 811–819. doi:10.1016/S0146-6380(98)00194-6

Goldberg, E.D., 1985. Black Carbon in the Environment. Wiley, New York.

Gordon, M. L., Cooper, G., Morin, C., Araki, T., Turci, C. C., Kaznatcheev, K., Hitchcock, A. P., 2003. Inner-shell excitation spectroscopy of the peptide bond: Comparison of the C 1s, N 1s, and O 1s spectra of glycine, glycyl-glycine, and glycyl-glycyl-glycine. J. Phys. Chem. A 107(32), 6144-6159.

Gottlich K., 1990. Moor- und Torfkunde. E. Schweizerbart’sche Verlagsbuchhandlung, Stuttgart.

Gravenhorst, G., 1978. Maritime sulfate over the North Atlantic. In Sulfur in the Atmosphere, 707-713.

Gump, D.J., Briner, J.P., Mangerud, J. and Svendsen J.I., 2017. Deglaciation of Boknafjorden, south-wethern Norway. Journal of Quarternary Science 32, 80-90.

Hansson, K. M., Samuelsson, J., Tullin, C., Åmand, L. E., 200). Formation of HNCO, HCN, and NH 3 from the pyrolysis of bark and nitrogen-containing model compounds. Combus. Flame 137(3), 265-277.

Heilmann, S., 2012. Auswirkungen eines erhöhten Schwefelversorgungszustandes auf Futterleguminosenbestände unter langjähriger ökologischer Bewirtschaftung am Beispiel des Lehr- und Versuchsbetriebs Gladbacherhof, Masterthesis, Faculty of agricultural sciences, Justus-Liebig-University Gießen.

Hernæs, P., 1997. Karmøys historie – som det stiger frem. Bind 1: Fra istid til 1050. Karmøy Kommune, Kopervik.

Hitchcock, A.P. and Mancini, D.C., 1994. Gas Phase Core Excitation Database (online). http://unicorn.mcmaster.ca/corex/cedb-title.html.

Hitchcock, A.P., Ishii, I., 1987. Carbon K-shell excitation spectra of linear and branched alkanes. J. Electron. Spectrosc. Relat. Phenom. 42, 11–26. doi:10.1016/0368-2048(87)85002-8

Hitchcock, A.P., Newbury, D.C., Ishii, I., Stöhr, J., Horsley, J.A, Redwing, R.D., Johnson, A.L., Sette, F., 1986. Carbon K-shell excitation of gaseou s and condensed cyclic hydrocarbons: C_3H_6, C_4H_8, C_5H_8, C_5H_{10}, C_6H_{10}, C_6H_{12}, and C_8H_8. J. Chem. Phys. 85, 4849–4862. doi:10.1063/1.451719

Holliday, V.T., Gartner, W.G., 2007. Methods of soil P analysis in archaeology. Journal of Archaeological Science 34, 301–333.

Hubbe, A., Chertov, O., Kalinina, O., Nadporozhskaya, M., Tolksdorf-Lienemann, E., Giani, L., 2007. Evidence of plaggen soils in European North Russia (Arkhangelsk region). J. Plant Nutr. Soil Sci. 170(3), 329-334.

Ishii, I., Hitchcook, A.P., 1988. The oscillator strengths for C1s and O1s excitation of some saturated and unsaturated organic alcohols, acids and esters. J. Electron. Spectrosc. Relat. Phenom. 46, 55–84. doi:10.1016/0368-2048(88)80005-7

IUSS Working Group WRB, 2014. World reference base for soil resources 2014. International soil classification system for naming soils and creating legends for soil maps, World Soil Resources Reports No. 106.

Jaulneau, V., Lafitte, C., Jacquet, C., Fournier, S., Salamagne, S., Briand, X., Esquerré-Tugayé, M.-T., Dumas, B., 2010. Ulvan, a sulfated polysaccharide from green algae, Activates plant immunity through the jasmonic acid signaling Pathway. J. Biomed. Biotechnol. 2010, 1–11. doi:10.1155/2010/525291

Jenkinson, D. S., and Johnston, A. E., 1977. Soil organic matter in the Hoosfield continuous barley experiment. Rep Rothamsted Exp Stn.

Kaland, P.E., 1986. The origin and management of Norwegian coastal heaths as reflected by pollen analysis. In: Behre KE (ed) Anthropogenic indicators in pollen diagrams, Balkema, Rotterdam. 19-36.

Kalinina, O., Chertov, O., Nadporozhskaya, M., Giani, L., 2009. Properties of soil organic matter of plaggic anthrosols from Northwest Germany, Northwest and North Russia. Arch. Agron. Soil Sci. 55(5), 477-492.

Kertesz, M. A., 2004. The role of soil microbes in plant sulphur nutrition, in: Sulphur Metabolism in Plants Special Issue, J. Exp. Bot. 55, 1939 -1945.

Kiersch, K., Kruse, J., Eckhardt, K. U., Fendt, A., Streibel, T., Zimmermann, R., ... Leinweber, P., 2012a. Impact of grassland burning on soil organic matter as revealed by a synchrotron-and pyrolysis–mass spectrometry-based multi-methodological approach. Org geochem, 44, 8-20.

Kiersch, K., Kruse, J., Regier, T. Z., & Leinweber, P., 2012b. Temperature resolved alteration of soil organic matter composition during laboratory heating as revealed by C and N XANES spectroscopy and Py-FIMS. Thermochim acta, 537, 36-43.

Kim, C.S., Bloom, N.S., Rytuba, J.J., Brown, G.E., 2003. Mercury speciation by X-ray absorption fine structure spectroscopy and sequential chemical extractions: a comparison of speciation methods. Environ. Sci. Technol. 37, 5102–5108. doi: 10.1021/es0341485

Kleber, M., Röner, J., Chenu, C., Glaser, B., Knicker, H., Jahn, R., 2003. Prehistoric alteration of soil properties in a Central German Chernozemic Soil. Soil Sci. 168, 292–306. doi:10.1097/01.ss.0000064892.94869.3a

Kortleven, J., 1963. Quantitative aspects of humus accumulation and decomposition. Verslagen Landbouwkundige Onderzoekingen, 69(1).

Krouse, H.R., Stewart, J.W.B., Grinenko, V.A., 1991. Pedosphere and biosphere. In: Krouse GR, Grinenko VA (eds) Stable isotopes: Natural and anthropogenic sulphur in file environment, Scope 43, 267-306. John Wiley, New York.

Krouse, H.R. and Tabatabai, M.A., 1986. Stable S isotopes. In sulfur in Agriculture. Agronomy monograph 27 (ed. M.A. Tabatabai), 169-205. ASA-CSSA-SSSA, Madison.

Krouse, H.R., 1980. Sulphur isotopes in our environment. In: Fritz R Fontes JC (eds) Handbook of environmental isotope geochemistry. Elsevier, Amsterdam, 435-471.

Kruse, J., Eckhardt, K.U., Regier, T., Leinweber, P., 2011. TG-FTIR, LC/MS, XANES and Py-FIMS to disclose the thermal decomposition pathways and aromatic N formation during dipeptide pyrolysis in a soil matrix. J. Anal. Appl. Pyrolysis 90, 164–173. doi:10.1016/j.jaap.2010.12.002

Kusakabe, M., Rafter, T.A., Stout, J.D. and Collie, T.W., 1976. Sulphur isotopic variations in nature. 12: Isotopic ratios of sulphur extracted from some plants, soils, and related materials. N. Z. J. Sci. 19 (4), 433-440. ISSN: 0028-8365

Kvamme, M., 1982. En vegetasjonshistorisk undersøkelse av kultur- landskapets utvikling på Lurekalven, Lindås hd., Hordaland. Thesis, University of Bergen, Norway.

Lauer, F., Prost, K., Gerlach, R., Pätzold, S., Wolf, M., Urmersbach, S., Lehndorff, E., Eckmeier, E., Amelung, W., 2014. Organic fertilization and sufficient nutrient status in prehistoric agriculture? – Indications from multi-poxy analyses of archaeological topsoil relicts. PLoS ONE 9, e106244. doi:10.1371/journal.pone.0106244

Leinweber, P., Walley, F., Kruse, J., Jandl, G., Eckhardt, K.-U., Blyth, R.I.R., Regier, T., 2009. Cultivation affects soil organic nitrogen: pyrolysis-mass spectrometry and nitrogen K-edge XANES spectroscopy evidence. Soil Sci. Soc. Am. J. 73, 82. http://dx.doi.org/10.2136/sssaj2007.0444.

Lundberg, A., 2008. Changes in the Land and the regional identity of Western Norway: The case of Sandhåland, Karmøy. In: Jones, M. and Olwig, K.R., (eds.) Nordic landscapes: Region and belonging on the northern edge of Europe. U of Minnesota Press, Minneapolis & London, chapter 13, 344-371.

Lundberg, A., 1998. Karmøys flora. Biologisk mangfald i eit kystlandskap. Fagbokforlaget, Bergen.

Lundberg, A. and Handegård, T., 1996. Changes in the spatial structure and function of coastal cultural landscapes. GeoJournal 39(2), 167-178.

McDowell, R. W., and Stewart, I., 2005. Phosphorus in fresh and dry dung of grazing dairy cattle, deer, and sheep. Journal of environmental quality, 34(2), 598-607.

Mizota, C., Sasaki, A., 1996. Sulfur isotope composition of soils and fertilizers: Differences between Northern and Southern hemispheres. Geoderma 71, 77–93. doi:10.1016/0016-7061(95)00091-7

Myhre, B., 2000. The Early Viking Age in Norway. Acta Archaeol. 71, 35–47. doi:10.1034/j.1600-0390.2000.d01-4.x

Norges Geologiske Undersøkelse, 2017. Geologien i min kommune. https://www.ngu.no/emne/kartinnsyn, accessed 29.11.2017.

Novák, M., Buzek, F., Harrison, A.F., Přechová, E., Jačková, I., Fottová, D., 2003. Similarity between C, N and S stable isotope profiles in European spruce forest soils: Implications for the use of δ^{34}S as a tracer. Appl. Geochem. 18, 765–779. doi:10.1016/S0883-2927(02)00162-2

Novák, M., Bottrell, S. H., Fottová, D., Buzek, F., Groscheová, H., Žák, K., 1996. Sulfur isotope signals in forest soils of Central Europe along an air pollution gradient. Environ. Sci. technol. 30(12), 3473-3476.

Östlund, G., 1959. Isotopic composition of sulfur in precipitation and sea-water. Tellus, 11(4), 478-480.

Øvstedal, D.O., 1985. The vegetation of Lindfls and Austrheim, western Norway. Phytocoenol 13, 323-449.

Pessenda, L.C.R., Gouveia, S.E.M., Aravena, R., 2001. Radiocarbon dating of total soil organic matter and humin fraction and its comparison with ^{14}C ages of fossil charcoal. Radiocarbon 43 (2B), 595–601. doi:10.1017/S0033822200041242

Prøsch-Danielsen, L., and Simonsen, A., 2000. Palaeoecological investigations towards the reconstruction of the history of forest clearances and coastal heathlands in south-western Norway. Veg. Hist. Archaeobot. 9(4), 189-204.

Puschmann, O., Reid, S.J., Fjellstad, W., Hofsten, J. and Dramstad, W., 2004. Tilstandsbeskrivelse av norske jordbruksregioner ved bruk av statistikk. NIJOS-rapport 17/04. Norsk institutt for jord- og skogkartlegging. Ås.

Rees, C. E., Jenkins, W. J., and Monster, J., 1978. The sulphur isotopic composition of ocean water sulphate. Geochimica et Cosmochimica Acta, 42(4), 377-381.

Reimer, P., 2013. IntCal13 and Marine13 Radiocarbon Age Calibration Curves 0–50,000 Years cal BP. Radiocarbon 55, 1869–1887.

Rodionov, A., Amelung, W., Peinemann, N., Haumaier, L., Zhang, X., Kleber, M., ... Zech, W. (2010). Black carbon in grassland ecosystems of the world. Global Biogeochem. Cy. 24(3). doi:10.1029/2009GB003669

Rodionov, A., Amelung, W., Haumaier, L., Urusevskaja, I., Zech, W., 2006. Black carbon in the zonal steppe soils of Russia. J. Plant Nutr. Soil Sci. 169, 363–369.

doi:10.1002/jpln.200521813

Roffeis, C. and Corfu, F., 2014. Caledonian nappes of southern Norway and their correlation with Sveconorwegian basement domains. Corfu, F., Grasser, D. and Chew, D.M. (eds.). New Perspectives on the Caledonides of Scandinavia and Related Areas. Geological Society, London, Special Publications, 390, 193-221.

Rønneseth, O., 1974. Gard und Einfriedigung: Entwicklungsphasen d. Agrarlandschaft Jærens. Geogr. Ann., Spec. Issue Nr. 2. Stockhom. ASIN: B005OK8UHG

Samuel, N. T., Lee, C. Y., Gamble, L. J., Fischer, D. A., Castner, D. G., 2006. NEXAFS characterization of DNA components and molecular-orientation of surface-bound DNA oligomers. J. Electron. Spectrosc. Relat. Phenom. 152(3), 134-142.

Scharpenseel, H.-W., Pfeiffer, E.-M., Becker-Heidmann, P., 2014. Alter der Humusstoffe. Handbuch der Bodenkunde. 3.5:1–22.

Scharpenseel, H.-W., Tsutsuki, K., Becker-Heidmann, P., Freytag, J., 1986. Untersuchungen zur Kohlenstoffdynamik und Bioturbation von Mollisolen. J. Plant Nutr. Soil Sci. 149 (5), 582–597. doi:10.1002/jpln.19861490509

Scheffer, F., Schachtschabel, P., 2010. Lehrbuch Der Bodenkunde. 16. Auflage. Spektrum Akademischer Verlag, Heidelberg.

Schmid, E. M., Skjemstad, J. O., Glaser, B., Knicker, H., Kögel-Knabner, I., 2002. Detection of charred organic matter in soils from a Neolithic settlement in Southern Bavaria, Germany. Geoderma 107(1), 71-91.

Schmidt, M.W.I., Skjemstad, J.O., Gehrt, E., Kögel-Knabner, I., 1999. Charred organic carbon in German chernozemic soils. Eur. J. Soil Sci. 50, 351–365. doi:10.1046/j.1365-2389.1999.00236.x

Schnepel, C., Potthoff, K., Eiter, S., Giani, L., 2014. Evidence of plaggen soils in SW Norway. J. Plant Nutr. Soil Sci. 177 (4), 638–645. doi:10.1002/jpln.201400025

Schoenau, J.J. and Bettany, J.R., 1989. ^{34}S natural abundance variations in prairie and boreal forest soils. J. Soil Sci. 40, 397–413. doi:10.1111/j.1365-2389.1989.tb01283.x

Simpson, I. A., 1997. Relict properties of anthropogenic deep top soils as indicators of infield management in Marwick, West Mainland, Orkney. J. Archaeol. Sci. 24(4), 365-380.

Simpson, I.A., 1993. The chronology of anthropogenic soil formation in Orkney. Scott. Geogr. Mag. 109, 4–11. doi:10.1080/00369229318736870

Solomon, D., Lehmann, J., Kinyangi, J., Liang, B., Heymann, K., Dathe, L., ... Jacobsen, C., 2009. Carbon (1s) NEXAFS spectroscopy of biogeochemically relevant reference organic compounds. Soil Sci. Soc. Am. J. 73(6), 1817-1830.

Sølvberg, I. Ø., 1976. Driftsmåter i vestnorsk jordbruk, ca. 600–1,350. Universitets-forl., Oslo, Norway. ISBN: 8200015718.

Statistics Norway, 2017. Tabell: 09594: Arealbruk og arealressurser, etter arealklasser (km²). https://www.ssb.no/statistikkbanken, accessed 30.11.2017.

Sundve, E., 1977. Undersokelser av vegetasj onssyklus, suksesjonstendenser og jordsmonn i lyngmark [Investigations of vegetation cycles, succesion tendensies and soil in heaths]. Thesis (unpublished), University of Bergen, Norway.

Swift, M.J., Heal, O.W., Anderson, J.M., 1979. Decomposition in terrestrial ecosystems. Berkeley, California.

Tinner, W., Hubschmid, P., Wehrli, M., Ammann, B., Conedera, M., 1999. Long-term forest fire ecology and dynamics in southern Switzerland. J. Ecol. 87, 273-289. doi: 10.1046/j.1365-2745.1999.00346.x

Tcherkez, G., and Tea, I., 2013. 32S/34S isotope fractionation in plant sulphur metabolism. New Phytologist, 200(1), 44-53.

Urquhart, S. G., and Ade, H., 2002. Trends in the carbonyl core (C 1S, O 1S)→ π* C=O transition in the near-edge X-ray absorption fine structure spectra of organic molecules. J. Phys. Chem. B 106(34), 8531-8538.

Vorren, K.-D., Nilssen, E. and Mørkved, B., 1990. Age and agricultural history of the '-staðir' farms of North and Central Norway, Norsk Geografisk Tidsskrift – Norw. J. Geogr. 44:2, 79-102, doi10.1080/00291959008552247

Wakshal, E. and Nielsen, H., 1982. Variations of $\delta^{34}S$ (SO_4), $\delta^{18}O$ (H2O) and Cl/SO_4 ratio in rainwater over northern Israel, from the Mediterranean Coast to Jordan Rift Valley and Golan Heights. Earth and Planetary Science Letters, 61(2), 272-282.

Wiedner, K., Schneeweiß, J., Dippold, M. A., Glaser, B., 2014. Anthropogenic Dark Earth in Northern Germany - The Nordic Analogue to terra preta de Índio in Amazonia. Catena 132, 114-125. doi:10.1016/j.catena.2014.10.024

Wojdyr, M., 2010. Fityk: A general-purpose peak fitting program. J. Appl. Crystallogr. 43, 1126–1128. doi:10.1107/S0021889810030499

Zhao, F. J., Spiro, B., Poulton, P. R., McGrath, S. P., 1998. Use of sulfur isotope ratios to determine anthropogenic sulfur signals in a grassland ecosystem. Environ. Sci. technol. 32(15), 2288-2291.

Figure captions

Fig. 1 Locations of the humus-rich topsoils in Karmøy (Hi: Hillesland; F: Feøy; S: Sandhåland) and the Plaggic Anthrosols in Jæren (PE1: Njærheim; PE2: Byberg; PE3: Heigre; PE4: Årsvoll) in SW Norway.

Fig. 2

Soil profiles of the humus-rich topsoils (a) on Feøy (F) and (b) at Sandhåland (S) in Karmøy.

Fig. 3

Score plot of the Partial least squares discriminant analysis (PLS-DA) of the relative abundance of 10 important compound classes from Py-FIMS of the humus-rich topsoils in Karmøy and the Plaggic Anthrosols in Jæren (Table 3).

Fig. 4.

Normalized stacked carbon *K*-edge XANES spectra of the humus-rich topsoils in Karmøy and the Plaggic Anthrosols in Jæren (average spectra of the profiles PE1; PE2; PE3; PE4). The fitted features (a) = 284.0 eV; (b) = 285.0; (c) = 285.6 eV; (d) = 285.9 eV, (e) = 286.2 eV, (f) = 286.5 eV, (g) = 286.8 eV, (h) = 287.2 eV, (i) = 288.0 eV, (j) = 288.7 eV, (k) = 289.5 eV. The table shows the portions of peak heights.

Fig. 5 Score plot of the Partial least squares discriminant analysis (PLS-DA) of peak heights from the normalized stacked carbon *K*-edge XANES spectra of the humus-rich topsoils in Karmøy and the Plaggic Anthrosols in Jæren. The feature (a) = 284.0 eV; (b) = 285.0; (c) = 285.6 eV; (d) = 285.9 eV, (e) = 286.2 eV, (f) = 286.5 eV, (g) = 286.8 eV, (h) = 287.2 eV, (i) = 288.0 eV, (j) = 288.7 eV, (k) = 289.5 eV.

Fig. 6

Concentrations of black carbon (BC) of the humus-rich topsoils in Karmøy and the Plaggic Anthrosols in Jæren, in comparison to the BC concentrations from various soils (n = 94). The BC concentration was calculated from BPCA-C content multiplied with a conversion factor of 2.27 (Glaser et al., 1998). This factor provides a conservative minimum estimate of BC (Brodowski et al., 2005). The Letters show significant similarities between the soils ($P < 0.05$). [a] Acksel et al. (2016, 2017), [b] Gerlach et al. (2006, 2012), [c] Glaser et al. (1998), [d] Glaser and Amelung (2003), [e] Lauer et al. (2014), [f] Rodionov et al. (2010), [g] Schmid et al. (2002), [h] Schmidt et al. (1999), [i] Wiedner et al. (2014).

Fig. 7 ^{34}S-values of the humus-rich topsoils in Karmøy and the Plaggic Anthrosols in Jæren in comparison to the ^{34}S-values from marine and terrestrial materials and from various terrestrial soils. The zero point of the scale is the troilite from the Canyon Diablo meteorite (Thode, 1963) and the terrestrial mean. The letters denote the significant differences between the ^{34}S-values of the SOM and the organic matter ($P < 0.05$). [a] Böttcher et al. (2007), [b] Chukhrov et al. (1979), [c] Östlund (1959), [d] Gravenhorst (1978), [e] Erikson (1996), , [f] Mizota and Sasaki (1996), [g] Novak et al. (1996, 2003), [h] Zhao et al. (1998), [i] Krouse (1980), [j] Krouse and Tabatabai (1986), [k] Schoenau and Bettany (1989), [l] Acksel et al. (2017), [m] Kusakabe et al., 1976. ^{34}S-values of soils near 0‰ are considered to approximate the terrestrial mean (Krouse et al., 1991).

***Highlights (for review)**

Highlights

- The humus-rich topsoils at Karmøy in SW Norway were classified as Anthrosols.
- The soils at Karmøy and Jæren have formed between 500 BC and AD 1,000.
- Combustion residues were incorporated into the plaggic soils at Jæren.
- Marine biomass was used as soil amendment in the soils at Karmøy and Jæren.

Tab. 1 Chemical properties of the humus-rich topsoils in Karmøy compared to the Plaggic Anthrosols in Jæren (average data for the profiles PE1; PE2; PE3; PE4 from Schnepel et al., 2014). Significant differences in the soil parameters between the upper and underlying horizons are marked with * $P < 0.05$; ** $P < 0.01$ and *** $P < 0.001$. The underlined soil parameters of the soils in Karmøy indicate significant differences ($P < 0.05$) to the upper horizons and bold marked values to the underlying horizon of the soils in Jæren. ^{a}The C_t-stock was calculated for each horizon and summed for the whole profiles.

Site	Depth	pH	$CaCO_3$	C_{org}	C_t-stock		N_t	C/N	Exchangeable cations					CEC	BS	P_t
									Ca^{2+}	Mg^{2+}	Na^{+}	K^{+}	Σ			
		$CaCl_2$	g kg^{-1}	%	kg m^{-2}		%		cmol$_c$ kg^{-1}						%	mg kg^{-1}
Karmøy																
Hi	0-30	**4.2**	n.d.	**6.5**	16.5	^{a}23	**0.414**	**14**	12.2	1.9	**0.4**	**1.0**	**15.3**	**59.5**	26	**3,157.9**
	30-45	**4.7***	n.d.	3.2*	6.4		**0.186*****	**14**	10.8	1.1	**0.4**	0.3	12.5	51.6	26	2,085.3**
F	0-30	**5.0**	n.d.	**6.2**	12.2	^{a}17	**0.403**	**13**	**18.9**	2.1	**0.6**	0.3	**21.8**	**83.5**	26	**3,484.1**
	30-45	**4.6***	n.d.	4.9	4.4		**0.388***	**11**	11.2	0.8	**0.4**	0.1	12.5	46.9*	27	**3,315.7**
S	0-30	**7.4**	216.7	1.5	5.7	^{a}7	**0.079**	**19**	**24.5**	1.0	0.3	0.2	**26.0**	25.9	**97**	**764.7**
	30-45	**7.6**	299.3	0.5*	1.0*		0.025***	**19**	12.8*	0.5	0.2	0.1	13.6*	11.5	**100**	**407.4***
Jæren (Schnepel et al., 2014)																
PE1-PE4	0-30	5.4	n.d.	4.1	12.9	^{a}21	0.188	22	7.9	0.9	0.2	0.3	9.2	22.9	42	2,431.4
	30-60	5.3	n.d.	2.8**	7.9**		0.100**	28*	5.1	0.5	0.1	0.2	5.8	21.2	29	2,224.4

Table 2

Tab. 2 Physical properties of the humus-rich topsoils in Karmøy compared to the Plaggic Anthrosols in Jæren (average data of the profiles PE1; PE2; PE3; PE4 from Schnepel et al., 2014). Significant differences in soil parameters between upper and underlying horizons are marked with * $P < 0.05$; ** $P < 0.01$ and *** $P < 0.001$. The underlined soil parameters of the soils in Karmøy indicate significant differences ($P < 0.05$) to the upper horizons, and bold marked values to the underlying horizon of the soils in Jæren.

			Texture									
			Sand				Silt					
Site	Depth	Bulk density	Coarse[a]	Medium[b]	Fine[c]	Total	Coarse[d]	Medium+ Fine[e]	Total	Clay[f]	L*-Value	Munsel
		g cm^{-3}	%								(dry)	
Karmøy												
Hi	0-30	**0.9**	14	**12**	**17**	**42**	**17**	**28**	**45**	**13**	37.94	5Y 4-1
	30-45	**0.9**	25	**12**	**15**	**52****	**16**	**24***	**39****	**9***	42.69*	5Y 4-2
F	0-30	**0.7**	19	**17**	**14**	**49**	**16**	**27**	**43**	8	36.31	5Y 3-1
	30-45	**0.6**	25	**15****	**13**	**53**	15*	25	**40**	8	37.27	5Y 4-2
S	0-30	1.3	**4**	**71**	**18**	**94**	**2**	**2**	**4**	**2**	42.81	5Y 4-1
	30-45	1.3	**6***	**72**	**17**	**96**	**1**	**2***	**3**	**1****	**46.92**	5Y 5-1
Jæren (Schnepel et al., 2014)												
PE1-PE4	0-30	1.1	13	27	28	68	13	12	25	8	37.77	5Y 4-2
	30-60	1.1	17	25	27	69*	13	12	24	7	39.49**	5Y 4-2

[a] (0.63-2.0 mm); [b] (0.2-0.63 mm); [c] (0.063-0.2 mm); [d] (0.02-0.063 mm); [e] medium (0.0063-0.02 mm) + fine silt (0.002-0.0063 mm); [f] clay total (<0.002 mm)

Tab. 3 Relative abundance of 10 important compound classes of the soil organic matter in the horizons of the humus-rich topsoils in Karmøy and the Plaggic Anthrosols in Jæren (average data of the profiles PE1; PE2; PE3; PE4). Volatilized matter = VM; total ion intensity = TII; carbohydrates = CHYD, phenols + lignin-monomers = PHLM, lignin dimers = LDIM, lipids = LIPID, alkylaromatics = ALKYL, heterocyclic N containing compounds = NCOMP, sterols = STEROL, peptides = PEPTI, suberin = SUBER, free fatty acids = FATTY. Significant differences in the VM, TII and the proportions of compound classes between the upper and underlying horizons are marked with * $P < 0.05$; ** $P < 0.01$ and *** $P < 0.001$. The underlined values of the soils in Karmøy indicate significant differences ($P < 0.05$) to the upper horizons and bold marked values to the underlying horizon of the soils in Jæren.

Site	Depth	VM	TII	Proportions of compound classes in %TII									
		(%)	(10^6 counts mg^{-1})	CHYDR	PHLM	LDIM	LIPID	ALKYL	NCOMP	STEROL	PEPTI	SUBER	FATTY
Karmøy													
Hi	0-30	16.6	**372.4**	**1.1**	**2.1**	3.7	**6.5**	**5.5**	**0.7**	**7.0**	**1.8**	**1.0**	**4.1**
	30-45	12.4	229.0***	**1.0**	**3.1*****	4.5***	**8.2*****	8.9***	**0.7**	4.8***	**2.2*****	0.4***	2.0**
F	0-30	19.5	249.1	**1.1**	**2.2**	3.7	**6.2**	**5.5**	**0.8**	**6.3**	**2.0**	**1.1**	**3.0**
	30-45	12.9	**333.2***	**1.0***	**2.6***	**5.0****	7.6**	**7.8****	**0.6****	5.1*	**2.1**	0.5**	1.6*
S	0-30	14.2	**82.1**	**1.7**	4.2	**3.1**	**6.5**	8.1	1.1	4.1	2.8	0.5	2.2
	30-45	9.7	**33.6*****	**1.4***	**3.7**	**2.6*****	**6.8***	8.5	**1.0**	3.8*	**2.3****	0.4	1.8**
Jæren													
PE1-PE4	0-30	16.3	346.7	2.5	4.3	3.8	6.0	7.5	1.3	5.0	3.1	0.6	3.6
	30-60	10.4**	261.7***	2.4*	4.7	4.2***	7.2**	9.2**	1.2*	4.3	3.1	0.4*	2.1**

Table 4

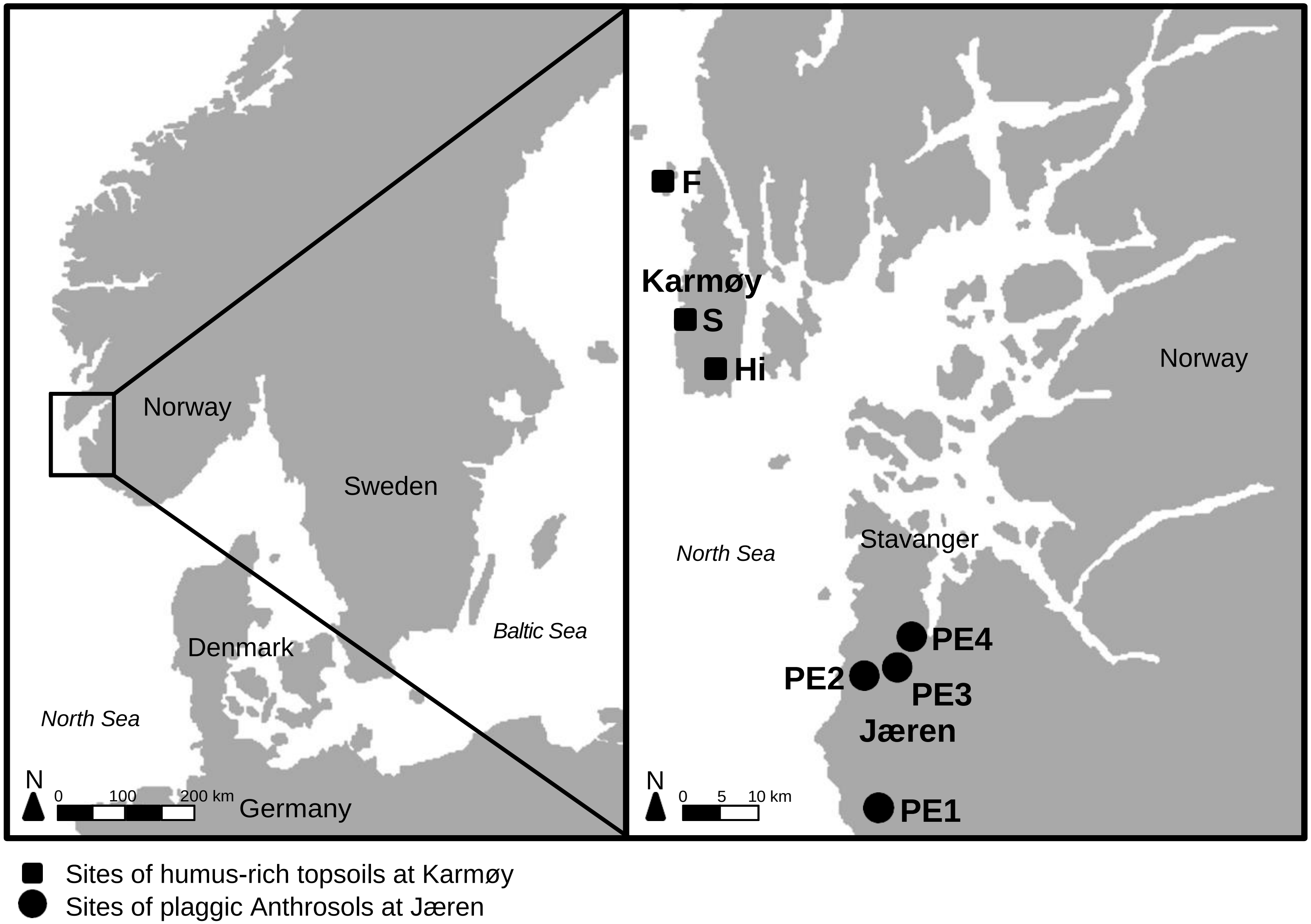
Tab. 4 Organic C-concentrations of the humin fraction (C_{humin}) and its C-proportions (in parenthesis), and the ^{14}C ages of the humin fraction of the humus-rich topsoils in Karmøy and the Plaggic Anthrosols in Jæren; calibrated with OxCal v4.1.7 using the IntCal13 calibration curve (Bronk Ramsey, 2009; Reimer, 2013).

Site	Depth	Sample no.	Lab code	Material	C_{humin} (g kg^{-1}) (% of soil C_{org})	cal AD (2 sigma)	Conventional age (BP)	
Karmøy								
Hi	30-45	N7-Hill	15049	humin fraction	13.7 (43)	980 – 1,040	1,010	± 20
F	30-45	N8-F	15050	humin fraction	17.8 (36)	1,260 – 1,300	722	± 19
S	30-45	N6-S2	15052	humin fraction	4.2 (49)	970 – 1,030	1,040	± 20
Jæren								
PE1	45-60	N4-Nj	15048	humin fraction	10.9 (36)	430 – 580	1,542	± 20
PE2	30-45	N2-By	15046	humin fraction	10.4 (40)	1,220 – 1,280	764	± 19
PE3	45-60	N3-Hei	15047	humin fraction	10.0 (36)	1,030 – 1,190	910	± 20
PE4	45-60	N1-Ar	15045	humin fraction	11.8 (37)	860 – 980	1139	± 19

Tab. 5 Diagnostic horizon properties of humus-rich soils according to the WRB (2014) in comparison to the soil properties determined for the soils in Karmøy.

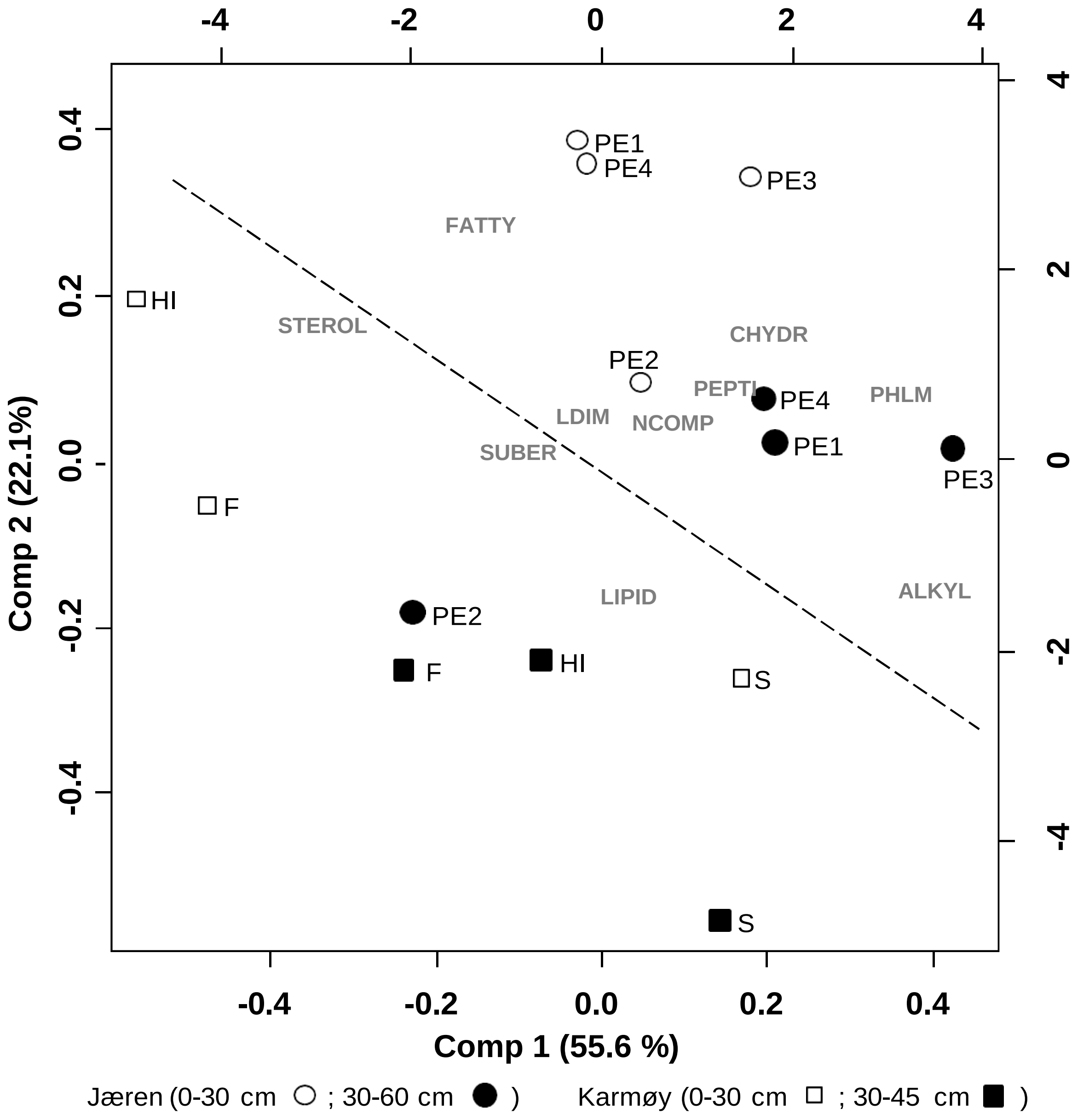
Profile	Thickness (cm)	Value (Munsell)	C_{org} (%)	BS (%)	Bioturbation	Raised land
Criteria for humus-rich horizons according to the WRB (2014)						
terric	> 20	source material	> 1	> 50	Possibly high	Yes
hortic	> 20	3 (moist)	> 1	> 50	> 25 % (Vol)	No
plaggic	> 20	5 (dry)	> 0.6	< 50	No	Yes
pretic	> 20	4 (moist)	> 1	-	< 25 % (Vol)	No
mollic	> 20	5 (dry)	> 0.6	> 50	high	No
umbric	> 20	< 5 (dry)	> 0.6	< 50	high	No
chernic	> 25	5 (dry)	> 1	> 50	high	No
Overview of the soil properties of the soils in Karmøy						
Hi	30	4 (dry)	6.5	26	high	No
F	30	3 (dry)	6.2	26	high	No
S	30	4 (dry)	1.5	97	high	No

Figure 1



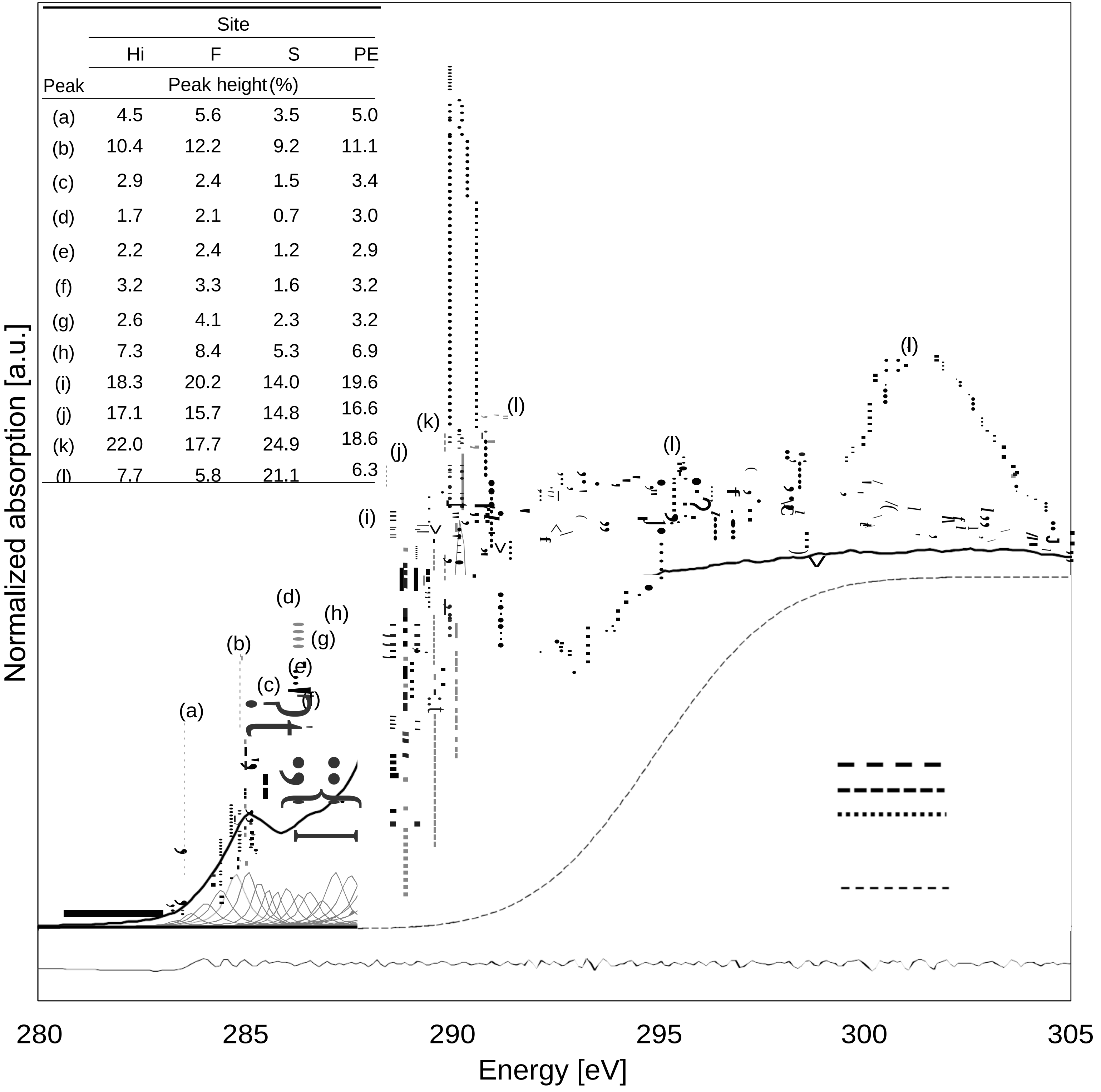
Acksel et al., Figure 1

Acksel et al., Figure 2



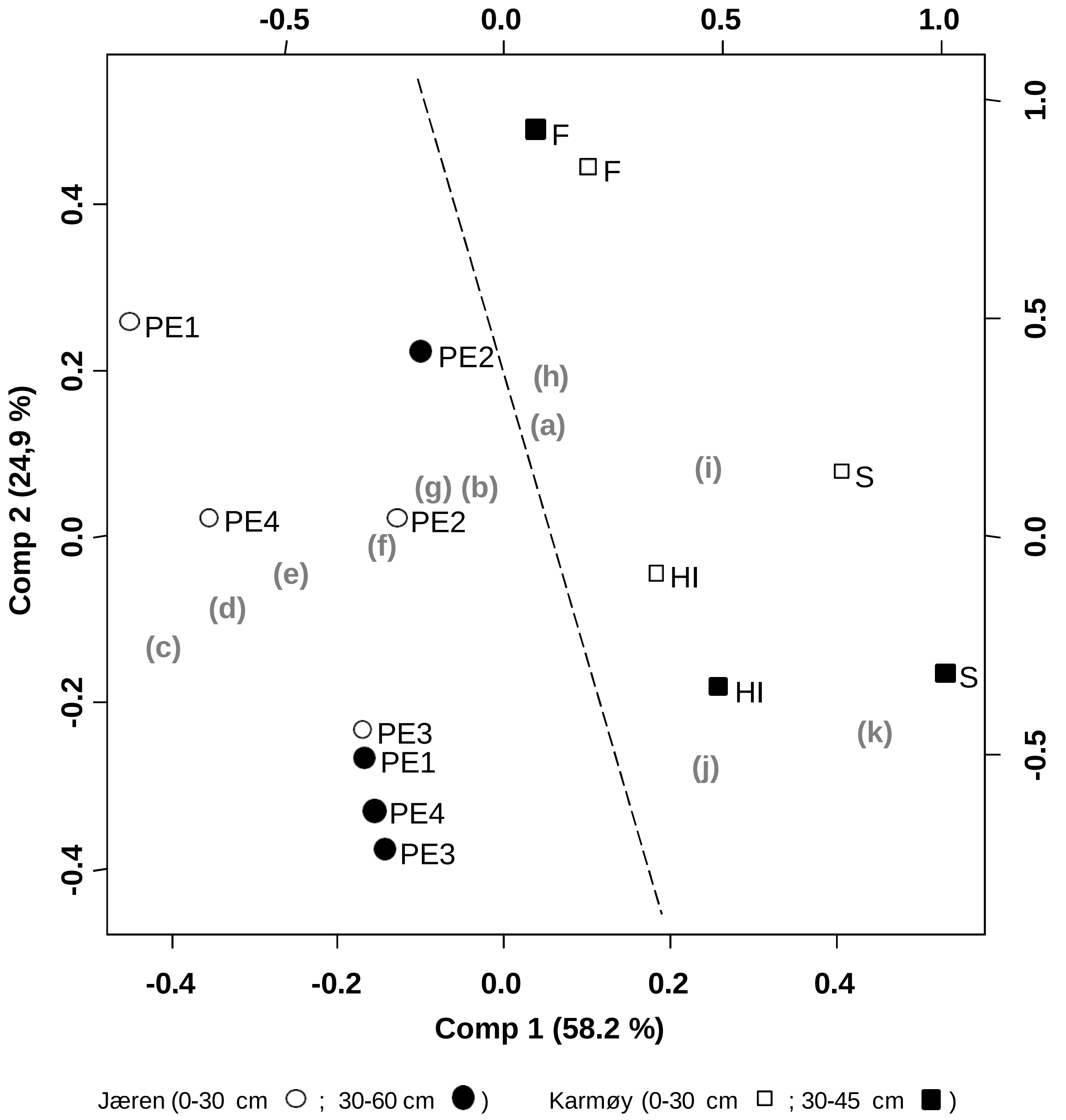
Acksel et al., Figure 3

Figure 4

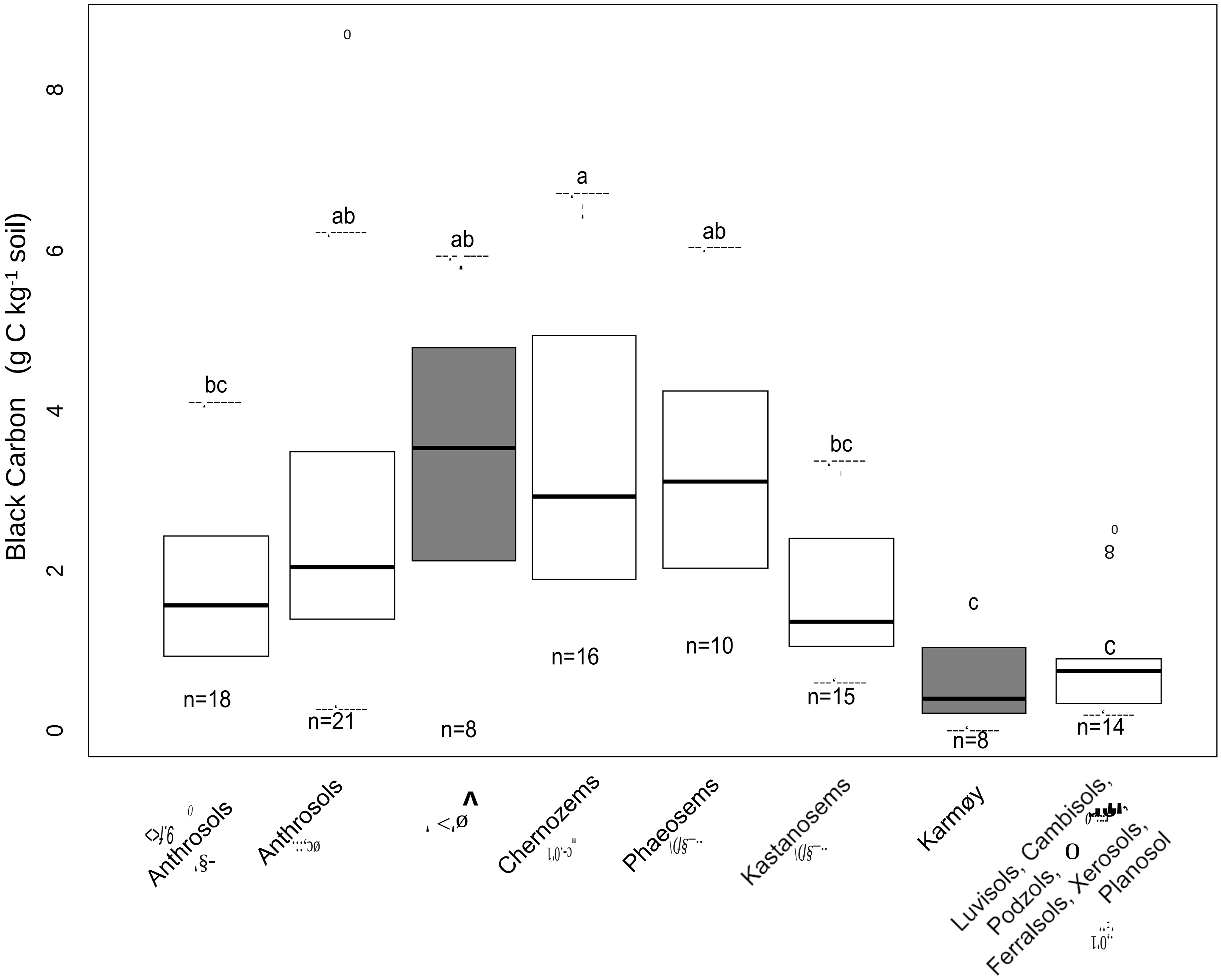


	Site			
	Hi	F	S	PE
Peak	Peak height (%)			
(a)	4.5	5.6	3.5	5.0
(b)	10.4	12.2	9.2	11.1
(c)	2.9	2.4	1.5	3.4
(d)	1.7	2.1	0.7	3.0
(e)	2.2	2.4	1.2	2.9
(f)	3.2	3.3	1.6	3.2
(g)	2.6	4.1	2.3	3.2
(h)	7.3	8.4	5.3	6.9
(i)	18.3	20.2	14.0	19.6
(j)	17.1	15.7	14.8	16.6
(k)	22.0	17.7	24.9	18.6
(l)	7.7	5.8	21.1	6.3

Figure 5

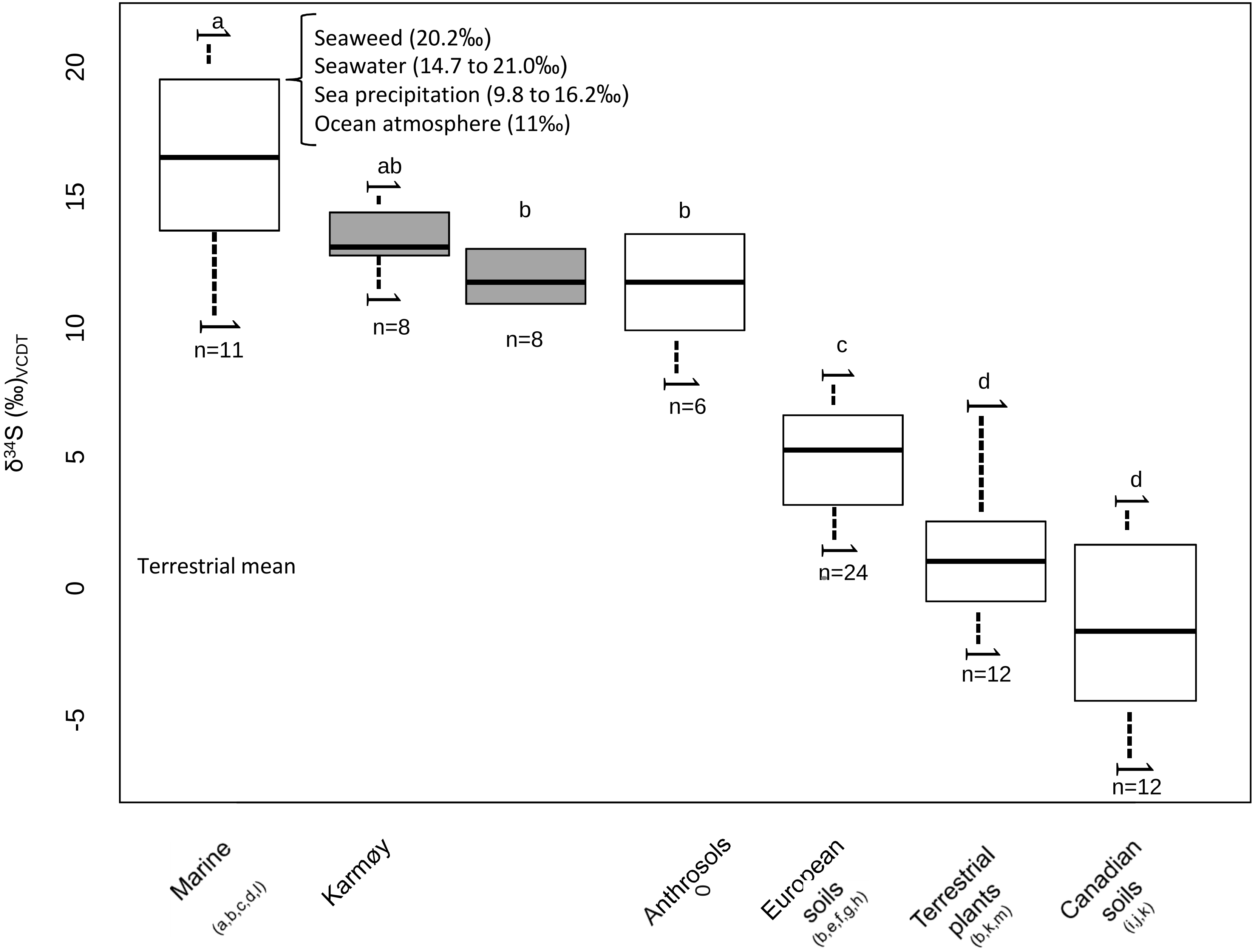


Acksel et al., Figure 5



Acksel et al., Figure 6

Figure 7



Acksel et al., Figure 7