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Analysis of peat soil organic carbon, total nitrogen, soil water content and basal respiration: Is there a 'best' drying temperature?

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ABSTRACT

Soil needs to be dried in order to determine water content, soil organic carbon content (SOC) and total nitrogen content (N). Water content is commonly measured using standard methods that involve drying temperatures of 105–110 °C. Recommended drying temperatures differ for the determination of SOC and N. However, at moderate drying temperatures, microbial activity might lead to organic matter mineralisation and nitrification, and thus to an underestimation of SOC and N. Furthermore, low drying temperatures might not dewater soils sufficiently to correctly determine water content or bulk density. Chemical processes such as thermal decomposition and volatilisation might occur at higher temperatures. This raises the question of whether the same sample can be used to determine water content, SOC and N. Further, the effect of drying, especially at different temperatures, on basal respiration of peat soils determined by incubation experiments is so far unknown. Effects of drying temperature might be especially severe for peat soils, which have high SOC and water contents.

This study systematically evaluated the effect of different drying temperatures (20, 40, 60, 80 and 105 °C) on the determination of mass loss (proxy for water content), SOC and N over a wide range of 15 different peat soils comprising amorphous, *Sphagnum* and sedge peat substrate. The investigated peat soils had SOC contents ranging from approximately 16.8–52.5% with different degrees of decomposition. They were thus separated into two 'peat groups' (amorphous and weakly decomposed). In a subsequent investigation, an incubation experiment was carried out on a subset of five peat soils to investigate the pre-treatment effect of different drying temperatures on basal respiration. The results showed that amorphous samples should be dried at 105 °C to determine water content. The weakly decomposed peat soils in the study had reliable water contents for drying temperatures above 60 °C. For temperatures below 80 °C, the determined SOC and N were biased by residual water. This could be corrected for weakly decomposed samples, but for amorphous samples only for drying temperatures ≥60 °C. Thus, mineralisation of soil organic matter is likely to take place at lower drying temperatures which are not recommendable especially for amorphous peat prone to high mineralisation rates. This is supported by the results of the incubation experiment: The effect of peat type (amorphous topsoil vs. weakly decomposed subsoil) was greater than the effect of different drying temperatures, which nonetheless affected respiration rates. The differences between all five soils were consistent, irrespective of the drying temperature. Thus, incubation experiments might be possible using peat dried at moderate temperatures.

1. Introduction

Soil needs to be dried in order to undertake various soil analyses, e.g. to determine water content, soil organic carbon content (SOC) and total nitrogen content (N). Water content (or soil moisture) is commonly measured with standard methods that use drying temperatures of 110 ± 5 °C (ASTM, 2019), 105–110 °C (BS, 1990), 105 ± 5 °C (DIN EN ISO 11461, 2014; DIN ISO 11465, 1993) or 100–110 °C (Gardner, 1986) to remove all pore water from the soil. For the determination of basic chemical properties (e.g. SOC and N), recommended drying temperatures differ. The *German Institute for Standardization* (DIN) advises a drying temperature below 40 °C (DIN ISO 19747, 2009). In research

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projects, however, practices differ substantially.

Several temperature-dependent physical, chemical and biological processes occur during drying (Soulides and Allison, 1961) and may influence laboratory results. It is known that drying affects soil characteristics such as nutrient extractability and soil pH (Payne and Rechcigl, 1989; van Erp et al., 2001; Erich and Hoskins, 2011), which in turn affects the determination of nutrients, e.g. potassium (Erich and Hoskins, 2011), inorganic and organic phosphorus (Turner and Haygarth, 2003; Erich and Hoskins, 2011), nitrate-N (van Erp et al., 2001) and exchangeable and fixed ammonium-N of organic and inorganic sources (Harding and Ross, 1964; Frye and Hutcheson, 1981; Wiltshire and du Preez, 1994).

Less is known about how different drying temperatures influence the results of SOC or N analyses. Microbial activity is stimulated at moderate drying temperatures (~25–40 °C) (van Erp et al., 2001), and soil-altering processes such as carbon mineralisation or nitrification occur (Birch, 1959). This could lead to an underestimation of SOC and N. At higher temperatures microbial activity decreases, but chemical processes such as thermal decomposition of protein-like components of soil organic matter (SOM) (Wiltshire and du Preez, 1994) or volatilisation of ammonia or volatile organic compounds (VOC) might occur. At 105 °C, Samuelsson et al. (2006) found losses of VOC from various biogenic materials of 0.1–2% (e.g. birch and pine bark, Miscanthus, milled peat etc.). To the authors' knowledge, no study has been undertaken on VOC losses from soils at drying temperatures of between 40 °C and <105 °C. MacFarlane and Allen (1965) and O'Kelly (2004) reported possible charring of SOM at temperatures above approximately 80–85 °C for peat and organic clay soils. In MacFarlane and Allen's study (1965), the charring effects on fibrous peat samples were observed through a magnifying glass. However, the SOM values determined were similar between drying temperatures of 20–105 °C. O'Kelly (2004) dried organic soils with increasing drying temperatures. After a temperature of 150 °C was reached, the samples were rewetted and dried again at 60 °C. The dry weight at a temperature of 60 °C in the first drying was compared with the dry weight after the second drying, and the differences attributed to the charring of SOM. However, SOC and N contents were not determined. Gardner (1986) stated that oxidation is likely to occur at temperatures above 50 °C for organic material. Marachi et al. (1983) observed burned peat fibres after oven-drying at 110 °C. Kasozi et al. (2009) subjected Histosol samples to temperatures of 180 °C followed by a cooling to ambient temperatures and found that after cooling, the samples regained the lost mass. They concluded that as the reaction had been reversible, mass loss could not be attributed to the loss of organic components.

In scientific studies, drying for the determination of SOC and/or N is performed over a wide range of temperatures, from room temperature (Bremner and Jenkinson, 1960; Evgrafova et al., 2018) to 40 °C (Poeplau et al., 2016), 60 °C (Vesterdal et al., 2002), 65 °C (Alcántara et al., 2017) or 105 °C (Torn et al., 1997). To avoid biases in soil analysis, some authors have dried a separate sample at 105 °C to determine bulk density or water content (e.g. Poeplau et al., 2016). However, drying samples at different temperatures is time-consuming and thus not always feasible. This raises the question of whether water content, SOC and N can be determined using the same sample. Either samples dried below 105 °C need to yield reliable water content and bulk density values, or samples dried at 105 °C need to yield reliable SOC and N contents. Wilson et al. (2009) did not find any statistically significant influences caused by drying temperature on bulk density values for three different mineral soils dried at 40, 70 and 105 °C. Despite the studies mentioned above, no study appears to have evaluated the effects of different drying temperatures on water content, SOC and N simultaneously with the same sample.

The physical, chemical and biological processes during drying might be enhanced for peat soils because they contain a high amount of SOC and also large porosities (up to 98%), which allow very high water contents (Dettmann et al., 2019). In contrast to the study on mineral soils by Wilson et al. (2009), lower drying temperatures might not dewater peat soils sufficiently enough to determine water content or bulk density due to the high water contents. Mineralisation or volatilisation of SOM might be enhanced by the high SOC contents of peat soils. The processes described above may explain why peat soils are dried at a variety of different temperatures for water content and bulk density determination, ranging from 60 °C (Marachi et al., 1983; Tiemeyer et al., 2017), 65 °C (Moore et al., 2017), 70 °C (Kellner and Lundin, 2001; Könönen et al., 2015), 80 °C (Dettmann et al., 2014; Mustamo et al., 2016; Bechtold et al., 2018; Säurich et al., 2019), 85 °C (Lukenbach et al., 2015; Farrish and Grigal, 1985), 86 °C (Nagare et al., 2011) and 95 °C (Farnham and Finney, 1965; Price and Whittington, 2010) to 105 °C (Holden and Ward, 1998; Oleszczuk et al., 2004; Schwärzel et al., 2006; Gnatowski et al., 2010; Rocha Campos et al., 2011; Roßkopf et al., 2015; Hewelke et al., 2016; Bourgault et al., 2018). However, the above-cited studies mostly do not provide any information about why drying is carried out at a specific temperature. It is believed that in some cases there was a trade-off between different soil analyses. However, it appears that there is no scientific basis for the temperature at which peat soils should be dried. This hampers a meta-analysis of soil physical properties, for example, or comparisons between different studies during which soil samples are dried at different temperatures.

Another important aspect is that dried samples can be archived for decades in order to perform measurement repetitions or different soil analyses in future. Especially in the case of long-term studies or large-scale inventories, drying might be inevitable for logistical and financial reasons. Subsequent analyses may also differ from the original purpose. One example is soil incubation experiments, e.g. involving archived soils from long-term studies. Soil incubation experiments are commonly performed to investigate basal soil respiration in order to determine mineralisation of SOM. This is often done with rewetted soil samples, although there are drying and re-wetting effects of this on the microbial community. Nonetheless, Meisner et al. (2013) and Jones et al. (2019) have been able to show similar respiration rates within five days of incubation for mineral soil samples that have been dry for a year or for decades respectively. Besides drying itself, drying temperature might also influence the microbial community, SOM composition and consequently respiration rates. To the authors' knowledge, there have been no studies on peat soils investigating the effects of pre-treatment (drying or even drying at different temperatures) on basal soil respiration in incubation experiments.

The aim of this study was to systematically evaluate the effect of different drying temperatures on the determination of mass loss (ML) (proxy for water content), SOC and N over a wide range of different peat soils containing amorphous, *Sphagnum* and sedge peat substrate. Analyses were performed on 15 peat soils with SOC contents ranging from approximately 16.8–52.5% and different degrees of decomposition. All the samples were dried at 20, 40, 60, 80 and 105 °C. In a second experiment, a subset of five peat soils was chosen to investigate the effect of different drying temperatures on basal respiration in incubation experiments.

2. Material and methods

Two consecutive experiments were conducted. First, the influence of drying temperatures on ML, SOC and N was determined (referred to below as the drying experiment). Second, an incubation experiment was performed to determine drying effects on basal respiration, investigating both dried and field-moist samples.

The experiments were conducted starting with field-moist samples. Fifteen different peat soils were investigated (referred to below as 'soils'). At each site, a soil profile was dug and mapped in accordance with the German soil classification system (Ad-hoc-AG Boden, 2005). The degree of decomposition was determined according to the von Post scale, which is based on the consistency of plant remains and soil water colour (von Post, 1922). All samples were taken from specific horizons

in the soil profiles and stored in plastic bags. After sampling, the samples were frozen until analysis. The basic soil properties are listed in the appendix (Table A.1). Porosities and bulk densities were determined from six separate intact samples which were taken from the same horizons as the samples in the plastic bags. Therefore, six steel cylinders with a sharpened bottom edge and a volume of 244.29 cm^3 (height: 6 cm, diameter: 7.2 cm) were carefully inserted vertically into the soil. Then, the whole sample was excavated and checked for any signs of compression, damage or edge effects. When in doubt, sampling was repeated.

In the laboratory, the bottoms of the samples were covered by nylon gauzes and samples were saturated slowly from the bottom for at least 24 h. Afterwards, samples were dried at 80 °C for 14 days. Porosities and bulk densities were determined by standard mass calculations. We did not account for shrinkage of the samples and assumed soil volume to be constant. Porosity (equal to saturated water content) was determined based on the weights of the samples after saturation. At this stage, we assumed full saturation.

All sites were anthropogenically disturbed and under agricultural use. The land use of all sites, except of one site which was used as arable land, was grassland. Eight of the 15 soils originated from drained bog and seven from drained fen peatlands. Six samples were classified as *Sphagnum* peat, eight as amorphous and one as sedge peat. The amorphous samples covered a wide range of strongly degraded peat with diverging SOC contents (16.8–47.4%) and therefore bulk densities (0.197–0.626 g cm^3), while the *Sphagnum* and sedge peat samples were more similar to each other (Table A.1). The amorphous samples originated from topsoils and the *Sphagnum* and sedge peat samples from subsoils. Sample depth is given in Table A.1.

2.1. Drying experiment

The samples were defrosted before the experiment. For every soil and every temperature, four replicates each of 25 g were dried at 20, 40, 60, 80 and 105 °C until mass constancy (±0.01 g) (FD720, Binder GmbH, 78,532 Tuttlingen, Germany). Mass was measured with an accuracy of ±0.0001 g (CUBIS, SARTORIUS AG, Göttingen, Germany). After drying, any roots were manually removed and the samples sieved using a 2-mm mesh. Subsequently, a subset of the samples was ground for SOC and N analysis measured by dry combustion (RC 612/TRUMEC, LECO Corporation, St. Joseph, USA). The limit of determination was ±0.0136% for SOC and ±0.0056% for N. Inorganic carbon was not determined because the samples did not contain carbonate. For quality control, each SOC and N analysis was performed with two subsamples. The differences observed between the two SOC subsamples ranged between 0.0136 and 0.78% (median: 0.06%, standard deviation 0.10%). The differences between the N subsamples ranged from 0.0056 to 0.05% (median: 0.005%, standard deviation: 0.007%).

2.2. Evaluation

For all samples and temperatures, mass loss [%] (ML) was compared with ML at 105 °C (ML_{105}) with $\Delta ML = ML - mean(ML_{105})$. The term $mean(ML_{105})$ represents the mean ML of the four replicates of every soil at 105 °C. Assuming that the soils were completely dry at 105 °C, residual water contents in SOC and N analysis were accounted for by correcting measured SOC and N values using Eq. (1) (not shown for N).

$$SOC_{cor} = \frac{SOC}{100 - \Delta ML} \quad (1)$$

The SOC and N values for all samples and temperatures were compared with the mean SOC and N values determined at samples dried at 105 °C with $\Delta SOC = SOC - mean(SOC_{105})$ or $\Delta N = N - mean(N_{105})$, respectively. The terms $mean(SOC_{105})$ and $mean(N_{105})$ represents the mean values of the four replicates of every soil dried at 105 °C.

2.3. Incubation experiment

The batch incubation experiment was performed with five soils also used in the drying experiment (Table A.1). Additionally, undried, field-moist samples were included (referred to as 'moist samples' below). In the case of samples from the drying experiment, water was added to 1.5 g of each replicate to adjust to a water content corresponding to 80% water-filled pore space (WFPS), which is the ratio of soil water content to soil porosity. The level of 80% was chosen as several studies have shown maximum carbon dioxide (CO_2) fluxes at a WFPS of around 80% (Kechavarzi et al., 2010; Norberg et al., 2018; Säurich et al., 2019). The necessary amount of water was determined with:

$$\Phi = \frac{(\phi \cdot m_{soil})}{b_d} \cdot 0.8 \quad (2)$$

where Φ [cm^3] is the added amount of water, ϕ[$cm^3\ cm^{-3}$] the porosity, m_{soil} = 1.5 g the soil mass and b_d [g cm^{-3}] the bulk density.

Water was added to the dry samples and the samples were manually stirred periodically to overcome potential hydrophobicity. Stirring was repeated on three consecutive days until all samples were moisturized. Afterwards, the samples were adjusted to the target weight (Φ + 1.5 g) either by evaporation or the addition of water.

In the case of the moist samples, the field water content was determined first so as to be able to adjust the water content to a WFPS of 80%. Samples were homogenised through a 5 mm sieve and an aliquot of the sample was dried at 105 °C. Based on the measured water contents, weights were calculated to match 1.5 g dry soil weight. Depending on the field water content, samples were either dried or wetted to the target weight corresponding to 80% WFPS.

After adjusting the water content, all the samples were pre-incubated aerobically for five days at a temperature of approximately 20 °C. Afterwards, CO_2 and N_2O fluxes were measured using gas-tight 250-ml glass flasks at a room temperature of 20 °C. Between sampling dates, the flasks were open to the atmosphere and thus the bottles were weighed before sampling and the water content adjusted where necessary. The first sample was taken shortly after the flasks were closed with a screw cap with a septum using a gas-tight syringe. The second sample was taken approximately 24 h later. Samples were transferred to evacuated 20-ml vials. The pressure in the flasks was measured after the first and before the second sampling to identify leaks. Sampling took place on days 1 and 2, 5 and 6, 13 and 14, 21 and 22 and finally on days 68 and 69.

The CO_2 concentration was measured with a gas chromatograph (Series GC-2014; Shimadzu Deutschland GmbH, Duisburg, Germany). Basal respiration was calculated as:

$$CO2 - C\ flux = \frac{100 \cdot M \cdot (c_2 - c_1) \cdot P \cdot V_{glass}}{R \cdot T \cdot \Delta h \cdot m_{soil}} \quad (2)$$

where respiration is expressed as CO_2-C flux [μg CO_2-C g^{-1} $soil^{-1}$ h^{-1}], M = 12.01 g mol^{-1} the molecular mass of carbon, c_1 and c_2 the CO_2 concentrations [ppm] at sampling time 1 and 2, P [hPa] mean pressure between sampling 1 and sampling 2, V_{glass} [m^3] the volume of the incubation glass flasks, R = 8.314463 J mol^{-1} K^{-1} the ideal gas constant, T [°K] the room temperature, Δh [h] the time between sampling 1 and 2 (approximately 24 h) and m_{soil} the dry mass of the soil [g].

To derive cumulative SOC losses for the whole experiment (μg CO_2-C g^{-1} $soil^{-1}$ 69 d^{-1}), fluxes were linearly interpolated between sampling campaigns and summed for the experiment duration of 69 days. The SOC losses per total SOC (SOC loss) [%] during the experiment were calculated by the SOC contents determined in the drying experiment. For the moist samples, the mean SOC contents of all samples of the soils were used to calculate total SOC content.

2.4. Statistical analysis

All data analyses were performed using the statistical software package R (R Core Team, 2020). For the drying experiment, seven outliers were identified by visual checks on Cleveland dot plots. These replicates were also not considered for the incubation experiments, and a further three replicates were eliminated due to the identification of incorrect m_{soil} after drying the samples at the end of the experiment.

For both the drying and incubation experiments, data were divided into two groups by the degree of decomposition. One group contained almost undecomposed and moderately decomposed samples with von Post values of 2 and 5. This group is referred to as **weakly decomposed,** although it also contained moderately decomposed samples. The second group contained **amorphous** samples with von Post values of 9 and 10. These groups are termed 'peat groups' below, while the specific horizons are referred to as 'soil'. Each 'soil' was represented by four individual samples (=replicates) for each temperature. Sample size differed between the drying (weakly decomposed samples n = 139; amorphous samples n = 154) and incubation experiments (weakly decomposed samples n = 46; amorphous samples n = 67).

The effect of temperature on ML, SOC, N and basal respiration was analysed using linear mixed-effect models, with soil as the random factor (R package nlme, Pinheiro et al., 2020). Tukey's honest significance test (α = 0.05) for linear mixed-effect models implemented in the Rpackage emmeans (Lenth, 2020) was used to determine significant differences, which are indicated by letters in the figures.

3. Results

3.1. Mass loss

All the samples were dried for 30 days. The samples dried at 40, 60, 80 and 105 °C reached weight constancy after two days. Samples dried at 20 °C needed 12–28 days to reach constant weight.

The mass loss (ML) for each drying temperature is depicted in Fig. 1a, separated into **amorphous** samples (highly decomposed peat, von Post 9 and 10; n = 154) and **weakly decomposed** samples (almost undecomposed and moderately decomposed peat, von Post 2 and 5; n = 139). Fig. 1b shows the differences between ML (ΔML) and the mean ML of every soil at 105 °C. It should be noted that ΔML at 105 °C differs from zero because ΔML was calculated for every sample using the mean ML at 105 °C of the four replicates from every soil. The mean ML for every soil and temperature is also listed in the appendix (Table A.1).

The ML of the **amorphous** samples was lower, but more variable between different soils than the **weakly decomposed** samples. For the **amorphous** samples, the 2.5–97.5% quantiles of ML ranged from 45.1 to 81.8% (median: 60.1%). The **weakly decomposed** samples lost 84.8–93.6% (median: 88.5%) of their mass.

On average, the highest ML was observed at 105 °C. The influence of drying temperature on ML differed between the two peat groups (Fig. 1a). The **amorphous samples** had a significant ML decrease for each temperature step from 105 to 20 °C. As with ML, ΔML also decreased significantly from 105 to 20 °C for each drying temperature (Fig. 1b). The influence of drying temperature on ML and ΔML was less pronounced for the **weakly decomposed** samples. No significant differences were observed between 60, 80 and 105 °C. Pairwise comparisons showed significant differences between 60 and 40 °C and between 40 and 20 °C.

Not every replicate and/or soil showed a clear characteristic of decreasing ML and ΔML from 105 to 20 °C because some individual samples behaved contrary to expectations. This can be seen in Fig. 1b by ΔML values above zero. A comparison between ML of the four replicates for each temperature and soil with the mean ML at 105 °C showed a greater ML for some samples at lower temperatures than at higher temperatures and vice versa.

3.2. Chemical soil properties

3.2.1. Soil organic carbon

Measured and corrected soil organic carbon [%] (SOC) contents and the difference with SOC at 105 °C (ΔSOC) for each temperature are depicted in Fig. 2. The 2.5–97.5% quantiles of SOC ranged from 16.1 to 48.7% (median: 34.8%) for the **amorphous** samples and from 46 to 54.2% (median: 50.7%) for the **weakly decomposed** samples.

Measured SOC contents were on average lower for lower drying temperatures. The **amorphous** samples dried at 20 °C had a median of −2.6% for ΔSOC values. For 40 and 60 °C, the median of ΔSOC was −1.2%. No significant differences were observed between the drying

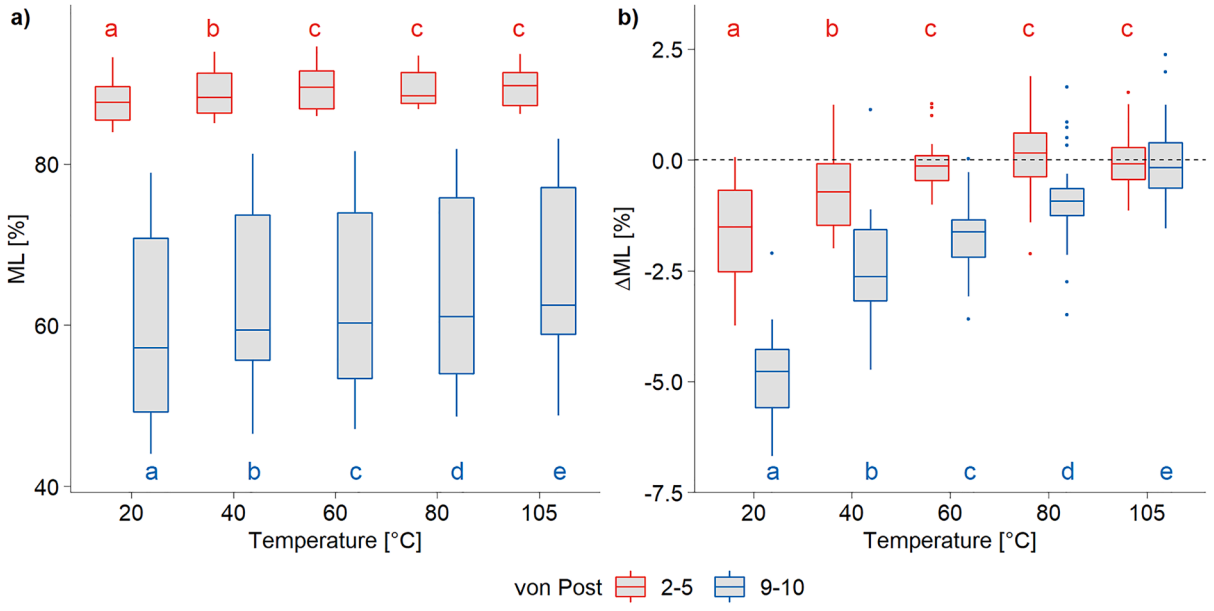

Fig. 1. a) Mass loss [%] (ML) and b) difference between ML (ΔML) and ML at 105 °C for drying temperatures 20, 40, 60, 80 and 105 °C separated into weakly decomposed (von Post 2 and 5; n = 139) and amorphous (von Post 9 and 10; n = 154) samples. Whiskers illustrate values within 1.5 times of the interquartile range below the first quantile or above the third quantile respectively. Outliers (dots) are >1.5 times and <3 times the interquartile range.

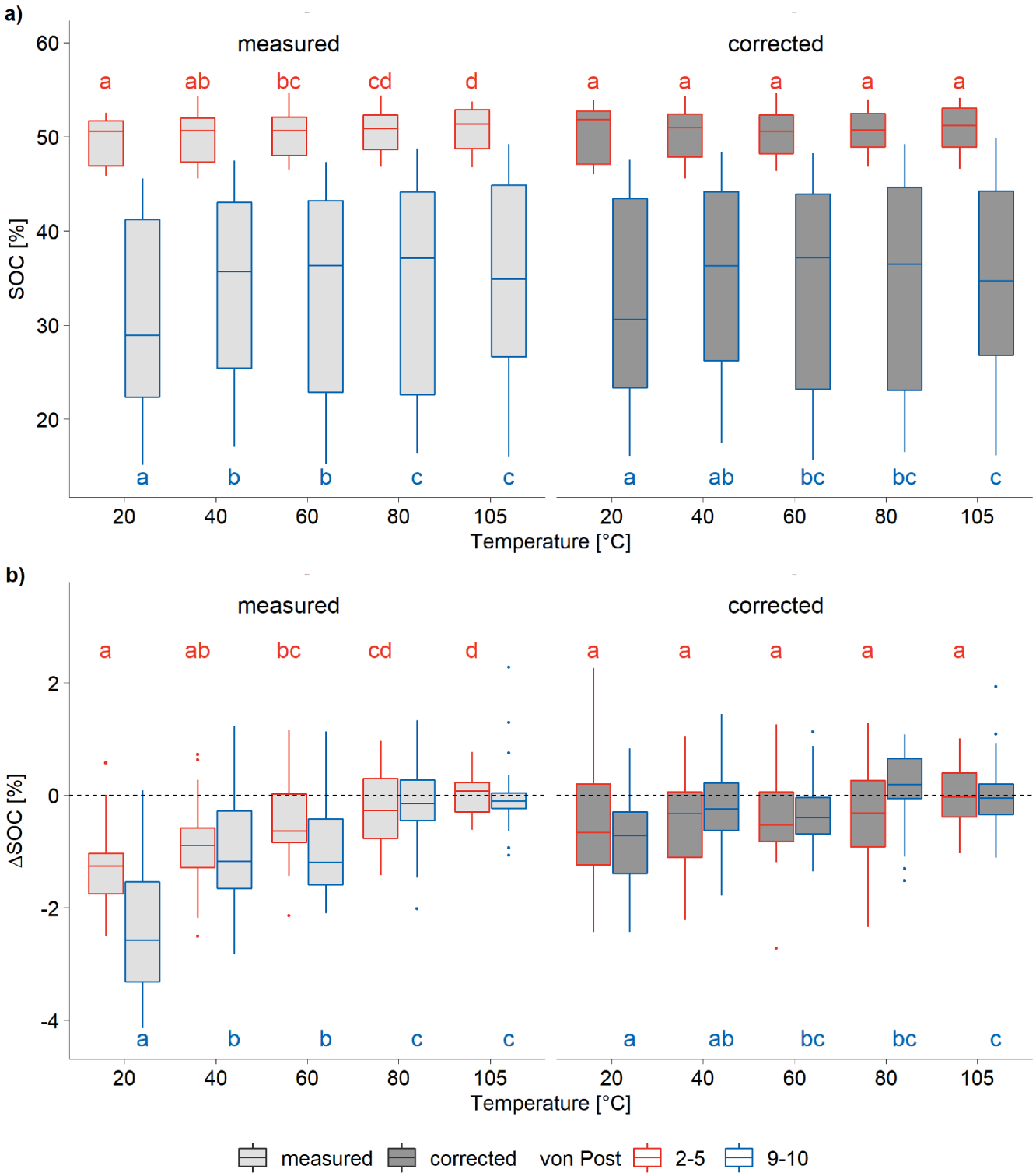

Fig. 2. Measured values of a) soil organic carbon content [%] (SOC) and b) difference from SOC (ΔSOC) at 105 °C for measured and corrected SOC values for weakly decomposed (von Post 2 and 5; n = 139) and amorphous (von Post 9 and 10; n = 154) samples at drying temperatures of 20, 40, 60, 80 and 105 °C. Whiskers illustrate 1.5 times the interquartile range below the first quantile or above the third quantile respectively. Outliers (dots) are >1.5 times and <3 times the interquartile range.

temperatures of 80 (median ΔSOC: −0.1%) and 105 °C.

Correcting the SOC data with ML led to increasing SOC and ΔSOC values. For 20, 40, 60 and 80 °C, medians of ΔSOC increased to −0.7, −0.2, −0.4 and 0.2% respectively. Despite the correction, values at 20 and 40 °C still differed from those at 105 °C.

Influences of drying temperatures were less pronounced for the **weakly decomposed** samples. The medians of ΔSOC were −1.3, −0.9, −0.6 and −0.3% for 20, 40, 60 and 80 °C respectively. Correcting the SOC values, the medians of ΔSOC increased to −0.7 (20 °C), −0.3 (40 °C), −0.5 (60 °C) and −0.3% (80 °C). After correction there were no significant differences between SOC contents analysed after drying at different temperatures for the **weakly decomposed** samples.

As with ML, the SOC and ΔSOC values per temperature differed for some samples and/or soils from the shown characteristic. This is depicted by ΔSOC values above zero in Fig. 2b.

3.2.2. Nitrogen

In contrast to SOC, the **amorphous** samples had higher N contents than the **weakly decomposed** samples (Fig. 3a). The 2.5–97.5% quantiles of N ranged from 1.1 to 3.2% (median: 1.9%) for the **amorphous** samples and from 0.4 to 1.9% (median: 1%) for the **weakly decomposed** samples.

Considering all the data, the **amorphous** samples had almost the same medians for every drying temperature, ranging from 1.7% (20 and 80 °C) to 1.8% (40, 60, 105 °C) (Fig. 3a). Nonetheless, the differences were partially significant. When comparing N contents to those of the

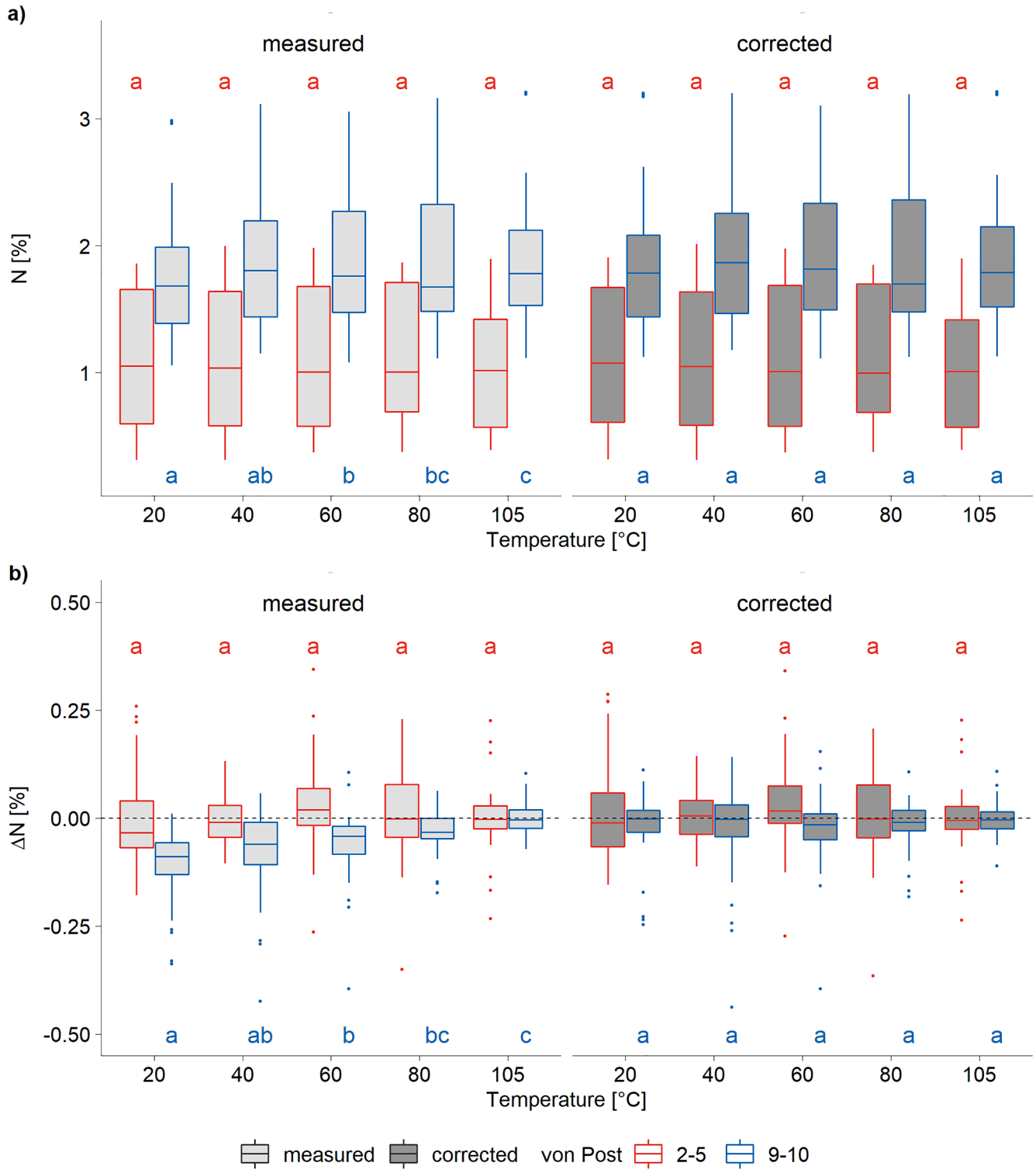

Fig. 3. Measured values of a) nitrogen content [%] (N) and b) difference from N (ΔN) at 105 °C for measured and corrected N values for weakly decomposed (von Post 2 and 5; n = 139) and amorphous (von Post 9 and 10; n = 154) samples at drying temperatures of 20, 40, 60, 80 and 105 °C. Whiskers illustrate 1.5 times the interquartile range below the first quantile or above the third quantile respectively. Outliers (dots) are >1.5 times and <3 times the interquartile range.

same soil dried at 105 °C, the effects of drying temperature became more obvious. This is reflected by the decreasing ΔN values in Fig. 3b. After correction of N values, no significant differences were observed between the temperatures. For the **weakly decomposed** samples, this was the case for both measured and corrected N values.

3.2.3. Carbon to nitrogen ratio

The carbon to nitrogen [–] (CN) ratios of the **weakly decomposed** samples (2.5–97.5% quantiles: 25.5–130 [–], median: 50.4 [–]) were considerably higher than those of the **amorphous** samples (2.5–97.5% quantiles: 12.2–30.9 [–], median: 17.5 [–]) (Fig. 4). Heterogeneity between soils and within each soil was higher for the **weakly decomposed** samples. All but one of the **amorphous** samples (relative standard deviation: 0.111 [–]) had relative standard deviations of between 0.009 and 0.043 [–]. The **weakly decomposed** samples had relative standard deviations of between 0.032 and 0.128 [–].

No significant influence of temperature was observed for any peat group. For the **weakly decomposed** samples, ΔCN values were slightly lower at 60 °C (Fig. 4b).

No correction was performed for CN ratios as the correction term (ΔML) is equal for C and N and thus corrected CN ratios would be equal to measured CN ratios.

3.3. Basal respiration

The respiration rates of the **amorphous** soils were two to three times higher than those of the **weakly decomposed** soils (Fig. 5a). The differences between the two peat groups were even more pronounced when comparing total SOC loss (Fig. 5b). For both **amorphous** and **weakly decomposed** soils, samples previously dried at 105 °C showed the highest respiration rates. Throughout the experiment, 0.8–5.2% (**amorphous** soils) and 0.2–0.9% (**weakly decomposed** soils) of the SOC was mineralised.

For the **weakly decomposed** soils, no differences were observed between moist samples and samples dried at 20, 40 and 60 °C. At higher drying temperatures, the respiration rates increased. The **amorphous** soils showed no significant difference between 20 and 80 °C, but the respiration rates of moist samples were lower than those from samples dried at 40, 60, 80 and 105 °C.

It should be stressed that the **weakly decomposed** samples only contained two soils with four replicates for each temperature (n = 46) and the **amorphous** samples contained three soils (n = 67).

4. Discussion

4.1. Determination of water content

The determination of water content is directly linked to the ML induced by drying. Owing to this straight dependency, only ML is discussed. There were two main findings regarding ML. First, ML was considerably higher for the **weakly decomposed** samples than for the **amorphous** samples. This was in line with expectations because porosity, which is directly linked to maximum water content, decreases with ongoing decomposition (Boelter, 1968; Quinton et al., 2008; Oleszczuk and Truba, 2013; McCarter et al., 2020), and thus the slightly and moderate decomposed soils of this study (referred to as **weakly decomposed**) have a higher porosity than decomposed peat soils (Table A.1). Second, ΔML for different temperatures was considerably higher for the **amorphous** samples, and differences in ΔML between the two peat groups increased with a lower temperature, i.e. the choice of drying temperature was especially relevant for strongly decomposed (amorphous) peat. This was presumably also a result of the different pore sizes and structures of **amorphous** and **weakly decomposed** peat soils. **Weakly decomposed** peat soils are characterised by large pores with easily accessible water (Mustamo et al., 2016; McCarter et al., 2020). Hence, a larger proportion of water is stored in pores that dewater easily at higher pressure heads (corresponding to low matric potentials, i.e. suction) (Buckingham, 1907). Thus, no significant differences were observed between 60, 80 and 105 °C as the pressure heads occurring at 60 °C or 80 °C already seemed sufficient to dewater all pores. On the other side, the **amorphous** samples needed higher temperatures to dewater the larger proportion of smaller pores compared to the **weakly decomposed** samples.

While there was no underestimation of the water content of **weakly decomposed** peat soils determined at 60, 80 and 105 °C, the results showed that drying temperatures below 105 °C can lead to a significant underestimation of the water content of **amorphous** samples, which in some samples was by >5% at drying temperatures of 20 °C. For the

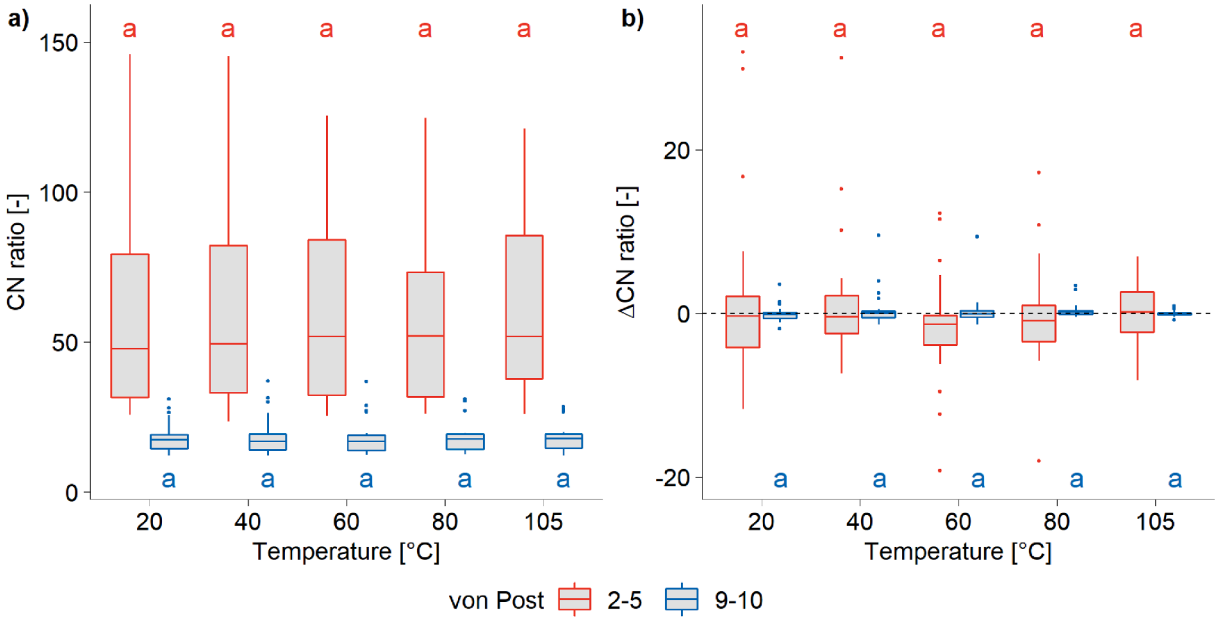

Fig. 4. a) Carbon to nitrogen [–] (CN) ratios and b) differences in CN (ΔCN) ratios for weakly decomposed (von Post 2 and 5; n = 139) and amorphous (von Post 9 and 10; n = 154) samples at different temperatures. Whiskers illustrate 1.5 times the interquartile range below the first quantile or above the third quantile respectively. Outliers (dots) are >1.5 times and <3 times the interquartile range.

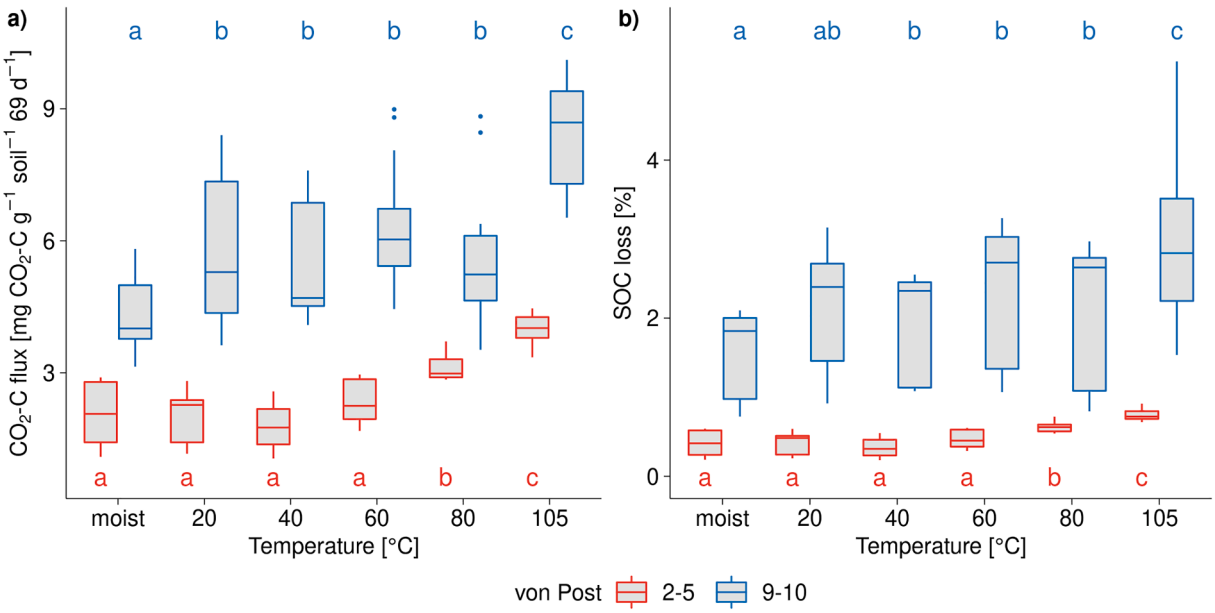

Fig. 5. a) Basal respiration rates and b) loss of soil organic carbon content per total SOC [%] (SOC loss) for weakly decomposed (von Post 2 and 5; n = 46) and amorphous (von Post 9 and 10; n = 67) samples at different temperatures. Whiskers illustrate 1.5 times the interquartile range below the first quantile or above the third quantile respectively. Outliers (dots) are >1.5 times and <3 times the interquartile range.

amorphous peat soils used in this study, this translates to a maximum bulk density difference of 0.057 g cm^3 or 21.1% (peat A5) and a mean bulk density difference of 13.9%. At drying temperatures of 80 °C, however, the maximum bulk density difference was only 0.014 g cm^3 or 4.4% (A6) and the mean bulk density difference in all **amorphous** peat soils was 2.7%.

4.2. *Influence on soil chemical parameters*

4.2.1. *Soil organic carbon and nitrogen*

The determination of SOC and N after drying at temperatures below 105 °C was biased by residual water in the sample. This was also reflected by lower ΔSOC and ΔN values for the **amorphous** samples, for which ΔML was also lower.

Correction of SOC values was unable to fully compensate for differences in SOC for temperatures below 60 °C. Therefore, it was assumed that lower SOC values were caused not only by residual water but by microbial activity as well, namely mineralisation of SOM to CO_2. This was especially the case for a drying temperature of 20 °C but also for 40 °C. The increasing ΔSOC at 40 °C (and higher temperatures), however, indicated that microbes succumb to desiccation with increasing temperatures (van Erp et al., 2001). Thus, microbial activity probably ceases to result in substantial SOC losses for temperatures above 40 °C since no significant differences were observed for the corrected SOC values at temperatures of 60, 80 and 105 °C for either the **amorphous** or **weakly decomposed** samples (Fig. 2b). In contrast to MacFarlane and Allen (1965) and O'Kelly (2004), who reported possible charring, oxidation and/or vaporisation of SOM for temperatures above approximately 80–85 °C, it could not be demonstrated that these processes lead to recognisable losses of SOC. The design of the present study did not allow final conclusions to be drawn on the effect of temperatures above 80 °C on SOC.

The results presented for the corrected ΔSOC values also indicated that potential respiration losses at low drying temperatures were greater for the **amorphous** samples, which in this study originated from the upper two horizons. This was supported by the results of the incubation experiment, which also showed higher respiration rates for the amorphous samples (Fig. 5), especially when scaling these to SOC loss. The median SOC loss of the moist samples after 30 days of the incubation experiment was 0.5%, therefore within the range of the corrected ΔSOC values at 20 °C (median 0.7%). Higher microbial activity in topsoils has also been reported by several other authors (Brake et al., 1999; Fisk et al., 2003; Preston et al., 2012; Säurich et al., 2019). Correction of SOC values at 20 and 40 °C was therefore unable to remove the differences with higher drying temperatures.

Sections 3.1 and 3.2 referred to the observation of high heterogeneities within the same soil. For temperatures above 60 °C, ΔSOC values above or below zero were interpreted as an effect of soil heterogeneity and variance within a soil, rather than an effect of temperature.

The influence of different drying temperatures on N values was only significant for the **amorphous** samples. There are probably three reasons for these findings. First, the **amorphous** samples had higher residual water contents (expressed with lower ΔML in Fig. 1b) at lower temperatures than the **weakly decomposed** samples. Second, the heterogeneity of N values within a soil was higher for N than for SOC values for both **amorphous** and **weakly decomposed** samples. For the **weakly decomposed** samples, the influence of residual water content on N determination was levelled out by the high heterogeneity of N values within a soil. Third, during decomposition, C is preferentially released from peat soils, while N is enriched. This was also reflected by the narrower CN ratios of the amorphous samples (Fig. 4a). Therefore, mineralisation losses during drying at low temperatures as discussed above will affect SOC contents more than N contents.

4.2.2. *CN ratio*

The determination of CN ratios is not biased by residual water contents because residual water affects the determination of both SOC and N in the same way. However, the interpretation of CN ratios in relation to different drying temperatures and degrees of decomposition is challenging. The CN ratio depends on SOC and N contents, which differ within and between soils and between different temperatures.

The results did not show any significant differences in CN ratios for different drying temperatures. This was not in line with expectations because the SOC values for drying temperatures of 20 and 40 °C could not be corrected, in contrast to the N values. On this basis, CN ratios should decrease with decreasing drying temperature. It was therefore concluded that the differences shown for corrected SOC values (not dependent on residual water) were compensated for by the high

heterogeneities of N.

4.3. Influence on microbial activity

Differences in basal respiration were more pronounced between the **weakly decomposed** and **amorphous** samples than the differences resulting from different previous drying temperatures. The **amorphous** topsoils had higher respiration rates than the **weakly decomposed** subsoils. This confirmed the results of the determination of SOC discussed in Section 4.2.1 and in several studies (Brake et al., 1999; Fisk et al., 2003; Preston et al., 2012; Säurich et al., 2019).

The results also showed that effects of drying and rewetting on the activity of the microbial community were not long lasting. In the case of **weakly decomposed** samples, the use of dried (≤60 °C) peat in incubation experiments seemed to be feasible after pre-incubation. In the case of **amorphous** peat, drying seemed to increase respiration compared with moist soil, but drying temperatures below 105 °C yielded different results. Similar results have been shown by Haney et al. (2004) for different mineral soils with field-moist samples and samples dried at 40 °C, and by Wang et al. (2015) for paddy soils stored at −20 and 4 °C or air-dried. Moreover, higher drying temperatures increased respiration rates. It is only possible to speculate about why the highest respiration rates were observed for the drying temperature of 105 °C. One reason could be that easily accessible SOM had already mineralised during drying for the soils dried below 105 °C. Although the results demonstrated that microbial activity could be restored to an extent at least similar to moist peat, only five different soils were incubated and there was no investigation of the microbial community present, for example. Therefore, care should continue to be taken when planning incubation experiments with dried peat soils.

5. Conclusions

This study compared the effect of drying on 15 different **amorphous** and **weakly decomposed** soils with SOC contents ranging from 16.8 to 52.2%. For an accurate determination of water content, this study's results showed that **amorphous samples** should be dried at 105 °C. For **weakly decomposed** peat soils, temperatures above 60 °C gave reliable results. It is believed that despite the significant differences, the determined water contents of **amorphous peat** soils dried at 80 °C were acceptable because ΔML values were only around −1%, which is the suggested accuracy of ASTM-D226-19 (international standard) and would result in mean bulk density differences of 2.7%. For determination of SOC, peat soils can be dried at temperatures ranging from 60 to 105 °C. Correction of the residual water content is advisable.

For the determination of microbial activity, the effect of peat type (amorphous topsoil vs. weakly decomposed subsoil) was greater than the effect of different drying temperatures. The differences between all five soils were consistent, irrespective of the drying temperature. It was therefore concluded that incubation experiments covering a wide range of different pre-treatments can be performed with soils. However, due to increased basal reparation rates, drying at temperatures of 105 °C might bias results.

The results of this study demonstrated that there is no "best" drying temperature and that the drying temperature should be chosen on the basis of the intended purpose of the study. However, this is not always clear because samples can often be stored for decades. Optimally, the drying temperature should be suited to future measurements and soil analyses, even though these may differ from the original purpose for which the samples were taken.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.geoderma.2021.115231.

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