PEATLANDS

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Procedure for Organic Matter Removal from Peat Samples for XRD Mineral Analysis

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Abstract

Ombrotrophic peatlands are recognized archives of past atmospheric mineral dust deposition. Net dust deposition rates, grain size, mineral hosts and source areas are typically inferred from down–core elemental data. Although elemental analysis can be time efficient and data rich, there are some inherent limitations. X–ray diffraction (XRD) analysis allows direct identification of mineral phases in environmental samples but few studies have applied this method to peat samples and a well–developed protocol for extracting the inorganic fraction of highly organic samples (>95%) is lacking. We tested and compared different levels of pre–treatment: no pre–treatment, thermal combustion (300, 350, 400, 450, 500 and 550 °C) and chemical oxidation (H_2O_2 and $Na_2S_2O_8$) using a homogenised highly organic (>98%) composite peat sample. Subsequently, minerals were identified by XRD. The results show that combustion is preferred to chemical oxidation because it most efficiently removes organic matter (OM), an important pre–requisite for identifying mineral phases by XRD analysis. Thermally induced phase transitions can be anticipated when temperature is the only factor to take into consideration. Based on the data required in this study the recommended combustion temperature is 500 °C which efficiently removes OM while preserving a majority of common dust minerals.

Keywords Mineral dust · Mineralogy · XRD analysis · Organic matter · Peat

Introduction

During the last three decades records from ombrotrophic (rain–fed) peat bogs have been increasingly used to reconstruct past changes in mineral dust deposition (paleodust), most commonly based on multi–element datasets from XRF and ICP–MS analyses (e.g., Shotyk et al. 2001; Marx et al. 2009; Le Roux et al. 2012; Kylander et al. 2016). In peat paleodust studies, a conservative lithogenic element such as Ti or Al, is typically taken as a proxy for mineral dust deposition and used to calculate elemental mass accumulation rates (MAR). The mineral hosts can be inferred from elemental relationships, such as from comparing ratios of light, medium and heavy Rare Earth Elements (REEs) (Kylander et al. 2016). Shifts in the mineral composition of paleodust can be indicative of source areas changes, information that can be used to deduce changes in paleo wind and/or transport processes (Marx et al. 2018).

There are, however, methodological limitations to using elemental data alone to examine net dust deposition rates and mineral composition: (1) chemically similar minerals are difficult to distinguish; (2) dust events can vary widely in their mineralogical composition, leading to an under– or over– estimation of dust variability if a single element is used as a dust proxy (Kylander et al. 2016); and (3) minerogenic and biogenic minerals cannot be separated based on elemental data alone (e.g., calcium carbonates, biogenic silica). Additionally, we are limited by that fact that (4) some methods (e.g., ICP–MS) are destructive, leading to sample loss; and (5) some elements are lost during the pre–treatments (e.g., Si in HF digestions), complicating mineral predictions.

In comparison, X–ray diffraction (XRD) analysis identifies minerals directly and non–destructively with a detection limit ranging between 0.1 to 5 w/w % depending on factors such as sample composition, crystallinity of the minerals, peak positions, efficiency of milling and measurement settings. XRD

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analysis also allows for the identification of clay mineral phases (Moore and Reynolds 1997), enabling detection and distinction of long–range transported fine dust. The inorganic matter (IM) fraction of peat contains not only atmospherically derived minerals but also oceanic salt spray (e.g., Tolonen 1984); autogenic biogenic (amorphous) silica, carbonates, and oxalates (Bauer et al. 2011); charcoal (Kuhry 1995); and tephra (Wastegård 2005). These phases affect the elemental composition of bulk peat but do not typically give reflections in XRD analysis due to low crystallinity (Moore and Reynolds 1997). To date a limited number of studies have applied XRD analysis to peat samples and those undertaken were conducted on either untreated peat (e.g., Lopéz–Buendía et al. 2007; Smieja–Król et al. 2010) or on the ash residue after combustion at 550 °C (Le Roux and Shotyk 2006).

Within the field of mineral analysis the general recommendation is to apply minimal pre-treatments to avoid causing mineral phase changes (e.g., Moore and Reynolds 1997). This conflicts with another general recommendation that OM should be removed, typically with H_2O_2, prior to mineralogical analysis if it exceeds 3–5% of the total weight (Moore and Reynolds 1997). The dominant peat type in the northern latitudes, *Sphagnum* peat, is highly resistant to decomposition (Rydin and Jeglum 2013). Therefore, OM removal methods that are effective for other soil types may not be applicable to peat samples. The amount of OM in these kinds of samples (typically >95%) compounds the challenges of extracting IM from peat without altering the chemical or physical composition of mineral phases (Andrejko et al. 1983; Moore and Reynolds 1997; Mikutta et al. 2005).

The two most commonly applied methods for extracting IM from peat are wet chemical oxidation and thermal combustion (dry ashing). Protocols for chemical oxidation of OM vary by discipline, but commonly involve hydrogen peroxide (H_2O_2) (e.g., Battarbee 1986), sodium hypochlorite (NaClO) (Andersen 1963), hydrogen chloride (HCl) and/or nitric acid (HNO_3). These chemicals, however, can cause partial or complete mineral dissolution (Mikutta et al. 2005), aggregation (Carrado et al. 2006) and/or oxidation of ferrous iron (Jones and Bowser 1978). For example, oxidation with H_2O_2 potentially dissolves magnesium oxides, carbonate and sulfide minerals (through acidolysis), and can cause disintegration of clay mineral layers (Mikutta et al. 2005).

Thermal combustion is commonly applied within paleo–studies to establish weight percentages of OM (determined from the loss–on–ignition) (Dean 1974). In peat studies temperatures from 450 °C (e.g., Marx et al. 2009; Kylander et al. 2013) to 550 °C (e.g., Le Roux and Shotyk 2006) have commonly been used, although a study by Beaudoin (2003) reported combustion at 375 °C as sufficient for OM removal. Boyle (2004)

later showed that, depending on soil or sediment type, temperatures of up to 550 °C are required to efficiently remove OM. Temperatures as low as possible are recommended to minimise thermally induced mineral phase changes. Physical and chemical alterations of less crystalline minerals (e.g., iron oxides and hydroxides) can start already at 40 °C (Kaiser and Guggenberger 2003), although many minerals are stable (or go through reversible changes) below 200–300 °C (Brown and Brindley 1984). Oxidation through thermal combustion (375–750 °C) can cause dehydroxylation, decomposition and structural as well as chemical transformations of minerals (Andrejko et al. 1983; Brown and Brindley 1984; Moore and Reynolds 1997).

The aim of this study is to establish an optimal protocol (i.e., causing the least amount of mineral alteration) for the analysis of the mineral composition of peat samples. This is achieved by comparing XRD data of samples with different levels of pre–treatment: no pre–treatment; thermal combustion at 300, 350, 400, 450, 500 and 550 °C; and wet–chemical oxidation using either H_2O_2 or $Na_2S_2O_8$. We used peat samples from a stratigraphic sequence from Store Mosse (SM) and a composite sample from Dumme Mosse (DM), two ombrotrophic bogs located in southern Sweden which are detailed elsewhere (Bindler 2003; Kylander et al. 2013, 2016, 2018). To verify the effects of the different pre–treatments, known clay mineral standards were added to bulk samples prior to the different pre–treatments.

Materials and Methods

Exploratory work was first conducted on freeze–dried SM peat samples (1 cm thick) from different depths. Based on these results a structured replicate study using the composite peat sample from DM was designed as summarised in Fig. 1.

To evaluate what (if) minerals can be detected in bulk peat, four untreated freeze–dried samples from the SM peat sequence were crushed in a mortar and packed for XRD analysis (Smieja–Król et al. 2010). One half of the analysed sample was then thermally combusted at 450 °C while the other half was chemically oxidised using H_2O_2 or $Na_2S_2O_8$. This procedure enabled comparison of XRD patterns of untreated, combusted and chemically oxidised sub-samples from the same composite sample.

Chemical oxidation with H_2O_2 was conducted after sieving the sample through either a 63 μm or a 125 μm mesh in order to physically remove larger organic particles, after which 17% H_2O_2 was added to the sample. The solution was left overnight at room temperature and subsequently heated to 80 °C in a water–bath. Additional H_2O_2 was added until the reaction stopped and the supernatant was clear.
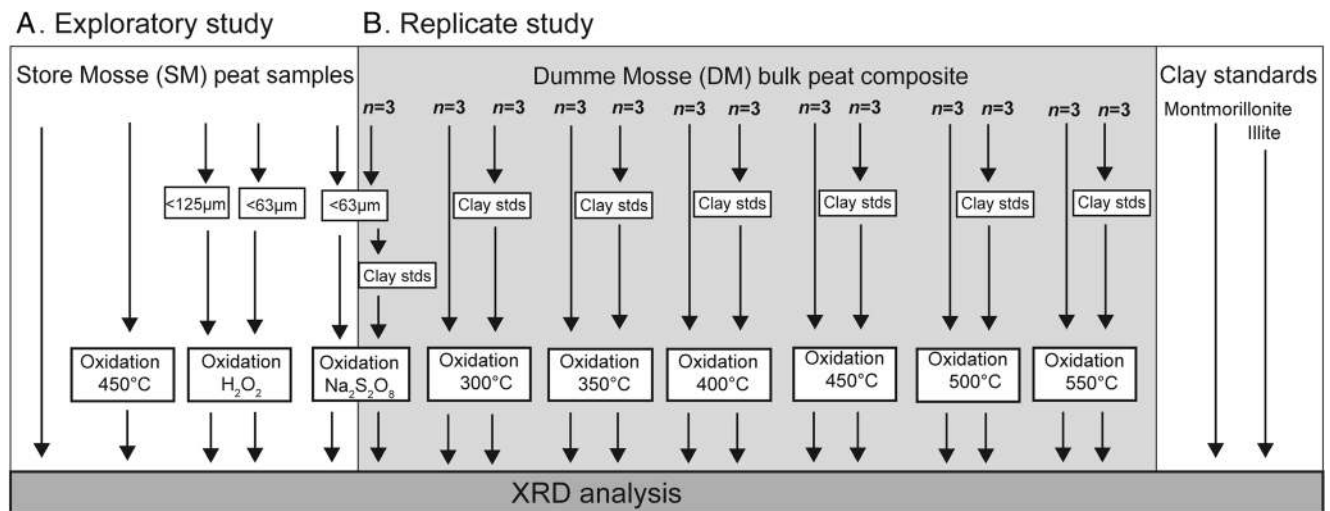
Fig. 1 Workflow scheme. Initial exploratory study (**a**) conducted on SM samples: analysis of bulk peat samples, followed by combustion at 450 °C, and chemical oxidation by H_2O_2 or $Na_2S_2O_8$. Based on the outcome of (**a**) the replicate procedure (**b**) was designed. A large homogenised peat composite was divided into sets of triplicates ($n = 3$) for each extraction method ($n = 39$). Clay standard reference patterns were retrieved by gently crushing the samples, followed by c. 30 s of insonication and subsequent (oriented) mounting on silver membrane filters of the <2 μm fraction (Moore and Reynolds 1997), and XRD analysis

Chemical oxidation by $Na_2S_2O_8$ was conducted on the <63 μm residue, using 2 moles of $Na_2S_2O_8$ per mole carbon to be oxidised, combined with sodium hydrogen carbonate ($NaHCO_3$) as a buffer (Meier and Menegatti 1997). $Na_2S_2O_8$ and $NaHCO_3$ (mass ratio: 2:1) were dissolved in 100 ml distilled water and added to glass beakers containing the sample. The solution was heated to reaction temperature (80 °C) and additional solution was added until the liquid was clear and the residue changed colour from brown to grey.

The replicate procedure (Fig. 1b) was designed based on the results of the exploratory tests (Fig. 2). Given the performance of oxidation with H_2O_2 in the exploratory study, it was not included in the second stage of development (described below). An air–dried (35 °C) composite sample of peat collected in 2002 at DM (a 1 m section, approximately from 2–3 m depth) was homogenised and ground to a fine–grained powder for several minutes in a food processor. To verify the effect of thermal combustion and chemical oxidation on the minerals a batch of six sub-samples (500 mg each) per pre–treatment procedure were prepared: three with bulk peat and three with bulk peat and clay mineral standards (Fig. 1b). An equal mixture of illite (IMt–1, c. 25 mg) and montmorillonite (SAz–2, c. 25 mg) from the Clay Mineral Society Source Clay Repository was added. The sub–samples were then chemically oxidised with $Na_2S_2O_8$ or thermally combusted (Fig. 1b). The chemically oxidised samples initially resisted oxidation and were therefore subjected to a second oxidation procedure in a closed–circulation oven, following the protocol of Eusterhues et al. (2003).

XRD analyses were performed at the Department of Geological Sciences, Stockholm University, using a PANalytical X–ray diffraction system (X'Pert Powder). The samples were generally analysed from 2° to 70° 2θ, CuKα radiation at 45 kV and 40 mA, passing through a curved graphite monochromator to diffract single wavelength radiation to the detector, 1 s spinning time, programmable fixed divergence and receiving slits (1°), step size 0.020° and a count time of 1 s. The system is equipped with a point type detector (Proportional Xe). The ash residue was washed three times in water to remove soluble salts. The samples were transferred to 50 ml centrifuge tubes, filled with distilled water, insonicated and centrifuged 10 min at 4000 rpm. The supernatant was subsequently removed and the sample transferred to weighing boats, oven–dried (50 °C) overnight, and finally oriented on silver membrane filters using a vacuum filter apparatus to enhance the basal reflections of the clay minerals (Moore and Reynolds 1997). Mineral identification and processing of data (determination of background, smoothing, and peak localisation) was conducted in HighScore, a PANalytical software, with an integrated ICSD mineral database (2012). Brown and Brindley (1984) and Moore and Reynolds (1997) were also used to guide mineral identification.

To assess the efficiency of the different pre–treatments, selected samples were analysed for residual elemental carbon in a Carlo Erba NC2500 elemental analyser coupled with a Finnigan MAT Delta + mass spectrometer. The relative error for these measurements was <3%.

Fig. 2 a – c: Diffraction patterns of one representative sample from the pilot study (SM131–1) subjected to different pre-treatments. The results indicate that the OM in the untreated samples (a, blue) preclude the detection of minerals, H_2O_2 is inefficient in removing OM (b, green) and combustion at 450 °C is comparably efficient in removing OM (c, red). **d – h**: Diffraction pattern of five samples oxidised with H_2O_2 after initial sieving through 125 µm (d and e) or 63 µm (f – h) mesh.
i – l: Diffraction pattern of samples (<63 µm) oxidised by $Na_2S_2O_8$ The XRD data are available for download at the Bolin Centre data repository, https://bolin.su.se/data/Sjostrom-2018
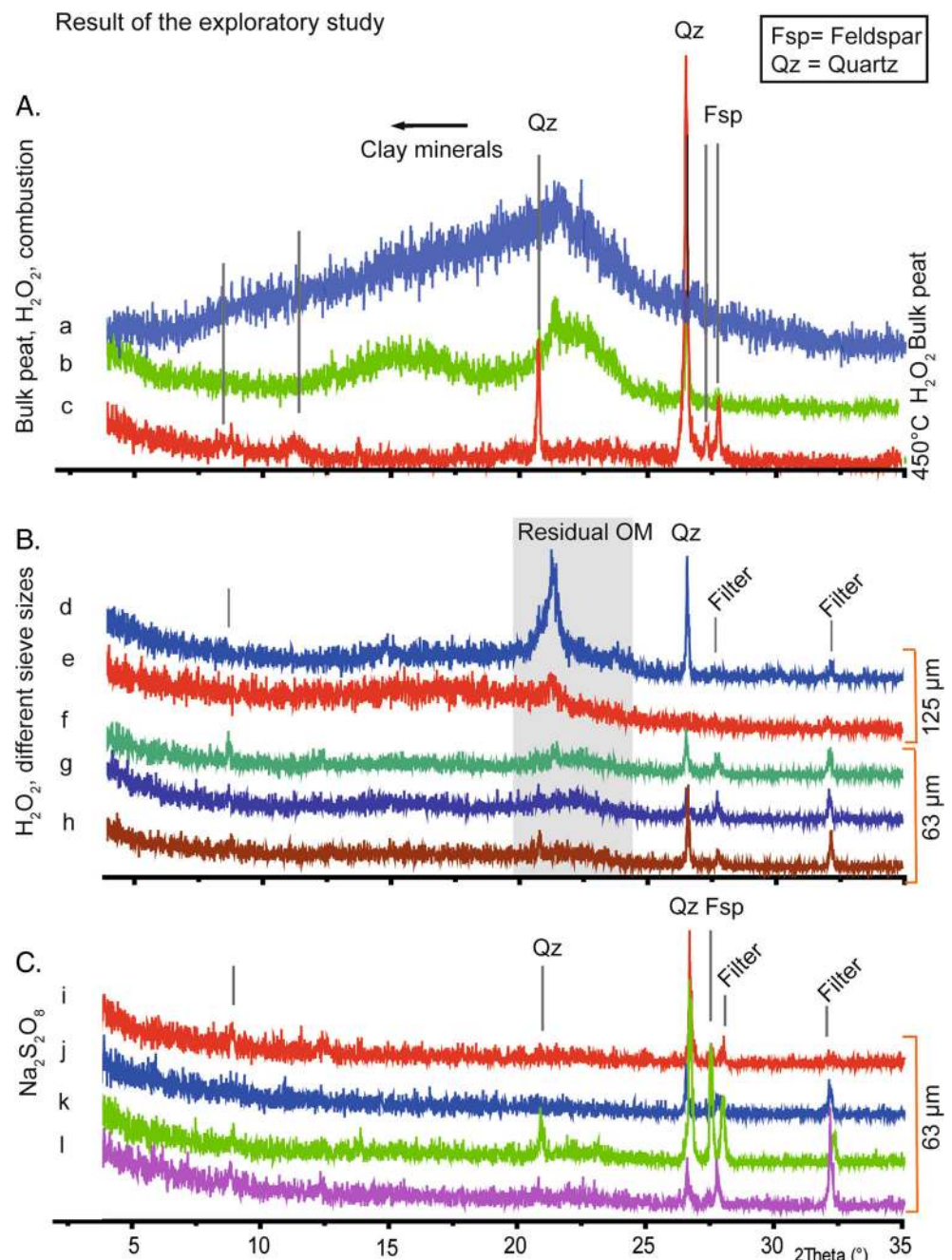
Results and Discussion

Results from the Pilot Study

The aim of this study was to test and evaluate methods for efficiently removing OM from highly organic peat samples while causing minimal alteration to its minerals. Figure 2a–c shows the diffraction patterns of a representative peat sample (SM131) from the exploratory study following no pre–treatment (a, blue), chemical oxidation using H_2O_2 (b, green) and combustion at 450 °C (c, red). The shape of the diffraction pattern of the untreated (a, blue) sample indicates the presence of significant amounts of amorphous OM (Mandile and Hutton 1995), with no detectable mineral peaks. The sinuous shape of the diffraction pattern of the H_2O_2 treated sample (b, green) with two wide humps, around 15° and 22° 2θ are indicative of weak crystallinity, most likely from residual amorphous OM. The diffraction pattern of the thermally combusted sample (450 °C) (c, red) lacks the broad reflections originating from residual OM and displays a number of identifiable mineral peaks including clay minerals, quartz and feldspars.

These results demonstrated that the OM in untreated samples prevented detection of minerals, motivating further evaluation of pre–treatment methods. The initial tests using H_2O_2 for chemical oxidation showed that OM removal was incomplete even after 5–10 days of oxidation.

In an attempt to decrease the chemical oxidation time another batch of SM samples were prepared for oxidation by H_2O_2. This time the samples were sieved through a smaller mesh (63 μm) to physically remove more of the OM fraction. The shape of the subsequent XRD patterns indicated that initially removing particles >63 μm increased oxidation efficiency but the broad reflection from residual OM was still present (Fig. 2d – h). Ultra acidic conditions were measured (pH 1–2) during the oxidation process, raising concern about acid dissolution of minerals (e.g., calcite, clays) during the chemical pre–treatment (Mikutta et al. 2005).

The shape of the diffraction patterns of the first batch of samples oxidised with $Na_2S_2O_8$ indicated that increased oxidation was achieved compared to oxidation with H_2O_2, with a low amounts of residual OM (Fig. 2 i – l). In all samples quartz was identified, but the diagnostic peak (3.34 Å) was considerably lower than in the ash residue. In three out of four samples two minor clay mineral peaks were detected at 8.8° 2θ (Fig. 2i, k and l) and 12° (7 Å). All remaining peaks between 20° and 35° 2θ were related to quartz, feldspar or the filter.

Replicate Procedures Using the DM Composite Sample

Chemical oxidation of OM by $Na_2S_2O_8$ in the first batch of DM sub-samples were unsuccessful. From visual inspection, it was clear that OM remained (brown colour) after 10–15 h at reaction temperature. Two samples (DM27 and DM28) were therefore rinsed and oxidised a second time, following the procedures of Eusterhues et al. (2003). After this second oxidation, 40 wt% elemental C still remained. Another batch of samples (n = 3; DM19, 20 and 51) were prepared for oxidation (including clay mineral standards) following the same procedure but the oxidant was dissolved in less water (25 ml). Following this treatment ~8 wt% elemental C remained. The diagnostic peaks of the clay mineral standards were present in the diffraction patterns (Fig. 3, j). Overall, the results show that chemical oxidation with $Na_2S_2O_8$ was inconsistent. Considerably longer oxidation times were also required for the peat samples than for the soil types investigated by Mikutta et al. (2005). Additionally, in all samples oxidised with $Na_2S_2O_8$ (both SM and DM), the quartz and feldspar peaks were significantly lowered (or absent) compared to the XRD

pattern of the ash residue (Fig. 2), either indicating that these minerals were sieved off prior to oxidation or that the oxidation procedure caused complete or partial dissolution of these minerals.

Subsequent testing of thermal combustion used six sub-samples combusted at each tested temperature: 300, 350, 400, 450, 500 and 550 °C (Fig. 3, a – f). High amounts of residual OM (Table 1) in the samples combusted at 300 and 350 °C prevented identification of minerals (Fig. 3, a and b), while quartz was identified in the samples ashed at 400 °C (c). In the ash residue of samples combusted at 450 (d), 500 (e), and 550 °C (f) quartz, feldspar and minor amounts of clay minerals were identified. Elemental analysis of these samples showed that 4–5 wt% total C remained in the 450 °C residue, while 0.5 wt% and 0.6 wt% respectively were measured in the 500 and 550 °C ash residue. The weight percentage of the ash residues after combustion at the different temperatures are summarised in Table 1.

The XRD analysis of the 450 (Fig. 3, g), 500 (h) and 550 °C (i) ash residues (including standards) allowed identification of illite and quartz, but the diagnostic basal peak of montmorillonite at 6° (15 Å) is absent in the data of all three samples, likely an effect of the combustion causing a collapse of the mineral and concomitant peak shift from 6° (15 Å) to 8° (10 Å) (Moore and Reynolds 1997). This also explains the more intense and broader 8° (10 Å) peak in the data. Montmorillonite, however, can still be identified based on the other peaks of the mineral.

Protocol Recommendations

Direct XRD mineral observations can supplement more established paleodust record methodologies (i.e., elemental analysis), by verifying or adjusting assumptions about the relationship of elemental data to minerals. By removing OM from peat samples, direct observation and identification of minerals is enabled.

The chemical oxidation tests showed that the bulk sample needed to be split by sieving off larger particles (> 63 μm) to avoid oxidation times exceeding several weeks. The results indicate that sieving not only remove organic particles but also parts of the inorganic mineral matrix (data not shown). H_2O_2 was inefficient in oxidising OM, with significant amounts of residual OM remaining even after 10 days of oxidation. $Na_2S_2O_8$ was more efficient than H_2O_2, given that the protocol of Eusterhues et al. (2003) was followed, but still less efficient in removing OM compared to combustion. The clay mineral standards remained unaltered after oxidation by $Na_2S_2O_8$, in line the findings of

Result, replicate study
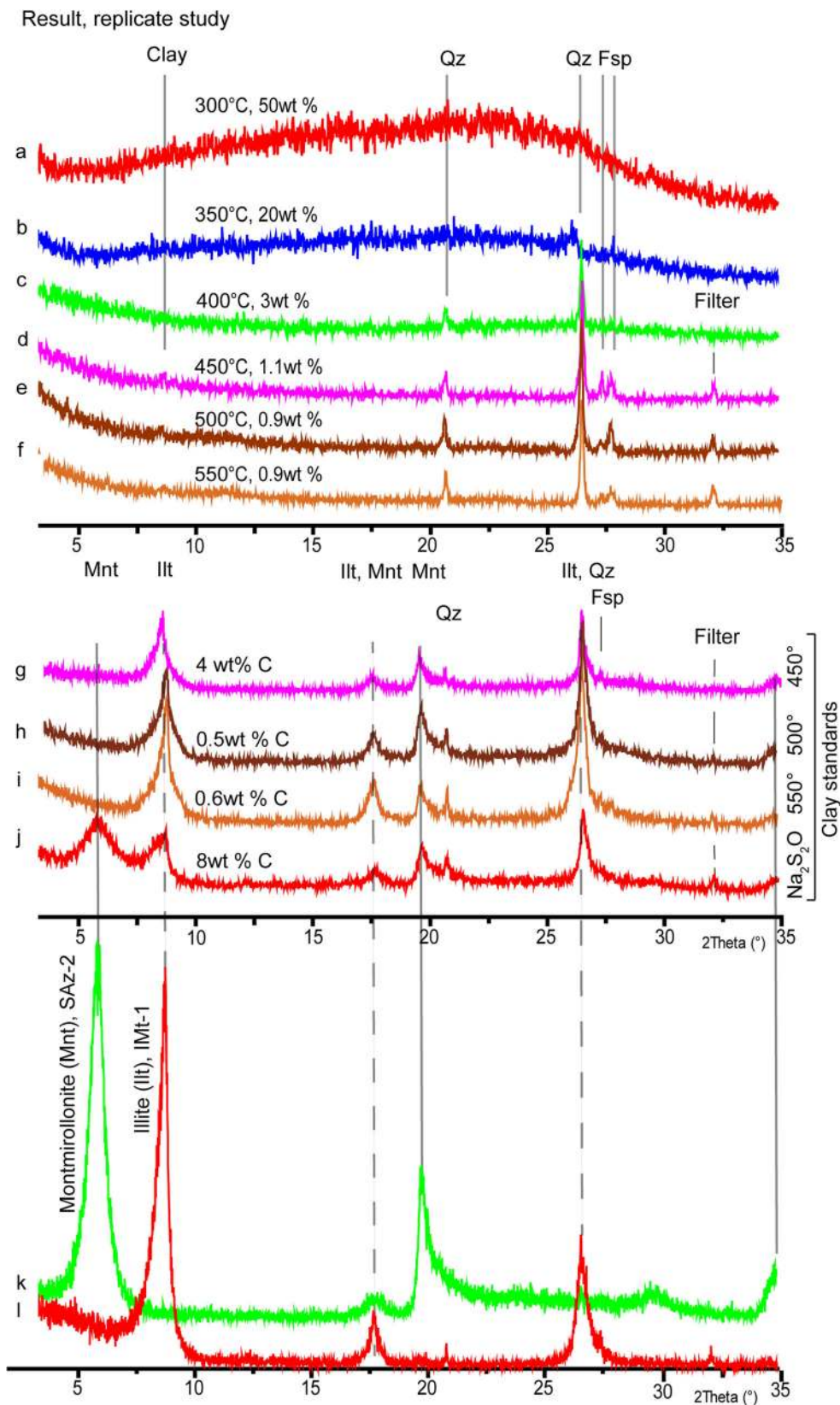
Mikutta et al. ([2005](#)), but 8 wt% C remained (*c.* 16% OM). Furthermore, the outcome of the procedure was inconsistent with several oxidation rounds required for some samples.

Fig. 3 Result of XRD analysis following combustion or chemical oxidation of DM samples. **a – f:** Uppermost lines depict one representative result per combustion temperature (without added clay standards), including wt% of the ash residue. **g – j:** The lines show one representative sample after the different pre-treatments, with clay standards added prior to the treatment, including residual carbon values. **k and l:** The lowermost lines display the diffraction patterns of untreated clay standards as a reference of peak positions of the respective minerals. The XRD data is available for download at the Bolin Centre data repository, https://bolin.su.se/data/Sjostrom-2018

Combustion at 500 °C was efficient in removing OM resulting in an ash content of *c.* 0.9 wt% and residual total carbon of 0.5 wt%; an additional increase in temperature did not increase OM removal efficiency (Fig. 3, Table 1). Combustion at 450, 500 and 550 °C allowed for the identification of illite, quartz and feldspar but caused dehydroxylation, and related peak loss, of montmorillonite. Combustion at 450 °C and lower left significant amounts of residual OM and is not recommended for mineral identification.

Given the importance of efficiently removing OM to allow identification of minerals in a sample matrix we find that combustion is generally recommended over chemical oxidation due to greater OM removal efficiency compared to chemical oxidation. Commonly occurring atmospheric dust minerals, such as quartz, feldspars, calcite, dolomite and some clay minerals (e.g., illite) (Scheuvens and Kandler 2014), all remain unaffected by the combustion temperature suggested here. Many minerals of igneous origin likely remain unaltered

after combustion at this temperature and constant pressure (1 atm) while more sensitive minerals, e.g., some clay minerals (smectite, kaolinite, and montmorillonite), sulphates, and iron oxides, could transform, which should be taken into consideration during the identification process.

Thermal treatment is commonly applied to identify minerals, and literature describing the expected effects after thermal treatment is therefore available (i.e., Neumann 1977; Brown and Brindley 1984; Földvári 2011) that can be used to guide mineral identification after combustion. For example, hydrous oxides of aluminium and iron will lose water on heating and transform to anhydrous corondrum and hematite, respectively (Brown and Brindley 1984). Some clay minerals (mica, smectite, vermiculite and chlorite) will give a reflection at 10 Å after combustion between 500–600 °C (Brown and Brindley 1984). A reflection around 10 Å can therefore be indicative of transformed clay minerals and used as a guide of where in a sequence it might be worth to be conduct further analysis to identify clay minerals (e.g., chemical oxidation by $Na_2S_2O_8$ followed XRD analysis).

By applying the protocol suggested above, the ash residue, which is routinely assessed within peat paleodust studies, can be utilised to give valuable information about the mineral composition of the dust. This information can be used to complement and guide interpretation of elemental datasets. Furthermore, establishing the mineral composition can be helpful in dust source tracing efforts and understanding potential fertilization effects of mineral dust to bog ecosystems.

Table 1 Ash residue in wt% after combustion at different temperatures

Sample	Sample weight, g	T° C.	Ash weight, g	Ash residue, wt%
40	0.473	300	0.242	51.07
41	0.471	300	0.234	49.67
42	0.462	300	0.228	49.35
30	0,457	350	0.105	22.89
31	0.466	350	0.098	21.05
32	0.477	350	0.090	18.87
43	0.465	400	0.012	2.54
44	0.485	400	0.018	3.67
45	0.463	400	0.011	2.40
52	0.512	450	0.006	1.11
53	0.491	450	0.005	1.07
54	0.491	450	0.005	1.06
57	0.437	500	0.005	1.05
59	0.503	500	0.004	0.86
60	0.502	500	0.004	0.88
33	0.467	550	0.004	0.92
34	0.460	550	0.004	0.91
35	0.467	550	0.004	0.90

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