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HIGH-RESOLUTION PEAT CORE CHRONOLOGY COVERING THE LAST 12 KYR APPLYING AN IMPROVED PEAT BOG SAMPLING

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ABSTRACT. This work focuses on building a high-resolution age-depth model for quantitative palaeoclimate study from the Mohos peat bog, East Carpathian mountains. The investigated core presents a continuous peat profile for the last 12 kyr. The chronology was based on 36 accelerator mass spectrometry radiocarbon (AMS ^{14}C) analyses of the separated *Sphagnum* samples from different depths of the profile. Dry *Sphagnum* samples for AMS dating were prepared using the classic acid-base-acid (ABA) method followed with an oxidative bleaching step to get clean cellulose. *Sphagnum* cellulose samples were measured by AMS using the EnvironMICADAS at the ICER (Debrecen, Hungary). A high-resolution chronology was obtained with the use of Bayesian age-depth modeling. Peat accumulation rate has been calculated and the sections with variable accumulation rate values were observed along the profile.

KEYWORDS: AMS radiocarbon dating, coring, peat bog, *Sphagnum*.

INTRODUCTION

Peats are important archives from the onset of the Holocene up to the present day. Peat bogs survive environmental changes and they can preserve events at high resolution in a continuously accumulating sediment. Stable isotopes preserved by the cellulose in the fossilized *Sphagnum* components can be determined, which allows for the reconstruction of temperature during the formation of sediment. In such studies, ombrotrophic peat bogs are more suitable, as they are fed by rainwater and there is no surface inflow (de Vleeschouwer et al. 2010).

Vegetation of peat bogs is dominated by *Sphagnum* species and dwarf shrubs may accumulate large amount of sediments (often up to 10–11 m) which are high in organic material content (~65%), which in a few cases may reach even 95–99% (Charman 2002).

In order to make an environmental reconstruction based on these sediments, it is important to have a reliable sampling method which provides (as far as possible) undisturbed sediment core of adequate quality and amount for the analyses, to provide an accurate and detailed chronology. For high-resolution studies, samples of sufficient quality and quantity are necessary (De Vleeschouwer et al. 2010). Various types of sampler equipment/methods and their modifications appeared from the beginning of the 1950s. One of the most common and widely used is the Russian type corer, introduced by Belekopytov and Beresnevich in 1955, which was further developed by Jowsey (1966).

The advantage is that it is very easy to use, requires no special technique and consists of lightweight pieces making them easy to carry even in a backpack. The equipment is available in several diameters (28–100 mm) and allows for the extraction of a half or one-meter-long core at once and in one piece. Disadvantages of this corer are the compaction and sediment mixing that may occur during sampling.

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Peat samples are fibrous and may contain hard twigs and trunks. Drilling tools must be able to penetrate through these tougher parts without mixing the sediment. The shape and design of the cutting edge is also important. Piston corers designed for lake sediments may be modified for peat sampling, as was described by Wright (1993) and Cumming et al. (1993).

Our aim in this study was twofold: (1) to develop a novel, more robust and reliable sampling corer for peat bogs, which allows less disturbed sampling with minimal chance of core compression or mixing, and (2) to construct a high-resolution age-depth model for the Mohos peat bog.

One of the most widespread dating methods for peat deposits is radiocarbon (^{14}C) dating. A wide range of materials from peat could be used for this purpose. Unfortunately, there is no consensus regarding selecting and preparing material for proper ^{14}C dating of peat. A number of different preparation techniques are applied, such as removing roots before dating bulk peat sediments (Törnqvist et al. 1992), or selecting remains of *Sphagnum* species (Kilian et al. 1995) to avoid possible reservoir effects. However, the existence of a reservoir effect in bulk peat ^{14}C samples could not be confirmed by Blaauw et al. (2007). Others have recommended different fractions of bulk peat (humin, humic, and fulvic acids), but from different studies it is clear that there are still many questions and efforts to understand peat formation and to select the most appropriate samples (Shore et al. 1995; Nilsson et al. 2001; Brock et al. 2011). Previously, bulk peat was used for dating as earlier beta-counting measurements required grams of carbon for analysis. During bulk sediment dating, modern rootlets penetrating down to the older peat sections can cause problems due to recent contamination. Accelerator mass spectrometry (AMS) ^{14}C techniques allow us to date plant fragments, in very small quantities (0.01–1 mg of carbon for analysis), avoiding contamination and also enabling to date a greater number of thinner peat sections. Thereby the more precise dating of short-term events and abrupt changes can be obtained.

Another critical point is the number of samples representing the core sections. It is questionable how much information we lose if we have only a few measured dates from a sediment. However, there are suggestions for the appropriate number of dates to sample per sequence for the meaningful application of the Bayesian technique (Blaauw et al. 2007). In our study, we targeted 3–4 points per millennium also taking into account places where deposition rate changes occurred. For higher-resolution dating, it is necessary to have decadal or centennial timescales for detailed studies of environmental change.

The work presented in this paper forms part of a larger research project that aims to reconstruct the temperature based on measuring the $\delta^{18}O$ from peat cellulose from the Mohos peat bog, East Carpathians. Studies of $^{18}O/^{16}O$ oxygen isotope ratio of alpha-cellulose from peat samples indicate that variations in the isotopic composition provide estimates of climate change and palaeotemperature (Hong et al. 2000, 2001). Palaeotemperature reconstructions based on the oxygen isotopic analysis have only been conducted for a handful of studies concentrated on the Holocene period in the Carpathian region (e.g. Constantin et al. 2007; Demény et al. 2013; Magyari et al. 2013; Dragusin et al. 2014). As peat bogs occur in many areas in the Carpathian region, this technique has great potential in palaeotemperature studies from this area (Willis et al. 1997, 1995; Magyari et al. 2001; Wohlfarth et al. 2001; Björkman et al. 2003).

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Based on previous studies from the Mohos peat bog (Tantau et al. 2003; Schnitchen et al. 2006; Longman et al. 2017), we expected a more than 10-m-long well-preserved ombrotrophic peat sequence, which would be an excellent record for palaeotemperature studies.

STUDY SITE

The Mohos peat bog (46°8′3.60″N, 25°54′19.43″E, 1050 m asl, 80 ha area) and Lake St. Anna (46°07′35″N, 25°53′17″E, 946 m asl) is situated in the Ciomadul Massif (South Harghita Mountains), part of a Neogene-Quaternary volcanic range in the East Carpathians, Romania (Figure 1). The Ciomadul is part of Călimani-Gurghiu-Harghita, the inner Neogene-Quaternary volcanic range of the East-Carpathian mountains. The mountain is the youngest volcano of the Carpathian-Pannonian region, based on detailed geochronological studies, the oldest rocks range from 1 Ma to 220 ka (Szakács et al. 1993; Pécskay et al. 1992, 1995) and the most recent volcanic events occurred in the past 27–50 ka (Harangi et al. 2015; Szakács et al. 2015; Karátson et al. 2016). Post-volcanic activity in the form of gas upwelling (mofettas and solfataras) and mineral water springs are still observable today.

The Mohos peat bog occupies one of the twin volcanic craters and it is unique to this area as meters of continuous *Sphagnum* peat sediment accumulated during the Holocene period (Tantau et al. 2003).

The bog belongs to the temperate zone and the only source of water is from precipitation (800–1000 mm per year) (Karátson et al. 2013). It has one outflow called Veres stream, which starts from the northeast segment of the peat bog. Today, the peatbog is partly covered with *Pinus sylvestris*, *Betula pubescens*, and there are species of *Ericaceae* with *Sphagnum* species (Tantau et al. 2003).

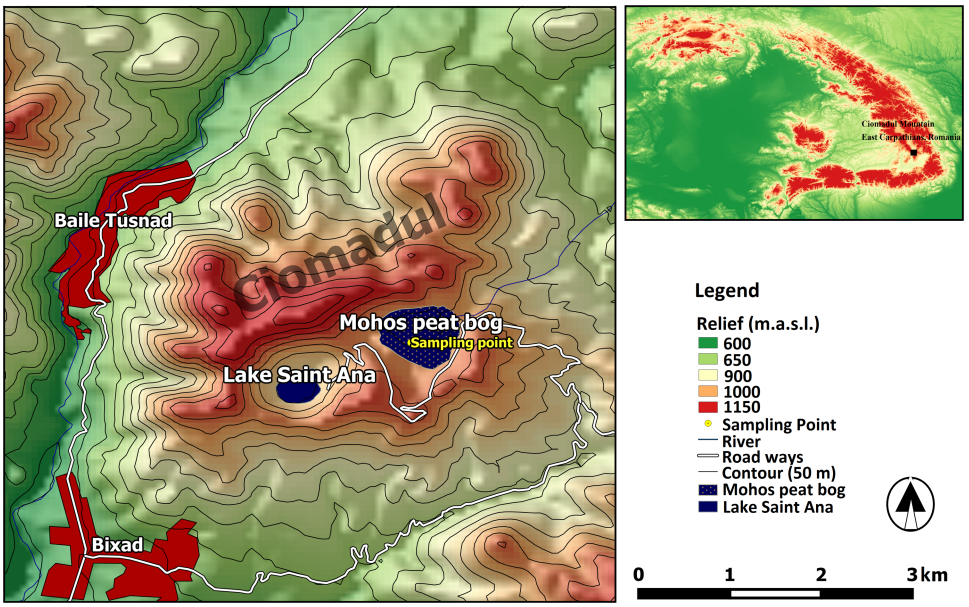


Figure 1 Location of the studied peat section.

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MATERIALS AND METHODS

Coring

Our first coring from the Mohos peat bog was carried out in 2004 using a Russian sampler (Schnitchen et al. 2006). Since we received slightly disturbed samples with the Russian sampler there was no measured ^{14}C analysis on this sediment. As our aim was also to sample the lake sediment under the 10-m peat section, a modified piston corer was used operated with a tripod in summer 2012. The coordinates of sampling location were 46°08′05.5″N, 25° 54′16.8″E. The modified piston corer consisted of aluminium tubes (52 mm ID, 60 mm OD; Figure 2), a 1-m-long and 76 mm in diameter stainless steel pipe, which was used as a sampler with a piston and sharp cutting edge. The advantage of the sharp cutting edge is that it could penetrate through the roots or fibrous layers, and even tree branches. To support an easy penetration of sampler through the peat fibrous layers, a breakthrough/electric hammer was used instead of hand-operated hammer. This system provided a smooth penetration and recovery of undisturbed and uncompressed samples. High-frequency blows cut off the fibers and the sampler did not compress the peat in this process. Also, a casing was used to prevent closing up of the peat layer. Although this sampler contains more elements and is heavier than a Russian corer, it eliminates the compaction and can continue sampling even through dense material.



Figure 2 A) Our team during coring the Mohos peat bog; B) example of 1-m-long uncompressed peat core; C) cutting edge.

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Ten 1-m-long core sections were extruded from the stainless steel pipes on-site. Sections were wrapped in plastic film, stored in PVC tubes and transferred to the laboratory, where they were kept in a cold room until subsampling. In total, the core length was 19.5 m. The sequence above 9.30 m, which is the focus of this study, shows a continuous record up to present, consisting of peat.

Lithostratigraphy

The core sequences were photographed in the field and also in the laboratory. Stratigraphic descriptions were made and the simplified stratigraphy is presented in Table 2. Four main stratigraphic units were separated. The bottom half of the deepest core contained lake sediment, while peat constituted the rest of the core.

Radiocarbon Analyses, Age-Depth Model, and Deposition Time (DT)

Subsamples were selected for ^{14}C dating along the almost 10-m-long core. For ^{14}C dating, 3–4 samples were selected from each 1-m-long core section. One sample was taken at the middle of the section and two samples were taken 10 cm from both ends of the section. Using this sampling procedure, we obtained 29 samples from 10 sections for the first time.

For ^{14}C dating we separated *Sphagnum* leaves and stems to avoid age reversals caused by down penetrating rootlets. We used a cellulose extraction, instead of the commonly used acid-base-acid sequence (Molnár et al. 2013a) to avoid any possible contamination by other organic peat fractions.

First, a physical preparation was performed to separate roots and larger sediment constituents. The subsamples were wet sieved (1 mm to 40 μm Retsch sieve) with doubly deionized water. The remaining material between 40–200 μm fractions was saved for dating. After sieving, the residue was examined using a light microscope in sterile Petri dishes. Visible roots were removed from the samples at this stage.

The next step was the chemical cleaning of the samples. Dry *Sphagnum* samples for AMS dating were prepared using the modified BABAB (base-acid-base-acid-bleaching) method (Nemec et al. 2010) with revised amounts for peat cellulose preparation. After the chemical preparation, samples were redried in freeze-dryer. *Sphagnum* cellulose samples were combusted in a sealed tube using MnO_2 as the oxidizing agent at 550°C (Janovics et al. 2017). The liberated carbon dioxide was cryogenically cleaned and converted to graphite by Fe/Zn reduction (Orsovszki and Rinyu 2015; Rinyu et al. 2015, 2013). The samples were measured on the EnvironMICADAS accelerator mass spectrometry (AMS) at the Isotope Climatology and Environmental Research Centre (ICER, Atomki, Debrecen, Hungary) (Molnár et al. 2013a, 2013b).

For the calibration of the measured conventional radiocarbon ages, CALIB 7.04 was used (Stuiver and Reimer 1993) and IntCal13 calibration curve (Reimer et al. 2013). The age-depth model was obtained using BACON, a Bayesian age modeling software package (Blaauw and Christen 2011). The top of the core (0 cm) was assigned to age AD 2012.

After the primary modeling, more subsamples were taken from under-represented parts of the sequence where more rapid changes have been observed. These subsamples were treated in the same way described earlier. In total 36 ^{14}C samples were measured for the age-depth model (Table 1). The deposition time (DT) was calculated by the age-depth model and expressed as yr cm^{-1} (Bennett 1994). The inverse of deposition time is accumulation rate, calculated as cm yr^{-1}.

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Table 1 AMS ^{14}C dating results with calibrated years (cal yr BP) for Mohos profile. Radiocarbon ages were calibrated with the IntCal13 calibration curve (Reimer et al. 2013) using Calib software version 7.0.4 (Stuiver and Reimer 1993).

Lab ID	Depth (cm)	^{14}C age (yr BP ± 1σ)	Cal age (cal yr BP ± 2σ)
DeA-5064	51–52	343 ± 20	315–409 (0.62)
			420–480 (0.38)
DeA-5237	82–83	510 ± 22	510–546 (1.00)
DeA-5238	110–111	524 ± 21	513–553 (0.97)
			612–619 (0.03)
DeA-5065	150–151	1212 ± 21	1065–1183 (0.94)
			1211–1227 (0.06)
DeA-5428	180–181	1572 ± 22	1408–1527 (1.00)
DeA-5239	210–211	1647 ± 24	1423–1431 (0.01)
			1441–1456 (0.03)
			1521–1612 (0.96)
DeA-5066	252–253	2251 ± 23	2159–2255 (0.64)
			2299–2340 (0.36)
DeA-5412	283–284	2431 ± 22	2357–2499 (0.77)
			2595–2613 (0.05)
			2636–2691 (0.18)
DeA-5413	310–311	2467 ± 22	2383–2390 (0.01)
			2431–2713 (0.99)
DeA-5067	350–351	2912 ± 24	2965–3083 (0.77)
			3090–3145 (0.22)
			3152–3156 (0.01)
DeA-5240	380–381	2971 ± 48	2985–3252 (0.96)
			3294–3327 (0.04)
DeA-5241	411–412	3254 ± 26	3404–3429 (0.09)
			3443–3561 (0.91)
DeA-5773	420–422	3441 ± 24	3633–3731 (0.79)
			3745–3773 (0.06)
			3789–3825 (0.15)
DeA-5774	431–433	3930 ± 24	4259–4268 (0.01)
			4287–4437 (0.99)
DeA-5775	441–443	4034 ± 28	4424–4571 (1.00)
DeA-5068	453–454	4134 ± 27	4539–4540 (0.001)
			4549–4555 (0.008)
			4568–4730 (0.68)
			4733–4744 (0.02)
			4746–4821 (0.29)
DeA-5414	484–485	4229 ± 27	4653–4669 (0.05)
			4703–4757 (0.34)
			4809–4854 (0.61)
DeA-5415	510–511	4464 ± 26	4974–5019 (0.13)
			5029–5086 (0.20)
			5101–5141 (0.11)
			5160–5284 (0.55)
DeA-5242	551–552	4880 ± 30	5587–5655 (1.00)

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Table 1: (*Continued*)

Lab ID	Depth (cm)	^{14}C age (yr BP ± 1σ)	Cal age (cal yr BP ± 2σ)
DeA-5776	560–561	5021 ± 26	5660–5771 (0.51)
			5806–5891 (0.49)
DeA-5777	571–572	6185 ± 31	6985–7172 (1.00)
DeA-5423	582–583	6501 ± 34	7324–7402 (0.38)
			7407–7476 (0.62)
DeA-5782	612–613	6776 ± 34	7580–7672 (1.00)
DeA-5417	660–661	7254 ± 39	7984–7987 (0.005)
			7996–8169 (0.99)
DeA-5418	684–686	7436 ± 39	8182–8346 (1.00)
DeA-5424	711–712	7704 ± 37	8412–8560 (1.00)
DeA-5069	754–755	7928 ± 34	8632–8813 (0.64)
			8825–8872 (0.13)
			8877–8978 (0.23)
DeA-5783	787–788	8251 ± 37	9091–9109 (0.02)
			9111–9327 (0.88)
			9344–9402 (0.1)
DeA-5425	813–814	8389 ± 42	9300–9492 (1.00)
DeA-5778	825–826	8612 ± 46	9520–9685 (1.00)
DeA-5779	838–839	8985 ± 42	9930–9995 (0.15)
			10004–10064 (0.10)
			10122–10236 (0.74)
DeA-5070	850–851	9503 ± 38	10609–10613 (0.004)
			10658–10869 (0.66)
			10950–11071 (0.33)
DeA-5420	888–889	9883 ± 47	11201–11402 (1.00)
DeA-5426	910–911	9985 ± 37	11269–11616 (1.00)

Sample Preparation, Ash Content, and Loss-on-Ignition

Sediment cores were sliced into 1–2 cm thick subsamples for other analyses such as loss on ignition (LOI) and chemistry. Samples were dried at 105°C in an oven for 24 hr. To remove organic and water components, powdered samples were ignited at 550°C in a muffle furnace for 4 hr. Ash content and loss-on-ignition (LOI_{550}) values were calculated after Heiri et al. 2001.

RESULTS AND DISCUSSION

Coring Results, Lithostratigraphy, Age-Depth Modeling Results

The total length of the recovered core was 19.5 m. The sequence above 9.30 m (11,115 ± 42 yr BP), which is the focus of this study, shows a continuous record up to present, consisting of peat with different degrees of humification. A simplified stratigraphy of the sequence (0–930 cm) is presented in Table 2.

Since our goal was to establish a robust chronology for the Mohos peat section, we chose cellulose preparation, so that all ages were determined from the same material, minimizing the potential problems that have been described elsewhere (Kilian et al. 1995; Shore et al. 1995; Brock et al. 2011). We hope to determine what time resolution can be achieved in Holocene studies.

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Table 2 Simplified stratigraphic description of the Mohos peat sequence.

Unit	Depth (cm)	Description
4	0–20	Light brown, slightly humidified peat
	20–24	Dark brown peat
	24–80	Light brown peat
	80–100	Dark brown peat
	100–115	Light brown with pine bark
	115–147	Medium humidified reddish brown peat
	147–161	Light brown peat with pine bark
	161–183	Reddish brown peat
	183–200	Light brown peat
	200–420	Medium humidified dark brown peat
3	420–430	Medium humidified dark brown peat with small fragments of macroremains
	430–580	Medium humidified dark brown peat
2	580–606	Light brown peat with pine bark
	606–660	Reddish brown peat
	660–670	Reddish brown peat with small fragments of macroremains
	670–850	Reddish brown peat
1	850–890	Blackish peat intercalation
	890–910	Brown peat, more compact
	910–930	Lake sediment

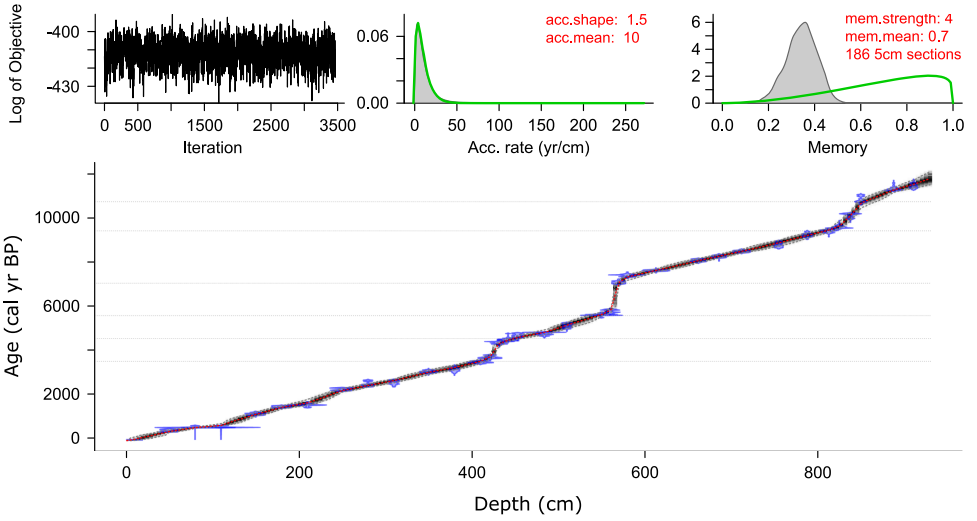


Figure 3 Age-depth model of the Mohos peat section.

In total 36 ^{14}C AMS measurements were obtained, which are reported in Table 1. The yield of the cellulose preparation was 30–48%. All ages were used in the BACON model, and no dates were excluded as outliers. The age-depth model of the sequence is shown in Figure 3. According to the age-depth model, the top 930 cm of the Mohos peat bog covers the range from 2012 AD to 11,770 cal yr BP. The 95% confidence intervals span between 6 yr (at 1 cm) and 954.4 yr (at 566 cm).

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Deposition Time, Loss-On-Ignition, and Ash Content Results

Peat accumulation rate changes were observed along the profile. The sediment deposition time (DT, yr cm^{-1}) varied between 3 and 151 yr cm^{-1}. We were able to separate periods of relatively stable peat bog growth and constant sediment deposition time, as well as five events of significant deposition-rate change. The first period is between 930 and 850 cm (11,770–10,680 cal yr BP) related to lower DT values (16 yr cm^{-1}). During this Early Holocene period organic rich lake sediment is characteristic with moss leaves from the fringing mire and this can be clearly documented in several areas from Europe (Kalnina et al. 2014). This development continues even up to today in the case of the Mohos.

There is another change in DT between 10,680 and 9650 cal yr BP (850–825 cm), where DT increased to about 42 yr cm^{-1}. There is also a lithostratigraphic change at 850 cm. During the period between 9650 and 7340 cal yr BP (820–580 cm), DT was reduced to 9 yr cm^{-1}. During this time period, St. Anna Lake in the neighboring crater showed a different trend with faster accumulation (5–38 yr cm^{-1}), when the lowest water depths were found and the crater was occupied by Sphagnum-bog and bogpools (Magyari et al. 2009).

From 7340 cal yr BP (582 cm), the DT increased by a large amount from 19 to 151 yr cm^{-1} up to 5650 cal yr BP (550 cm). During this time period, St. Anna Lake had increasing water depth (Magyari et al. 2009).

After this time, DT ranged over lower values between 7 and 20 yr cm^{-1} until 4290 cal yr BP (430 cm). A shorter period between 4290 and 3720 cal yr BP (430-420 cm) is characterized by increased DT (55 yr cm^{-1}), where small fragments of macrofossils were observed in the humified peat. Faster accumulation rate (5–38 yr cm^{-1}) and increasing water depth were observed from the St. Anna Lake between 5700 and 3800 cal yr BP (Magyari et al. 2009). Peat deposition in the uppermost part of the sediment (above 3720 cal yr BP) was relatively constant, DT ranged between 3 and 19 yr cm^{-1}.

There was no major change in the loss on ignition and ash content. Loss on ignition content from the Mohos peat bog values are between 80 and 99% and ash content is between 0.02 and

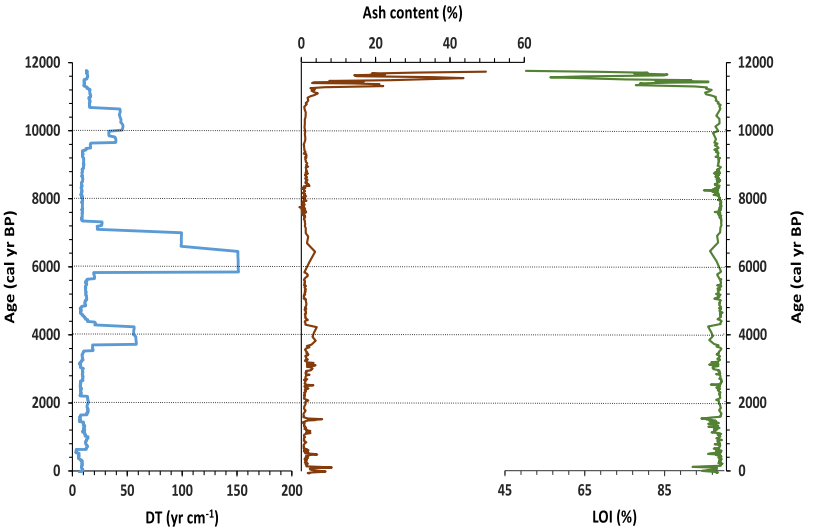


Figure 4 Deposition time (yr cm^{-1}) vs. age (cal yr BP), ash content (%) vs. age (cal yr BP) and loss-on-ignition (LOI_{550}) vs. age curve.

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8%, which is typical for ombrotrophic bogs (Figure 4). Slight changes can be seen based on loss on ignition and ash content, similar to stractigraphic changes.

CONCLUSION

The 910-cm continuous peat profile studied from Mohos peat bog covers the range from 2012 AD to 11,440 cal yr BP. Our excellent record demonstrates that ^{14}C dating of peat deposits is best obtained when cellulose is extracted from the plant material. The use of cellulose avoids problems with incomplete humate extractions with the usual acid-base-acid cleaning.

Fine peat accumulation rate changes (sections with lowest accumulation values) were observed along the profile. Significant changes beginning between ca. 7300–5600 cal yr BP and 4200–3700 cal yr BP with increase in DT suggest lower peat accumulation rates. In further studies we will focus on special intervals to investigate environmental changes during the Holocene. Thanks to the chronological model the past environmental record of Mohos peatbog can be compared to other records on unified calendar time scale.

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