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Impact of climate change on the ecology of the Kyambangunguru Crater Marsh
in southwestern Tanzania during the Late Holocene

Sarah Coffinet^{a1}, Arnaud Huguet^a, Laurent Bergonzini^b, Nikolai Pedentchouk^c, David Williamson^d,
Christelle Anquetil^a, Mariusz Gałka^e, Piotr Kołaczek^e, Monika Karpińska-Kołaczek^{e, f, g}, Amos
Majule^h, Fatima Laggoun-Défargeⁱ, Thomas Wagner^j, Sylvie Derenne^a

^aSorbonne Université, CNRS, EPHE, PSL, UMR METIS, Campus Pierre et Marie Curie, 4 place
Jussieu, 75252 Paris cedex 05, France

^bUniversité Paris Saclay, UPS Univ Paris 11, CNRS, UMR GEOPS, rue du belvédère, Bât 504, 91405
Orsay cedex, France

^cSchool of Environmental Sciences, University of East Anglia, Norwich Research Park, Norwich, NR4
7TJ, United Kingdom

^dSorbonne Université, Institut de Recherche pour le Développement, MNHN, CNRS, UMR LOCEAN,
Centre IRD France Nord, F-93143, Bondy cedex, France

^eDepartment of Biogeography and Paleoecology, Faculty of Geographical and Geological Sciences,
Adam Mickiewicz University in Poznań, Krygowskiego 10, 61-680 Poznań, Poland

^fLaboratory of Wetland Ecology and Monitoring, Faculty of Geographical and Geological Sciences,
Adam Mickiewicz University in Poznań, Krygowskiego 10, 61-680 Poznań, Poland

^gCentre for the Study of Demographic and Economic Structures in Preindustrial Central and Eastern
Europe, University of Białystok, Plac Uniwersytecki 1, 15-420 Białystok, Poland

^hInstitute of Resource Assessment, University of Dar Es Salaam, P.O. Box 35097, Dar Es Salaam,
Tanzania

ⁱUniversité d'Orléans, CNRS, BRGM, UMR ISTO, 1A rue de la Férollerie 45071 Orléans, France

^jHeriot-Watt University, Lyell Centre for Earth and Marine Science and Technology, School of
Energy, Geoscience, Infrastructure and Society (EGIS), Edinburgh, EH14 4AS, United Kingdom

Corresponding author email address: scoffinet@marum.de ; +49 421 218 65740

¹ Present address: Organic Geochemistry Group, MARUM - Center for Marine Environmental
Sciences & Department of Geosciences, University of Bremen, Leobener St. 8., Bremen, Germany

Abstract

Instrumental records of temperature and hydrological regimes in East Africa evidence frequent droughts with dramatic effects on population and ecosystems. Sources of these climatic variations remain largely unconstrained, partly because of a paucity of Late Holocene records. Here, we present a multi-proxy analysis of a 4-m continuous sediment core collected in the Kyambangunguru crater marsh, in southwest Tanzania, covering the last 4000 yrs (cal. BP). We used microscopic (macro-remains, microfossils, palynofacies, pollen), elemental (carbon, nitrogen contents), molecular (br GDGTs, *n*-alkanes) and compound-specific isotopic ($\delta^2\text{H}$ *n*-alkanes) investigations to reconstruct the environmental history of the marsh. The multi proxy record reveals that, 2500 years ago, the marsh underwent a major ecological transition from a lake to a peatland. Temperature and hydrological reconstructions evidence warmer and drier conditions between 2200 and 860 cal. BP, which probably triggered the establishment of a perennial peatland. This study is one of the first combined temperature and precipitation record of Late Holocene in the region and highlights changes in the spatial distribution of the East African climate regimes. Several cold periods are observed, between 3300 and 2000 cal. BP and since 630 cal. BP, the latter corresponding to the Little Ice Age. Moreover, wetter conditions are reported during the Medieval Climate Anomaly in contrast to other north-eastern African records suggesting that Tanzania is located at the transition between two hydro-climatic zones (north-eastern versus southern Africa) and has experienced variable contributions of these two zones over the last millennium.

Keywords

Holocene, Paleoclimatology, Paleolimnology, East Africa, Continental biomarkers, Organic geochemistry, Stable isotopes, Palynology.

1. Introduction

Tropical highlands are major sources of food and freshwater for more than 35 tropical countries (Williamson, 2014). The climate dynamics and variability of these topographically complex environments, however, remain poorly studied. While it has been shown that the Quaternary climatic trends in East Africa were primarily controlled by orbital forcing (e.g. Garcin et al., 2006; Tierney et al., 2008), the shorter scale climate dynamics of this region is largely unconstrained. Notably, mid- to late Holocene records of many East African lakes (e.g. Gasse, 2000; Wanner et al., 2011) suggest rapid and frequent, high amplitude, climatic fluctuations at the centennial scale. These fluctuations and their consequences are not well understood due to a general lack of highly resolved records (Nicholson et al., 2013). Furthermore, the timing and intensity of these events are not always synchronous from site to site (Tierney et al., 2011, 2013). Here, we present detailed records of climate and ecosystem changes from a sequence of sediments covering the late Holocene (the last 4000 years) in the Kyambangunguru marsh. This marsh is located in the Rungwe Volcanic Province (RVP; southwest Tanzania), a highland representing one of the four major food crop producing regions in the country (Majule, 2010).

Marshes and peatlands have a great potential for quantitative high-resolution palaeoclimatic records (Amesbury et al., 2012; Blackford, 2000) notably in the tropics (e.g. Bonnefille et al., 1990; Bourdon et al., 2000; Page et al., 2011; Rucina et al., 2010; Swindles et al., 2018). However, they are highly dynamic ecosystems where the vegetation cover and the hydrology functioning can be totally modified at a centennial scale (Loisel and Yu, 2013). This may complicate the interpretation of climatic proxies, notably those based on biological markers as their fluctuations may be related to ecological, local change rather than regional climatic change. A major challenge in using marsh/peat records as climatic archives is thus to disentangle biological signals linked to dynamic changes of the peatland ecosystem itself from those that are driven by local to regional environmental change (Chambers et al., 2012; Morris et al., 2015). The focus of this study is to investigate the internal, ecological changes within the marsh in the context of regional climatic variations. We aim to retrieve detailed (quantitative) air temperature and (qualitative) hydrological condition records of the late Holocene from the southernmost part of East Africa to test whether rapid and high amplitude climatic events (e.g. Russell and Johnson, 2005; Wanner

et al., 2011) were recorded in this area in comparison to other East African records. Additionally, the multi-proxy approach, combining microscopic observations and geochemical characterization, intends to determine potential feedbacks of these rapid climatic events in the tropical highland wetlands as well as potential human impact in the region.

Analysis of pollen, non-pollen palynomorphs (NPPs), macro-remains, palynofacies and bulk elemental (C and N content) determination was conducted to characterize the ecological states of the wetland, complemented by biomarker-based proxies to determine past variations in air temperature and hydrology. Branched glycerol dialkyl glycerol tetraethers (br GDGTs) and compound specific long chain *n*-alkane hydrogen isotopic composition ($\delta^2\text{H}_{\text{wax}}$) were used for mean annual air temperature and hydrological conditions reconstruction, respectively. Br GDGTs are membrane lipids produced by unknown bacteria (Sinninghe Damsté et al., 2000) whose relative abundances in environmental samples have been shown to correlate with temperature and pH (Weijers et al., 2006, 2009). This enabled the reconstruction of past pH and air temperatures from the br GDGT distribution in sediments, peats and soils (Nichols et al., 2014; Peterse et al., 2011; Weijers et al., 2007a). Long chain *n*-alkanes are constituents of the epicuticular wax layer of leaves (Eglinton and Hamilton, 1967). It has been shown that their hydrogen isotopic composition ($\delta^2\text{H}_{\text{wax}}$) reflects the hydrogen isotopic composition of the water taken up by the plants (e.g. Estep and Hoering, 1980; Sauer et al., 2001; Sessions et al., 1999). Accordingly, they can be used to reconstruct variations in local palaeohydrology as shown in several lacustrine sedimentary archives from the Quaternary and the Holocene in East Africa (e.g. Loomis et al., 2015; Powers et al., 2005; Tierney et al., 2008; Verschuren et al., 2000). The combined use of these two proxies allows distinguishing the temperature from the hydrological signal which has been a major limitation in lake-based East African climatic reconstructions (Verschuren, 2003). Moreover, in settings with high sedimentation rates like marshes, they can offer highly detailed and independent reconstruction of the temperature and the hydrological conditions.

2. Regional setting: the Rungwe Volcanic Province and the Kyambangunguru marsh

The Rungwe Volcanic Province (RVP; SW Tanzania; Fig. 1A), is a large volcanic mountain region (1500 km²) located at the triple junction of the Malawi Rift, Rukwa/Tanganyika Rift and the Usanga Basin in the southern part of the East African Rift System (Fontijn et al., 2010, 2012). The RVP is delimited by the Poroto Mountains in the north, Lake Malawi in the south and the Livingstone escarpment in the west (Fig. 1B). The area is known to be seismically active with volcanic eruptions occurring from the late Miocene (9.2 Ma) to the 19th century, with hot spring activity still found today (Branchu et al., 2005). The region contains three major stratovolcanoes: the Ngozi, Kyejo and Rungwe (Fontijn et al., 2010, 2012). South of these high-altitude sites and north of Lake Malawi lies the Karonga plain. Several monogenic maar-type craters were created during late Pleistocene phreatomagmatic explosions along the Mbaka fault system and are now filled by closed lake hydro-systems (Fontijn et al., 2012; Fig. 1B). The region belongs to the humid equatorial zone of Africa, mainly determined by the migration of the Intertropical Convergence Zone (ITCZ), a key atmospheric feature of tropical atmospheric circulation with low-pressure air masses accompanied by high precipitation. The ITCZ reaches its southernmost position (centred at ca. 15°S, Fig. 1A) in January, resulting in seasonal fluctuations between hot humid conditions from November to May and relatively colder and dry conditions from June to October (Fig. 1C). The RVP is among the most humid regions of Tanzania along with the coastal zone (Basalirwa et al., 1999). It is characterized by a different rainfall distribution with persisting rainfall in April-May. Nivet et al. (2018) showed that the Indian tropical Ocean and the Austral Ocean are the main sources of moisture in the area, with only a minor influence of the Congo Air Mass. Thus, the currently observed variability of the regional rainfall is likely highly impacted by the Indian Ocean Dipole, through Sea Surface Temperature anomalies. Over the last century, climatic trends from the RVP point towards drier conditions associated with a shorter rain season (Williamson et al., 2014) and a continuous increase in temperature ($\approx 1^{\circ}\text{C}$ for the last 100 yr.; Branchu et al., 2005). Typical vegetation of the region includes Zambezian Miombo-type woodland at low altitude and Afromontane vegetation at higher altitude (Garcin et al., 2006, Williamson et al., 2014). In many locations, the woodland has been replaced by diverse crops (banana, rice, cocoa, tea, coffee, maize;

Coffinet et al., 2017; Williamson et al., 2014). The RVP is today one of the main agricultural resources of Tanzania (Majule, 2010).

The Kyambangunguru marsh (9°22' S - 33°47' E, 660 m a.s.l.) is located in one of the numerous maar craters of the RVP, between the Mbaka River and the Mbaka fault. These maar craters are essential water and biodiversity resources for the region. At Kyambangunguru, no human activity has been recorded nor is known within the crater (no land or water use). Human settlement expands in the plains surrounding the volcano (mainly family-scale farming) but not on its slopes. The inner marsh covers about 0.04 km² and its catchment area – limited to the crater slopes elevated ca. 100 m above the water table – is relatively small (0.20 km²; Delalande et al., 2008a, Fig. 1D). According to the Lwifwa Masoko station of the University of Dar es Salaam located at Lake Masoko, 7.5 km to the south east, mean precipitation (P) is up to 2099 mm.yr⁻¹, with April being the most humid month (470 mm in average) and September the driest one (8 mm in average; Nivet et al., 2018). Air temperature fluctuates around 22 °C throughout the year; July is the coldest month (19 °C on average) and November the warmest (25 °C on average). The crater depression is filled with peat like deposits overgrown by marsh-type vegetation and the water level (H) varies around 70 cm of amplitude over the year (Fig. 1C). At the end of the rainy season, the marsh resembles a shallow lake with floating vegetation mats and patches of free water surface (Fig. 1E) while the water table considerably decreases during the dry season (Fig. 1C). On a monthly scale, water level fluctuations (ΔH) correlate with the rainfall (P): $\Delta H = 1.03 P - 18$ (in cm; n = 12; r = 0.97). During the humid season, the marsh water is warm (around 25 °C), low mineralized and slightly acidic (pH around 5.8). On the contrary, during the dry periods, it is characterized by higher mineral concentration as well as pH increase up to around 6.4, because of water evaporation at the surface (Delalande, 2008; Delalande et al., 2008a). The water residence time in the marsh is short (a few months), based on the isotopic water budget of the marsh in- and outputs ($\delta^{18}\text{O}$ and $\delta^2\text{H}$ of H₂O; Delalande et al., 2008a), suggesting a prominent influence of climate in the marsh water budget. The isotopic signature of the marsh water during the humid season ($\delta^2\text{H}\text{-H}_2\text{O} = -7\text{‰}$) is close to the mean annual isotopic signature of precipitation recorded at Lwifwa Masoko station (Nivet et al., 2018). At the end of the dry season, the marsh water isotopic signature ($\delta^2\text{H}\text{-H}_2\text{O}$) becomes more ²H-enriched (between 7 and 16‰, Fig. 1C) demonstrating significant evaporation resulting from the

drier climatic conditions. The slopes of the crater are steep and covered by Zambezian-type (Miombo) forest, dominated by *Brachystegia*, *Uapaca* and *Acalypha* tree species, all common to the region (White, 1983). Shrubs of Rubiaceae and Myriaceae families are present at the edge of the marsh. The vegetation of the marsh is dominated by sedges (*Carex*, *Cyperus*) while floating (*Nymphaea*) and submerged macrophytes are abundant in the depressions filled with water during the rainy season.

3. Materials and methods

3.1. Core retrieval and sampling

A 4-m long core was collected with a Wright corer in the Kyambangunguru marsh in December 2012. The coring process was stopped at 4 m because of the thickness of the Rungwe Pumice tephra (Fontijn et al., 2012) and thus covers the most recent history of the Kyambangunguru wetland. The core was sampled in 1 cm thick slices at the Lwifwa-Masoko station of University of Dar es Salaam and kept at -20 °C until further treatment. 21 samples were chosen for dating while 35 samples were selected every 12 cm to perform the elemental (carbon and nitrogen content), molecular (br GDGTs, *n*-alkanes) and isotopic ($\delta^2\text{H}_{\text{wax}}$) analyses. Total organic carbon (C_{org}) and nitrogen (N) contents were determined after decarbonation by elemental analysis at the Service Central d'Analyse du CNRS, Villeurbanne, France. 105 1 cm-thick samples (ca. every 3 cm) were selected for plant macrofossil analysis. Among these, 44 samples (distributed evenly along the core) were additionally analysed for pollen and non-pollen palynomorphs (NPPs). Within these 44 samples, 12 were used for palynofacies determination. Additionally, 5 surface soil samples (0-5 cm) from the catchment area were collected between the marsh and the top of the crater.

3.2. Absolute chronology and sediment accumulation rate

The chronology of the core is based on 21 Accelerator Mass Spectrometry (AMS) dates performed on bulk Total Organic Matter (TOM; 17 samples) and wood fragments (4 samples; Table 1) all along the core. Samples were subjected to acid-alkali-acid treatment to remove the mineral phase. AMS- ^{14}C and

associated $\delta^{13}\text{C}$ analyses were conducted on aliquots prepared according to the following AMS protocol: burning at 860 °C for 30 min under vacuum, in the presence of a Cu (II)-oxide/Cu (III)-oxide mix and of Ag string. The obtained CO_2 was graphitized on powdered Fe with H_2 at 650 °C for 100 min, and graphite was compressed in analytical pellets. Residual CO_2 gases were used for associated ^{13}C measurements on a SIRA 10 and are expressed in delta notation per mil versus V-PDB (Vienna Pee Dee Belemnite). Graphite preparation and $\delta^{13}\text{C}$ measurements were made at the GEOPS Laboratory (University of Paris-Saclay, France). ^{14}C counting was performed using the AMS facility at the LMC14 laboratory (Laboratoire de Mesure du Carbone 14; Artemis, Saclay; Cottureau et al., 2007). Analytical uncertainties, are $\pm 0.1\%$ for $\delta^{13}\text{C}$ and between 0.2 and 0.5 pMC (percentage of Modern Carbon) for ^{14}C activity.

Calibrated radiocarbon ranges were obtained using OxCal 4.3 software (Bronk Ramsey, 2009) with the ShCal13 (Hogg et al., 2013) and Bomb13SH3 (Hua et al., 2013) atmospheric curves as the calibration set (Table 1). A Bayesian age-depth model was used to establish an absolute chronology based on these calibrated ^{14}C date ranges. The age-depth model was constructed applying a *P_Sequence* function, with the parameters $k_0=1$ and $\log_{10}(k/k_0)=1$, in the OxCal v. 4.3 software (Bronk Ramsey, 1995, 2008). Additionally, boundaries reflecting potential changes in the rate of deposit accumulation were introduced to the model, based on observations of micro- and macrofossils (see section 4.5 and 4.6). These boundaries were defined as follows: (i) 417.5 cm: top of the tephra layer and bottom of the model, (ii) 191 cm: abrupt change from lacustrine to marshland conditions, (iii) 115.5 cm: distinct increase in water table and (iv) 0 cm: top of the core. Computing of the age-depth model led to the exclusion of two samples (SacA40028 and SacA38523). Dates with the lowest individual agreement between the modelled and the calibrated date, i.e. SacA40077 and SacA40076, were also excluded from the calculations until the lowest critical value of the agreement index (A_{model}) suggested by Bronk Ramsey (2008) for model reliability ($A_{\text{model}} = 60\%$) was achieved. Final A_{model} value of the chronology was 63%. The age is presented as a μ (mean) value of the modelled age expressed as calibrated year before present i.e. AD 1950 (cal. BP; Fig. 2), rounded to tens.

A mean value for the sedimentation accumulation rate (SAR), expressed in cm year⁻¹, was determined as the median value of the probability distribution of the modelled age (μ) for each depth (in cm) at which a date was modelled. The applied formula was the following:

$$SAR = \frac{1}{\mu_{depth-0.5} - \mu_{depth+0.5}} \quad (1)$$

3.3. Plant macrofossil analysis

About 5 cm³ of sediment were rinsed with warm water and sieved at 0.25 mm. Macrofossils were studied in transmitted light with a Nikon SMZ800 stereoscopic microscope at a magnification of 10 to 200. Species determination of individual plant macrofossils was performed based on the data from Velichkevich and Zastawniak (2006, 2009). The data were presented as numbers of detected macrofossils and were presented as diagram drawn in the POLPAL software (Nalepka and Walanus, 2003). Analysis was performed at the Adam Mickiewicz University in Poznań (Poland).

3.4. Pollen, non-pollen palynomorphs and microscopic charcoal

Samples for pollen, non-pollen palynomorph and charcoal analysis were prepared using standard laboratory procedures: adding 10% HCl to dissolve carbonates, heating in 10% KOH to remove the humic fraction and at least 24-hour treatment with HF to remove the mineral fraction followed by acetolysis (Berglund and Ralska-Jasiewiczowa, 1986). *Lycopodium* tablet of known number of spores (n=20848, produced by Lund University) was added to each sample for calculation of microfossil concentration (Stockmarr, 1971). Pollen and spores were counted with a biological microscope under 400× and 1000× magnification until the number of at least 500 pollen grains was obtained (Vincens et al., 2003, 2007). Pollen grains were identified using atlases (Gosling et al., 2013), internet-based databases such as African Pollen Database (<http://apd.sedoo.fr/>), and the Universal Pollen Collection (Institut des Sciences de l'Évolution Montpellier; <http://www.palyno.org/>). Non-pollen palynomorphs (NPPs) were identified using available literature (Gelorini et al., 2011; Miola, 2012; van Geel et al.,

2011). The NPP type numbers follow the convention of ‘HdV-number’ and ‘UG-number’, in which acronym ‘HdV’ means Hugo de Vries Laboratory of the University of Amsterdam (The Netherlands), whereas ‘UG’ is Universiteit Gent (Belgium) (Miola, 2012). Percentages of pollen grains originating from forest and savannah communities were calculated as the ratio of an individual taxon and the TPS (total pollen sum); the TPS consists of the sum of AP (arboreal pollen) and NAP (non-arboreal pollen but excludes any taxa originating from aquatic and wetland plants as well as spores and NPPs). Percentages of aquatic and wetland pollen taxa, as well as spores and NPPs, were calculated as the ratio of an individual taxon or NPP type and the TPS enlarged by this taxon or NPP type.

Microscopic charcoal particles (size range 0.02-0.5 mm) were counted on the same microscopic slides as the ones used for pollen counting until the total number of charcoal particles and *Lycopodium* spore standard was at least 200 in each sample (Finsinger and Tinner, 2005). Values are expressed as the charcoal accumulation rate (CHAR_{micro}) in grains cm⁻² year⁻¹ and were calculated based on the following formula proposed by Davis and Deevey Jr.(1964):

$$\text{CHAR}_{\text{micro}} = \text{CHAC}_{\text{micro}} \times \text{SAR} \quad (2)$$

where CHAC_{micro} is the concentration of microscopic charcoal particles (in grains or particles cm⁻³) and SAR is the sediment accumulation rate (in cm year⁻¹). The diagrams were prepared using the POLPAL software (Nalepka and Walanus, 2003) and the analyses were performed at the Adam Mickiewicz University in Poznań (Poland).

3.5. Quantitative palynofacies analysis

Sample preparation included treatment with 50 ml of HF overnight at 40°C followed by 50 ml of HCl for 30 min to eliminate siliciclastic and carbonate minerals. Samples were then rinsed until neutral pH. Quantification of the different types of organic matter (OM) is based on the methodology developed by Graz et al. (2010) using incorporation of a standard solution of *Cupressus* pollen at 10 mg.ml⁻¹ in each sample. Samples were prepared in thin sections before optical investigations using a transmitted light microscope with a 50× magnification. Particle identification was performed using the methodology

described by Boussafir et al. (2012) and Graz et al. (2010). Particles were classified into 3 types: (i) Ligno-Cellulosic tissues (LC) at different stages of degradation comprising fresh tissues detected as translucent LC (tLC), slightly degraded/amorphised LC (saLC) with cell structures that are still partially recognizable and totally degraded/amorphous LC, characterised by red aggregates of amorphous OM (rAOM) as originally described by Graz et al. (2010), (ii) mycelium fragments (myc; Graz et al., 2010) and (iii) planktonic remains (algal organic matter, algOM; Boussafir et al., 2012). Whenever necessary, particle identification was aided using UV excitation. The total mass of each particle type was determined after 40 counting according to Eq. 3:

$$m_{part} = \frac{m_{std} \times A_{part}}{A_{std}} \times \frac{d_{part}}{d_{std}} \quad (3)$$

with m_{part} : mass of the particle; m_{std} : mass of the standard; A_{part} : counted surface of the particle; A_{std} : counted surface of the standard; d_{part} : density of the particle; d_{std} : density of the standard. Densities used for the calculation were determined by Graz et al. (2010). Results are expressed as relative abundance (in terms of mass) of each particle to the total. Sample preparation and analysis were performed at the University of Orléans (France).

3.6. Biomarker analyses

3.6.1. Lipid extraction

After freeze-drying, samples were submitted to a modified Bligh-Dyer extraction as described in Coffinet et al. (2015). The total lipid extract was then separated into three fractions on a silica column with a succession of (i) DCM, (ii) DCM:acetone (2:1, v:v) and (iii) DCM:MeOH (1:1, v:v) followed by pure MeOH as solvents (Coffinet et al., 2015). The apolar fraction containing the *n*-alkanes and the intermediate polarity one (fraction 2) containing the GDGTs were analysed. Prior to analysis, the apolar fraction was further separated on silver nitrate impregnated silica columns (10%, w:w) in Pasteur pipettes (with heptane and then DCM as eluents) in order to purify the linear *n*-alkanes for compound-specific $\delta^2\text{H}$ analysis.

3.6.2. *n*-Alkane analyses

n-Alkanes were analysed at Sorbonne University (Paris, France) by gas chromatography coupled to a mass spectrometer (GC-MS) using an Agilent Network 6890 GC System coupled with a 5973 Mass Selective Detector, with electron impact at 70 eV. 1 µl was injected and separation was achieved using a Restek RXI-5 Sil MS silica capillary column (30 m × 0.25 mm i.d., 0.50 µm film thickness) with He as the carrier gas at 1 ml min⁻¹ flow rate. Initial temperature was set at 50 °C and increased to 320 °C at 4 °C min⁻¹. Samples were injected in splitless mode and the injector temperature was 280 °C.

The average chain length (ACL; Eq. 4) describes the *n*-alkane distribution profile of a sample and is used to determine the predominant origin of the *n*-alkanes. Typically, *n*-alkanes with chain with less than 21 carbon atoms are suggested to be produced by algae and cyanobacteria (e.g. Han et al., 1968) while *n*-alkanes with more than 25 carbon atoms more likely originate from terrestrial higher plants (Eglinton and Hamilton, 1967). *n*-Alkanes with chain length between 21 and 25 carbon atoms are predominantly found in aquatic macrophytes (Ficken et al., 2000).

$$ACL = \frac{\sum C_i \times i}{\sum C_i} \quad \text{where } i \text{ spans from 21 to 35} \quad (4)$$

The carbon preference index (CPI; Eq. 5) is a ratio assessing the relative importance of odd over even homologues and reflects the degree of maturity (organic matter degradation) of a sample. Immature samples have very high CPI (>> 1; Killops and Killops, 2005). Unusually low CPI (below 3) in recent sediments are generally considered as polluted by a source of mature organic matter (petroleum, wood burning; Bray and Evans, 1961).

$$CPI = 0.5 \times \left(\frac{\sum C_{odd \ 25-33}}{\sum C_{even \ 24-32}} + \frac{\sum C_{odd \ 25-33}}{\sum C_{even \ 26-34}} \right) \quad (5)$$

The P_{aq} index (Eq. 6) was developed by Ficken et al. (2000) and is a proxy for the relative contribution of emergent/terrestrial macrophytes compared to submerged/floating ones. Contribution of submerged and floating macrophytes is considered as high when P_{aq} values are higher than 0.4 and insignificant when P_{aq} values are lower than 0.1.

$$P_{aq} = \frac{C_{23} + C_{25}}{C_{23} + C_{25} + C_{29} + C_{31}} \quad (6)$$

n-Alkane hydrogen isotopic composition ($\delta^2\text{H}$ *n*-alkane) was measured at Newcastle University (United-Kingdom) using a Delta V+ isotope-ratio mass spectrometer (IRMS, Thermo Fisher) connected to a GC Ultra Trace (Thermo Fisher), a Finnigan GC Combustion III (Thermo Fisher) and a high temperature conversion (HTC) system set up at 1400 °C. The GC temperature was set to start at 50 °C and then to raise to 250 °C at 15 °C min⁻¹ and from 250 °C to 320 °C at 5 °C min⁻¹. Temperature was then held at 320 °C for 15 min. Every sample was analysed in duplicate and the ²H/¹H ratio was reported on the V-SMOW (Vienna standard mean ocean water) scale and expressed in delta per mil (‰). A mixture of *n*-C₁₆ to *n*-C₃₀ alkane standard and 5 α androstane standard (A. Schimmelmann, Indiana University) was run at the beginning and at the end of each sequence. Standard error of the measurements of the individual long chain *n*-alkanes (C₂₃-C₃₁) from this standard mix ranged between 0.3‰ and 1.1‰.

3.6.3. GDGT analysis

GDGTs were analysed at Sorbonne University (Paris, France) with a high-pressure liquid chromatography coupled to a mass spectrometer with an atmospheric pressure chemical ionization source (HPLC-APCI-MS, Shimadzu LCMS-2020). Separation was achieved with a Prevail Cyano column (2.1 mm x 150 mm, 3 μm ; Alltech, Deerfield, IL, USA) at 30 °C, using a mixture of hexane and isopropanol at 0.2 ml min⁻¹ according to Coffinet et al. (2015). Elution began at 99% A/1% B for 5 min followed by a linear gradient to 98% A/2% B in 45 min. A second linear gradient led to a mixture of 90% A/10% B in 10 min, maintained for 10 min and returned to the initial conditions (99% A/1% B) in 14 min, maintained for 10 min. Injection volume was 10 μl . Single ion monitoring (SIM) of the $[\text{M}+\text{H}]^+$ ions was used to detect the GDGTs.

Mean annual air temperatures (MAAT) were estimated using the calibration specifically developed for East African lakes by Loomis et al. (2012):

$$\text{MAAT} = 22.77 - 33.58 \times f(\text{III}) - 12.88 \times f(\text{II}) - 418.53 \times f(\text{IIc}) + 86.43 \times f(\text{Ib}) \quad (7)$$

where $f(x)$ is the fractional abundance of the compound *x* relative to the total br GDGTs and the roman numerals correspond to the different br GDGT compounds according to Weijers et al. (2007b) numbering.

pH was calculated using the soil calibrations developed by Tierney et al. (2010b) and based on the cyclisation ratio of br GDGTs (CBT; Weijers et al., 2007b):

$$\text{pH} = 10.32 - 3.03 \times \text{CBT} \quad (8)$$

$$\text{CBT} = -\log\left(\frac{[\text{Ib}] + [\text{IIb}]}{[\text{I}] + [\text{II}]}\right) \quad (9)$$

where the roman numerals correspond to the different br GDGT compounds according to Weijers et al. (2007b) numbering.

4. Results

4.1. Lithology

The bottom of the core corresponds to a pumice-rich tephra. The overlaying deposits (ca. 418 to 200 cm) consist of an alternating layer of peat, organic gyttja and silty clay material, with charcoal-rich layers (Fig. 2). Above 200 cm, the sediment mostly consists of peat at diverse stages of decay. Large amounts of higher plant macrofossils are observed at depth ranging between 175 and 75 cm, while the decomposition of plant fragments is more pronounced from 75 cm to the top of the sequence. The upper clayey part of the sequence contains two additional tephra layers at 65.5-63 cm and at 57-51 cm (Fig.2).

4. 2. Chronostratigraphy, age-depth model and sedimentation accumulation rate

As shown in Table 1 and Figure 2, dates were obtained all along the core and generally fit with the stratigraphy. Nevertheless, two age reversals are observed at the base of the core, based on Total Organic Matter (TOM) measurements (Fig. 2). The first one occurs between 371.5 and 402 cm depth (SacA44028) and the second corresponds to the bottom tephra layer of the core between 417.5 and 420.5 cm depth (SacA38523; Table 1). This could be attributed to stratigraphic misplacement or cross-contamination during coring. Therefore, these dates were not included in the age model. All the wood remain-based ages are very close to those from bulk TOM or slightly older (mean standard deviation

between the bulk TOM and the wood remain age is +2.3%). This minor mismatch could be due to the additional transport time of the wood remains from the catchment into the marsh sediment while the bulk TOM signature is expected to be predominantly autochthonous (see sections 4.4 to 4.6). Alternatively, “younger” TOM ages could result from microbial activity or organic matter derived from root development occurring after sedimentation (Trumbore, 2009). The $\delta^{13}\text{C}$ values obtained after sample preparation on residual CO_2 vary from -20.0‰ to -29.5‰ along the core (average value -25.3‰; Tab. 1). This range is compatible with organic carbon originating from the vegetation, likely C3 type plants, and consistent with $\delta^{13}\text{C}$ values measured at the nearby Lake Masoko (Gibert et al., 2002). Compared to the mean TOM $\delta^{13}\text{C}$ value (-24.8‰), the mean wood $\delta^{13}\text{C}$ value is lower (-27.2‰) also in agreement with a C3 type plant signature.

The investigated sediment sequence from Kyambangunguru shows continuous sedimentation of ca. 4080 calibrated years, spanning from ca. 4020 and -60 cal. BP (Fig. 2). The “apparent” sediment accumulation rate (SAR) ranges from 0.03 to 0.18 cm yr^{-1} (i.e., each cm thick sample of the profile records 6.3 to 33 years). The highest SAR was recorded in the bottom section of the profile (between 0.10 and 0.18 cm yr^{-1}), in the 417-191 cm section (from 4015 to 2280 cal. BP). Intermediate SAR values, ranging between 0.07 and 0.11 cm yr^{-1} , were recorded between 191 and 8 cm (from 2280 to 130 cal. BP), whereas the lowest SAR (0.03-0.06 cm yr^{-1}) was recorded in the top section of the profile between 8 and 0 cm (from 130 to -60 cal. BP). The σ error of the modelled dates ranged between 0.2 (top of the profile) and 53 years.

4.3. Palynofacies

The Kyambangunguru core is largely dominated (ca. 80% of the OM throughout the core) by lignocellulosic tissues (LC) from vascular plants (Fig. 3). Microscopic investigation of the LC reveals 3 stages of degradation. On average, fresh, well-preserved, tLC tissues represent 19% of the total OM (Fig. 3), while slightly degraded, saLC particles are the most abundant type of OM (ca. 39% on average) in the core. Amorphous rAOM represents ca. 11% of the total LC OM with an increase to 20% in the section from ca. 3140 to 830 cal. BP (300-62.5 cm). In addition to LC, fungal mycelia are observed in the layer

spanning the period between 239 and 60 cm (ca. 2670 to 800 cal. BP). Grey cell fragments and granular amorphous OM, related to planktonic-derived material, are found throughout the core (Fig. 3), but are particularly abundant at the base (below 330 cm, i.e. ca. 3360 cal. BP) and at the top of the profile (from 89.5 to 0 cm, ca. 1150 cal. BP to modern).

4.4. Plant macrofossil analysis

Plant macrofossils are dominated by macrophytes (sedges, submerged/floating plants and algae) and wood remains. *Eleocharis* sp. is present all along the core, while *Nymphaea* sp. (floating macrophyte), disappeared between 160 and 100 cm (ca. 1960 and 1270 cal. BP; Fig. 4). In addition, in the layer between 415.5 and 281.5 cm (ca. 4000 to 3000 cal. BP), macrofossils are composed of remains of algae (*Nitella* sp., Fig. 4) and submerged/floating plants (*Potamogeton* sp. and *Caldesia parnassiflora*; Fig. 4). These species are replaced by *Juncus* sp. from 239.5 to 139.5 cm (ca. 2670 to 1730 cal. BP). During this interval, there is a relative increase in abundance of wood remains in comparison to the other sections of the core. *Nitella* sp. together with *Carex* sp. and *Chara* sp. are the dominant remains found in the sequence from 139.5 to the surface (1730 cal. BP to modern; Fig. 4). At 105 cm (ca. 1330 cal. BP), *Aldrovanda vesiculosa* seed was found.

4.5. Pollen, NPPs and microcharcoal

265 taxa of pollen and spores as well as non-pollen palynomorphs (NPPs) – organic walled microfossils which are not pollen – were identified by palynological analysis. The results of this analysis were separated into two groups: indicators of terrestrial vegetation, i.e. vegetation surrounding the lake/marsh and indicators of aquatic vegetation reflecting mainly lake-marsh vegetation. Three pollen zones reflecting compositional changes in terrestrial (woodland and open land) communities (labelled as KY/tp-1 to -3 with tp: terrestrial pollen; Fig. 5) were defined. Additionally, the KY/tp-1 zone was subdivided into three subzones (KY/tp-1a-c). Pollen and NPPs reflecting lake/marsh vegetation changes enabled the establishment of 6 zones (KY/lpn-1-6 with lpn standing for local pollen and NPPs; Fig. 6).

4.5.1. Terrestrial vegetation

The KY/tp-1 zone (416–303 cm; ca. 4000–3160 cal. BP) is characteristic of the highest percentages of *Acalypha*, Moraceae, *Macaranga* and *Piptadenia/Piptadeniastrum/Entada* (Fig. 5). Moraceae and *Macaranga* pollen reached their maxima (27–28% and 11%, respectively) at 353–333 cm (ca. 3520–3380 cal. BP). Poaceae pollen increased until 397 cm (ca. 3850 cal. BP) when they distinctly decreased while Proteaceae pollen revealed a first maximum at 378 cm (ca. 3700 cal. BP). During the KY/tp-1b subzone (353–333 cm; ca. 3520–3380 cal. BP), Poaceae percentages fell to their minimum values (8%). During the KY/tp-1c subzone (333–303 cm; ca. 3380–3160 cal. BP) pollen values of Moraceae and *Macaranga* dropped simultaneously with the rise in Poaceae percentages and the appearance of palm pollen (*Elaeis guinensis* and *Raphia* type). During the entire KY/tp-1 zone charcoal (0.02–0.5 mm), accumulation rate (CHAR) was high and relatively stable without any distinct maximum (8450–37940 particles cm⁻² yr⁻¹; Fig. 5).

The KY/tp-2 zone (303–63 cm; ca. 3160–830 cal. BP) was characteristic of the highest values of *Uapaca* (15–24%; 162.5–133.5 cm; ca. 1990–1660 cal. BP) in the profile. However, at 123–113.5 cm (ca. 1540–1430 cal. BP) they substantially declined, simultaneously to a rapid increase in Poaceae values. At the beginning of the zone, *Raphia* pollen increased rapidly (303–283 cm; ca. 3160–3010 cal. BP; up to 15.5%). Values of Proteaceae increased from 234.5 cm (ca. 2630 cal. BP) and reached the maxima after 113.5 cm (ca. 1430 cal. BP). Simultaneously, or slightly prior to the Poaceae maximum in this zone (123–113.5 cm ca. 1540–1430 cal. BP), values of *Ricinus communis* and *Macaranga* increased (142.5 cm; ca. 1760 cal. BP), whereas percentages of *Nauclea* type (123 cm; ca. 1540 cal. BP), *Elaeis guinensis* (143 cm; ca. 1770 cal. BP), *Raphia* type (132.5 cm; ca. 1650 cal. BP) and later Rutaceae (113.5 cm; ca. 1430 cal. BP) decreased substantially or disappeared. Among pollen taxa related to arboreal plants occupying montane forest zones, *Apodytes* cf. *dimidiata*, *Olea*, *Podocarpus* and *Prunus africana* type were the most common. During the KY/tp-2 zone, three distinct phases of increase in CHAR values, potentially related to an increase in fire activity, were identified. These occurred at 297.5, 248.5 and 117.5 cm (ca. 3120, 2740, and 1480 cal. BP respectively) and were intersected by periods with CHAR

values lower than during the KY/tp-1 zone. From 83 cm (ca. 1070 cal. BP), CHAR values started to increase gradually.

The KY/tp-3 zone (63–5 cm; 830 to 60 cal. BP) was characterized by the highest percentages of Poaceae (67–90%), the continuous presence of *Ricinus communis* and distinct drops of Proteaceae and *Uapaca*. At 50 cm (ca. 690 cal. BP), *Syzygium* pollen value revealed a substantial increase (up to 16%). In general, CHAR values were higher than in the previous zone and two distinct maxima of their values were recorded at 50 and 10 cm (ca. 690 and 160 cal. BP).

4.5.2. Aquatic/marsh vegetation

During the KY/lpn-1 zone (416–383 cm; ca. 4000–3740 cal. BP) the algae *Tetraedron trigonum* type, *Tetraedron incus/caudatum* and *Coelastrum reticulatum* reached their maxima (Fig. 6). These algae taxa rapidly declined between 412 and 393 cm (ca 3970 and 3820 cal. BP). Along the KY/lpn-2 zone (383–303 cm; 3740–3160 cal. BP), Alismataceae (cf. *Caldesia*) was regularly observed (0.8–6.5%). Pollen of Nymphaeaceae (*Nymphaea* type) and their epidermis (UG-1241), *Potamogeton* and algae such as *Scenedesmus*, *Tetraedron minimum*, *Pediastrum* undiff., *Pediastrum angulosum* and *Botryococcus* were frequent during these two zones. At 323 cm (ca. 3310 cal. BP), *Nymphaea* type percentages increased simultaneously with a decline in *Potamogeton*. The KY/lpn-3 zone (303–193 cm; ca. 3160–2300 cal. BP) was characterized by an increase in Cyperaceae pollen and tissue fragment percentages (rise in fungal NPP UG-1176 and UG-1197) and a simultaneous drop in *Scenedesmus* and *Tetraedron minimum*.

The KY/lpn-4 zone (193–113 cm; ca. 2300–1430 cal. BP) was characterized by a prominent increase in Cyperaceae percentages (12–77%). The spores of *Lycopodiella caroliniana* were regularly present (0.2–18%) together with amoeba *Assulina muscorum*. Between 143 and 123 cm (ca. 1770 and 1540 cal. BP), monolete spores reached their maximum values in the core (64–69%). Values of *Nymphaea* type, UG-1241 (*Nymphaea* tissues), *Potamogeton*, Alismataceae (cf. *Caldesia*), *Pediastrum angulosum*, *Botryococcus* and *Scenedesmus* dropped markedly. NPPs of fungal origin increased distinctly, notably UG-1197 and *Entorrhiza* in the deeper part of the zone and HdV-172, UG-1077, UG-1176 and UG-1107 in the upper part.

The KY/lpn-5 (113–46.5 cm; ca. 1430–650 cal. BP) and KY/lpn-6 (46.5–5 cm; 650 to 60 cal. BP) zones were characterized by the increase in frequency of algae, mainly *Botryococcus*, *Scenedesmus* and *Pediastrum angulosum* and, among submersed macrophytes, of *Nymphaea* type pollen and their related tissues (UG-1241). In the KY/lpn-5 zone, Hallorhagaceae appeared (1.5–24%). *Entorrhiza* and fungal type UG-1107 reached maxima in the profile (59–76%). In the KY/lpn-6 zone, Alismataceae (cf. *Caldesia*) and *Potamogeton* became more frequent while Cyperaceae pollen percentages distinctly dropped.

4.6. Elemental analysis

C_{org} content is high throughout the core (39.0 - 57.8%; mean 51.0% ± 6.2; Suppl. Table 1), except in the tephra layer at the base of the core (5%). Total nitrogen (TN) varies from 1.5 to 3.6% except for the tephra layer (0.2%). Slightly lower TN values are observed between 199.5 and 100 cm (ca. 2350 and 1270 cal. BP; ca. 2%, Suppl. Table 1) leading to higher C/N ratios that range from 16 to 38 (Fig. 7 and Suppl. Table 1). In the other sections of the core, C/N ratios are relatively invariant at ca. 15.

4.7. Br GDGT abundance and distribution

Br GDGTs are abundant throughout the core (mean 121.5 ± 81.3 µg g⁻¹ of dry wt. peat), with maximal concentration observed at 150.5 cm (ca. 1850 cal. BP; Suppl. Table 1). CBT varies between 0.53 and 2.01 with a mean value of 1.31 and is higher above 180.5 cm (ca. 2180 cal. BP; 1.41-2.01; Suppl. Table 1). MBT is comprised between 0.65 and 0.89, with a mean value of 0.77 (Suppl. Table 1). The highest MBT values (>0.80) are found between 194 and 45 cm (ca. 2310 and 630 cal. BP).

4.8. *n*-Alkane distribution and their δ²H composition

Mid- to long-chain *n*-alkanes (>C₂₁) dominate most of the samples, with C₂₃ and C₂₅ being the most abundant (on average 23% and 21%, respectively; Suppl. Fig. 1). Two *n*-alkane distribution patterns are

observed in the core (Suppl. Fig. 1), defined as patterns A and B. Pattern A was identified in the sections from 417 to 180 cm and from 30 cm to the surface (ca. 3670 -2300 cal. BP and ca. 430 cal. BP-modern respectively). It is dominated by odd numbered *n*-alkanes, the most abundant one being C₂₃, with a decreasing trend in relative abundance from C₂₃ to C₃₁. Pattern B (Suppl. Fig. 1) was observed in the sections from 193 to 29.5 cm (ca. 2300 to 430 cal. BP). This distribution is characterized by a flattening of the *n*-alkane profile. The C₂₉⁺ *n*-alkanes (up to C₃₅ in some samples) as well as the C₂₃⁻ *n*-alkanes, especially C₁₉, increase while the C₂₃, C₂₅ and C₂₇ decrease, in comparison to pattern A. In pattern B, even numbered *n*-alkanes are also found in larger amount than in pattern A.

The CPI index confirms a strong odd-over-even predominance throughout the core (mean 8.1±3; Suppl. Table 2) while the ACL index varies between ca. 25 and 27 (Fig. 7; Suppl. Table 2). The P_{aq} index is systematically higher than 0.4, ranging between 0.50 and 0.85 (Fig. 7; Suppl. Table 2).

The δ²H values of odd mid to long chain *n*-alkanes (C₂₃-C₃₁) varies between -96‰ and -172‰ (Suppl. Table 2) and are higher in the deepest part of the core, between 417 and 391 cm (ca. 4010 and 3800 cal. BP; between -96‰ and -122‰; Suppl. Table 2). Long chain *n*-alkanes (C₂₉ and C₃₁) are more ²H-enriched (-130‰ on average; Suppl. Table 2) than mid chain (C₂₃ and C₂₅; -150‰ on average; Suppl. Table 2) compounds.

5. Discussion

The combination of palaeobotanical, elemental and molecular analyses enabled the reconstruction of the Holocene ecological history of the Kyambangunguru wetland together with regional climate. The proxy analyses revealed the presence of three major phases in the Kyambangunguru wetland ecosystem development over the last 4.0 ka cal. BP for which the interaction with regional and global climatic changes are discussed. The chronology of the sediment record was compared to published records of the region (Filippi and Talbot, 2005; Fontijn et al., 2012; Garcin et al., 2007) showing overall good consistency. The tephra layer at the base of the core, dated at ca. 4.1 ka cal. BP, corresponds to the Rungwe Pumice deposits identified at Lakes Masoko and Malawi and dated at 4.3 ka cal. BP and 4.3 to

3.6 ka cal. BP, respectively. The second tephra observed at ca. 0.9 ka cal. BP in the Kyambangunguru core can be related to the Aphyric Pumice deposits observed at 1.2 ka cal. BP in the Lake Masoko sediment record and between 1.1 and 0.6 ka cal. BP in the Lake Malawi one. The last tephra layer identified at Kyambangunguru is harder to define. It is dated at ca. 0.8 ka cal. BP and could either correspond to the Aphyric Pumice described earlier or to the Ngozi Tuff, even though this event was dated between 0.5 and 0.3 ka cal. BP at Lakes Masoko and Malawi. This regional comparison, even if it stresses its inherent time uncertainty, further validates the age-model used in this study.

5.1. Ecosystem, air temperature and precipitation variability at the Kyambangunguru crater marsh

Three units (labelled as Units I-III; Fig. 7), representing the major developmental phases of marshland ecosystem over the last 4.0 ka cal. BP, were distinguished based on the local plant community changes inferred from pollen, non-pollen palynomorph and plant macro-fossil analyses. In addition, the origin of the OM and its degree of preservation were determined using the palynofacies and C/N records (Bourdon et al., 2000) while the distribution of br GDGTs allowed reconstructing the pH of the site, following the approach described by Weijers et al. (2007b) and Tierney et al. (2010b). For the climatic reconstruction, the mean annual air temperature (MAAT) and the hydrological conditions over the last 4.0 ka cal. BP years were reconstructed using the br GDGT distribution and the hydrogen isotopic values of the *n*-alkanes ($\delta^2\text{H}_{\text{wax}}$), respectively. Br GDGT-derived MAAT were calculated with the East African lacustrine calibration developed by Loomis et al. (2012), i.e. Eq. 7, which takes into account *in situ* production of br GDGTs in lakes, as discussed in the Suppl. Information. The $\delta^2\text{H}_{\text{wax}}$ was determined as the weighted average of the $\delta^2\text{H}$ values of the C_{23} and the C_{25} *n*-alkanes which are expected to have recorded mainly the wetland water isotopic composition ($\delta^2\text{H}\text{-H}_2\text{O}$) variations during the accumulation of the sedimentary sequence (as discussed in the Suppl. Information). Because the study site has no outflows, the $\delta^2\text{H}\text{-H}_2\text{O}$ variations depend essentially on the rates of precipitation and evaporation and the $\delta^2\text{H}_{\text{wax}}$ can be interpreted as changes in the precipitation to evaporation ratio (P/E ratio; Gonfiantini, 1986; Sachse et al., 2004).

5.1.1. Unit I: ca. 4.0 – 2.3 ka cal. BP (417–193 cm) – shallow lake, cold and wet conditions

The palaeoenvironmental proxies suggest a relatively stable and persistent lake environment during this period. Planktonic remains are observed in the palynofacies (Fig. 3) and in the non-pollen palynomorph profile (NPP; Fig. 6) and the C/N values (ca. 15; Fig. 7) indicate a mixed aquatic and terrestrial source of the OM (Meyers, 1997). The GDGT derived-pH of ca. 7 (Fig. 7) is consistent with current pH values of the RVP crater lakes (ranging between 5.8 and 8.7; Delalande, 2008), further supporting the occurrence of a lake at this time. The high abundance of macro- and microremains (Figs. 4 and 6) from algae (*Nitella* sp.) and submerged/floating macrophytes (*Potamogeton* sp. and *Nymphaea* sp.) and the relatively high P_{aq} values (0.7-0.8; Ficken et al., 2000) suggest that the lake was relatively shallow and its water column likely harboured abundant macrophyte vegetation. The growth of *Caldesia parnassifolia* for instance may indicate a water depth lower than one meter (Gupta and Beentje, 2017; Sinkevičienė, 2016). In addition, the NPP record reveals a characteristic pattern of successive and rapid decline of *Tetraedron trigonum* type, *Coelastrum reticulatum* and *Tetraedron incus/caudatus*, all before 3.8 ka cal. BP (Fig. 6). This reduction is possibly related to the spread of *Nymphaea* whose floating leaves may have limited the light availability, although it did not seem to impact *Tetraedron minimum* and *Scenedesmus*, which might be more resistant species. The second stage of microalgal community retreat, which affected *Scenedesmus* and *Tetraedron minimum*, occurred at ca. 3.2 ka cal. BP and seem to have also been stimulated by *Nymphaea* expansion. Simultaneously, an increased input of fungal remains, UG-1176 and UG-1197, may indicate spreading of emerged marsh plants around the lake. This is further supported by the high C_{org} values (> 50%; Suppl. Table 1) and the predominance of lignocellulosic-derived OM in the palynofacies record partly originating from *Cyperus* (Laggoun-Défarge et al., 2008a) according to microscopic observation (Fig. 3).

Unit I is marked by an abrupt decrease in mean annual air temperature (MAAT) of about three degrees (from 26 °C to 23 °C, Fig. 8) at ca. 3.9 ka cal. BP. The MAAT continues to decrease by another three degrees to 20 °C at 2.7 ka cal. BP (Fig. 8). This trend is synchronous with the “3.3–2.5 ka. BP” cold Holocene event introduced by Wanner et al. (2011) which was also observed – to a lower extent – at ca.

4.0 – 3.0 ka cal. BP in Lakes Tanganyika and Malawi (Powers et al., 2005; Tierney et al., 2008; Fig. 8) and in Lakes Turkana and Challa (Berke et al., 2012; Sinninghe Damsté et al., 2012). At the same time, the $\delta^2\text{H}_{\text{wax}}$ record shows more negative values until ca. 3.0 ka cal. BP (up to -168 ‰; Fig. 9), which can be interpreted as an increase in the P/E ratio either due to higher precipitation or/and lower evaporation rates. A wetter environment is also inferred from the terrestrial pollens, with a decrease in pollen supply from grassland communities from ca. 3.7 to 3.4 ka cal. BP and an expansion of mountain forest and Zambezian Miombo woodland communities (Moraceae and *Macaranga* at ca. 3.5 – 3.4 ka cal. BP, *Uapaca* optimum, presence of *Apodytes cf. dymidata*, *Olea*, *Podocarpus* and *Prunus africana*). Wetter conditions at the same period were also inferred from a pollen record at Lake Masoko (Vincens et al., 2003). Notably, a synchronous *Uapaca* pollen optimum occurred both at Masoko and Kyambangunguru (after ca. 3.1 ka cal. BP, Vincens et al., 2003). $\delta^2\text{H}_{\text{wax}}$ values remain negative during unit I, although two positive peaks in $\delta^2\text{H}_{\text{wax}}$ are noticeable at ca. 3.6 and 2.8 ka cal. BP possibly indicating brief dry events interrupting the overall wetter period. These dry events are in agreement with observations by Russell et al. (2003) and Russell and Johnson (2005) at Lake Edward (Uganda; Fig. 9). Peaks in Mg content in calcite at 3.6 and 2.8 ka cal. BP were indeed linked to evaporative concentration of the lake (increase in the $[\text{Mg}^{2+}]/[\text{Ca}^{2+}]$ ratio in lake water) in response to short but pronounced drought events.

5.1.2. Unit II: 2.3 – 1.4 ka cal. BP (193–113 cm) – marsh/peatland formation under sustained warm and drier conditions

An abrupt, environmental change occurred at ca. 2.3 ka cal. BP leading to the establishment of a peatland with variable hydrological conditions (Fig. 7). The onset of unit II is marked by an apparent decrease in the sedimentation rate from 0.13 cm yr⁻¹ to 0.08 cm yr⁻¹ (Fig. 2), an increase in the Cyperaceae tissue and ferns (monolete spores) accumulation and a strong increase of the C/N ratio (up to 40), all evidencing peat soil formation. High C/N ratios (usually > 30 and sometimes up to 100) are indeed typical for peatlands, due to the high preservation of the OM in relation to the prevailing acidic and anoxic conditions (Laggoun-Défarge et al., 2008b; Meyers, 1997). Simultaneously, a substantial decrease or almost total disappearance of algae (*Scenedesmus*, *Pediastrum*, *Botryococcus* and

Tetraedron minimum) and aquatic plants such as *Nymphaea* spp. (Fig. 6) reflect a substantial fall in the water table. However, irregular appearances of these taxa, as well as of the macroalgae *Nitella* sp., indicate episodic inundation of the wetland (Fig. 4). The main constituents of the peatland community were *Juncus* sp., *Eleocharis* sp., ferns and perhaps other Cyperaceae species (Figs. 4 and 6). Permanent waterlogged conditions, and low pH (down to 5.2, Fig. 7) contributed to the establishment of plant taxa restricted to humid acidic environments, such as *Drosera*, *Lycopodiella caroliniana* and *Aldrovanda vesiculosa* (Figs. 4 and 6; (Gałka et al., 2015), and the appearance of protists frequently found in peatlands, such as the testate amoeba *Assulina muscorum* (van Geel, 1978). The increase in reddish amorphous OM (rAOM) and the appearance of fungal mycelia at that time (Fig. 3) also suggest the start of a terrestrialization process and the development of vascular plants within the marsh (Bourdon et al., 2000). The increase in wood remains in the macrofossil profile, related to the development of trees and shrubs at the edges of the marsh or even in the marsh itself, supports this interpretation.

Unit II displays the highest $\delta^2\text{H}_{\text{wax}}$ values of the core (up to -136 ‰; Fig. 9), suggesting low P/E ratio and thus the driest period of the last 4.0 ka cal. BP, consistent with several East-African records – Lake Edward (Uganda), Lake Turkana (Kenya), Lake Tanganyika (Tanzania) (Nash et al., 2016 and references therein). A major drought event is widely described at ca. 2.0 ka cal. BP, followed by a second period between ca. 1.7 and 1.0 ka cal. BP, during which successive minor drought events occurred (Alin and Cohen, 2003; Russell et al., 2007; Russell and Johnson, 2005; Verschuren and Charman, 2008). In the Kyambangunguru $\delta^2\text{H}_{\text{wax}}$ record, a positive excursion that could be interpreted as a drought event is observed at ca. 2.2 ka cal. BP. However, the major dry period seems to have occurred later, between ca. 1.7 and 1.4 ka cal. BP (centred at 1.5 ka cal. BP, Fig. 9). Around the same time (ca. 1.7 ka cal. BP), *Uapaca* pollen suddenly declined and fire activity increased, which contributed to the opening of the woodland canopy and a spread of the grassland communities (Fig. 5). The same abrupt changes are noticeable in charcoal and pollen data at Lake Masoko (Thevenon et al., 2003; Vincens et al., 2003) and in the Amboseli basin in Kenya (Rucina et al., 2010). Alternatively, these vegetation changes could have been induced by enhanced human activity. Indeed, a first spread of human settlement is considered to have occurred during the Late Iron Age, around 1.5 ka cal. BP (Marchant and Taylor, 1998; Vincens et al., 2003). However, several sites in East Africa (Lakes Turkana, Tanganyika, Naivasha, Challa, Edward

and Sacred Lake; see references in Marchant et al., 2018) support a widespread increase in aridity as the main cause explaining the sedimentological evidence of droughts, as discussed by Marchant et al. (2018). Notably, we promote that $\delta^2\text{H}_{\text{wax}}$ records, such as this study and Konecky et al. (2014), could help disentangling the human impact from the climatic one as they should only be marginally impacted by human activities. According to the br GDGT data, MAAT remained high during unit II, oscillating around 21.5-22 °C (Fig. 8), suggesting a dry and warm period during unit II. During the same period, higher temperatures are also reported in the records from Lake Challa (Sinninghe Damsté et al., 2012), Lake Turkana (Berke et al., 2012) and Lake Tanganyika (Tierney et al., 2008) (Fig. 8).

5.1.3. Unit III: ca. 1.4 ka cal. BP – modern (113–0 cm) – periodically flooded marsh and transition to colder conditions

At the onset of Unit III, an increase in the water table led to the reappearance of microalgae assemblages (mainly composed of *Botryococcus*, *Scenedesmus* and *Pediastrum angulosum*), and macrophytes such as *Nymphaea*, Alismataceae and Haloragaceae at ca. 1.4 ka cal. BP. However, until ca. 0.7 ka cal. BP, high percentages of Cyperaceae and of *Entorrhiza* spores are observed, which indicate that the peatland was subjected to pronounced water table fluctuations but without open water stages. Indeed, *Entorrhiza* is a genus of parasites that infect the roots of the Juncaceae (rush) and Cyperaceae (sedge) families when they are no longer in water (Riess et al., 2015; Vánky, 1998). At ca. 0.7 ka cal. BP, the water table likely increased substantially, which supported a spread of *Nymphaea* and contributed to the sharp decrease in Cyperaceae. Since ca. 0.4 ka cal. BP, *Tetraedron minimum* and *Scenedesmus* blooms became more frequent, and the structure of the microalgal communities resembled the one from the base of the core (start of Unit I). This time, however, large amounts of *Carex* remains and large fluctuations in pH (from 5.2 to 7.8; Fig. 7) suggest the coexistence of peat patches, likely acidic, and water depressions where the pH may have been higher, supporting the development of microalgal communities. This corresponds to the current status of the marsh. Continuous monitoring of the site during the last decade indicates the presence of a seasonally evolving ecosystem, i.e. (i) a shallow lake colonized by large sedge mats during the rainy season and (ii) a waterlogged marsh-like regime at the end of the dry season due to high evaporation rates (Delalande et al., 2008a, 2008b).

The period that corresponds to unit III at Kyambangunguru starts with relatively warm mean annual air temperatures, at ca. 22 °C, followed by an abrupt cooling to 20 °C at ca. 0.5 ka cal. BP (Fig. 8). A similar abrupt event was also identified at Lake Tanganyika (Tierney et al., 2010a; Fig. 8), in the Ethiopian Highlands and in southern Africa (Nicholson et al., 2013 and references therein) and could coincide with the transition between a “Medieval Climate Anomaly” (MCA; based on Jones et al. (2001) time boundaries: ca. 1.0 – 0.8 ka cal. BP) and a Little Ice Age (LIA; 0.7 – 0.1 ka cal. BP; Matthews and Briffa, 2005) in the African continent. The occurrence of climatic shifts in Africa that could be related to the European MCA and LIA events is currently under debate but a growing body of research seems to support such a cross-latitude connection (e.g. Lüning et al., 2018; Russell and Johnson, 2007; Tierney et al., 2013). At Kyambangunguru, the last 500 yrs exhibit the coldest temperatures of the record which would support the existence of a “Little Ice Age” equivalent in East Africa, in agreement with records from nearby Lake Malawi (Branchu et al., 2010; Powers et al., 2011; Fig. 8). At the transition between units II and III (ca. 1.4 – 0.8 ka cal. BP) the $\delta^2\text{H}_{\text{wax}}$ values decrease, consistent with the reestablishment of higher P/E ratio under wetter conditions and the spreading of Proteaceae and the recovery of the *Uapaca* woodlands, as observed in the pollen record. At 0.8 ka cal. BP, the canopy density started to decrease, the fire activity increased, which led to a spread in the grassland communities, maintained until nowadays. Similar conditions were recorded at Lake Masoko (Vincens et al., 2003). Intensification of human activity in the region could be responsible for such vegetation changes, especially as no shift is observed in the $\delta^2\text{H}_{\text{wax}}$ values during this period. Additional high-resolution reconstructions in the RVP and the neighbouring provinces are necessary to better assess the relative contribution of human versus climatic nature of these recent environmental shifts.

5.2. Implications for climate dynamics in East Africa during the late Holocene and its impact on highland wetland ecology

The temperature and P/E ratio records at Kyambangunguru show a high variability over the last 4.0 ka cal. BP. The main two warm periods, before ca. 3.7 ka cal. BP and between ca. 2.2 and 1.0 ka cal. BP, were generally accompanied by drier conditions (Figs. 8 and 9). A major dry event at 4.2 ka cal. BP is

a common feature in many tropical and temperate records and was interpreted as a southward migration of the ITCZ (e.g. Gasse, 2000; Mayewski et al., 2004). A severe drought has also been observed in many other East African sites around 2.0 ka cal. BP (Marchant et al., 2018) and could have been related to the dry and warm period centred at ca. 1.5 ka cal. BP at Kyambangunguru. The discrepancy in timing between the RVP and the other tropical East African records may be related to the unique location of the RVP at the southern end of the tropical climatic belt ($\approx 10^\circ\text{S}$), i.e. at the transition between two hydro-climatic regimes (the north-eastern and the southern ones). Two cold and wet periods were identified, between ca. 3.3 and 2.0 ka cal. BP and since ca. 0.6 ka cal. BP. The latter period is concurrent with the European Little Ice Age and suggests that this cold event may have occurred globally (e.g. Brown and Johnson, 2005). The warm and dry period at ca. 1.5 ka cal. BP and the two cold and wet events all correspond to cold periods identified by Wanner et al. (2011) supporting the idea that the Holocene climatic variability is at least partly driven by global scale events (e.g. solar and volcanic activities, changes in the thermohaline circulation). However, it emphasizes that these processes seemed to have contrasting effects at different latitudes (cooling vs. warming).

A major ecological shift that led to the transition from a shallow lake to a peatland at Kyambangunguru started at ca. 2.5 ka cal. BP, in a rather wet environment, 300 years before the $\delta^2\text{H}_{\text{wax}}$ exhibit an abrupt shift towards drier conditions (Fig 9). This time offset suggests that the ecological change recorded at Kyambangunguru was primarily due to a hydrosere succession, i.e. the natural progressive colonisation and infilling of a freshwater lake by different types of macrophytes leading to its transition to a swamp which could eventually turn into a forest (Charman, 2002). A drier peatland was established at ca. 2.2 ka cal. BP and sustained until ca. 1.4 ka cal. BP likely as a result of combined warm and dry conditions, which significantly lowered the P/E ratio of the wetland (Fig. 8 and 9). This is consistent with the abrupt change at ca. 2.2 ka cal. BP observed in the biotic communities: planktonic and *Nymphaea* communities disappeared, while *Cyperus* spp. took over in only a century (Fig. 6). This behaviour suggests a threshold effect, which may reflect an autogenic hydrosere development triggered by gradual warming conditions. Previous studies have shown the ability of *Nymphaea* spp. to form mats floating at the surface of the water (Charman, 2002; Ellery et al., 1990) and pointed it out as a common step in the hydrosere succession leading to the conversion of a freshwater body to a swamp (Kratz and DeWitt,

1986; Swan and Gill, 1970). The development of floating mats could be accelerated in the tropics, as the warm air temperatures hold up high vegetation productivity rates (Talling et al., 1998). The slight warming recorded at ca. 2.6 ka cal. BP (by the br GDGTs; Fig. 8) could then have acted as a positive feedback in promoting high rates of vegetation productivity. The consequences of this fast development of floating mats are (i) large inputs of OM in the sediment, which progressively infilled the lake, (ii) a vertical expansion of the mats by peat accumulation, and (iii) a horizontal expansion and partial covering of the surface of the lake. From ca. 3.3 to 2.3 ka cal. BP (Fig. 6), opposite trends are observed in the distribution of plankton and *Nymphaea* suggesting that the more *Nymphaea* mats would cover the lake surface, the less light would penetrate the water column leading to a decrease in microalgae abundance, supporting the proposed mechanism of lake infilling.

This terrestrialization process was interrupted at ca. 1.4 ka cal. BP, when pronounced fluctuations in the water table are revealed by the presence of *Enthorriza* in the microfossil record (ca. 1.4 – 0.7 ka cal. BP; Fig. 6). The water balance, recorded by the $\delta^2\text{H}_{\text{wax}}$, increased over this period, suggesting a relative increase in precipitation (Fig. 9) in agreement with the spread of Proteaceae and *Uapaca* in the terrestrial pollen. This likely re-flooded the marsh and created patches of open water, where phytoplankton, and then *Nymphaea*, could re-colonise the marsh (Fig. 6). A similar ecosystem change was encountered at the same period in a mountainous marsh in Madagascar (Bourdon et al., 2000). This period of water fluctuations, synchronous with the Medieval Climate Anomaly (MCA; 1.0 – 0.8 ka cal. BP), is the only period of the investigating 4000 yr record when conditions were warm and wet at Kyambangunguru, suggesting that its origin may be different from the other recorded climatic changes. The MCA is described as warm and dry in most north-eastern Africa (Lüning et al., 2017; Nash et al., 2016; Nicholson et al., 2013) and humid in southern Africa (e.g. Nash et al., 2016; Tyson and Lindesay, 1992; Woodborne et al., 2015). A recent literature review by Lüning et al. (2018) suggests a transition zone across Tanzania with increased signs of humidity along a NE-SW transect. Notably, Buckles et al. (2016) and Finch et al. (2017) recorded wet conditions at Lake Challa until ca. 800 BP and at the Kwasebuge peat bog until ca. 675 BP, respectively. Hence the Kyambangunguru record presented here extends this transitional zone to the southeast, in agreement with the hydroclimatic interpretation of a biogenic silica record by Johnson et al. (2004) at Lake Malawi.

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6. Conclusions

The detailed multi-proxy analysis of a 4-m peat core covering the late Holocene (4000 years) reveals rapid and profound ecological changes of the Kyambangunguru wetland in the Rungwe Volcanic Province (RVP), southwestern Tanzania. Around 2.2 ka cal. BP, a shallow crater lake turned into a peatland. Starting at ca. 0.9 ka cal. BP the water level in the marsh increased, creating a shallow lake during the rainy season and a peaty marsh during the dry season. These significant ecological fluctuations correlated with major changes of the Late Holocene East African climate. Notably, the air temperatures remained high and the reconstructed precipitation low between ca. 2.2 and 0.9 ka cal. BP, which allowed the peatland terrestrialization to sustain. This study represents the first detailed late Holocene quantitative air temperature reconstruction from the RVP region. We identified a succession of cold/warm/cold events, largely in phase with the other regional East African climate records and with the cold periods identified worldwide by Wanner et al. (2011). This further supports that global scale processes may be the main drivers of the Holocene climatic variability. Moreover, warm conditions during the MCA followed by abrupt cooling during the LIA were observed at Kyambangunguru and elsewhere in East Africa suggesting that these two recent events occurred globally. The precipitation pattern at Kyambangunguru during these two events is opposite to most of the more north-eastern African records and rather resembles the southern African climatic records. Recent additional precipitation records in Tanzania also show such a pattern. Tanzania seemed thus to be located at the transition zone between two hydro-climatic poles (north-eastern and southern Africa) and to have experienced a variable relative contribution of these two poles over the last millennium. This study further demonstrates that peatlands and marshes provide valuable, high resolution climatic archives in the tropics that offer novel avenues of research for understanding linkages between the Holocene climate variability and ecosystem change in the tropics.

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Figure and Table captions:

Figure 1: Regional setting and climatic conditions of the studied site. A: geographical location of the Rungwe Volcanic Province (RVP, adapted from Delalande, 2008), the green lines outline the maximal positions of the intertropical convergence zone (ITCZ) over the year, the blue lines delimitate the equatorial rain region and the histograms show the monthly mean precipitation values (mm) at the main East African weather stations; B: topographical map of the RVP and location of the Kyambangunguru marsh; C: lake level (black dots), precipitation (blue bars) and marsh water $\delta^2\text{H}$ composition (red dots) variations over one calendar year; D-E: pictures of the Kyambangunguru crater marsh taken in July 2014.

Figure 2: Lithology (A) and Bayesian age-depth model (B). The A values indicate the agreement between the modelled and the calibrated age and the A_{model} the agreement of the model itself. TOM samples are in black, wood samples in red and tephra ones in grey. Horizontal dotted lines represent the model boundaries (see text for details) and excluded dates are identified as outliers.

794 Figure 3: Quantitative palynofacies: relative abundance of the main organic aggregates analysed by
795 photonic microscopy in transmitted light. In green: ligno-cellulosic tissues (LC): (i) fresh LC detected
796 as translucent LC (tLC) in light green, (ii) slightly degraded/amorphised LC (saLC) in yellow-green and
797 (iii) totally degraded/amorphous LC, characterised by red aggregates of amorphous OM (rAOM) in dark
798 green. In orange: mycelium (myc) fragments. In blue: planktonic remains (algOM).

799 Figure 4: Plant macro-fossil diagram. Results are given in absolute numbers. For the legend of the
800 lithology column see Fig. 2. Roman numbers indicate the three ecological units as defined in the
801 discussion part.

802 Figure 5: Woodland pollen diagram for the Kyambangunguru marsh showing relative percentages of
803 the selected taxa. The grey pattern shows a 10x magnification. For the legend of the lithology column
804 see Fig. 2. Roman numbers indicate the three ecological units as defined in the discussion part.

805 Figure 6: Ferns, aquatic pollen and NPP diagram for the Kyambangunguru marsh showing relative
806 percentages of the selected taxa. The grey pattern shows a 10x magnification. For the legend of the
807 lithology column see Fig. 2. Roman numbers indicate the three ecological units as defined in the
808 discussion part.

809 Figure 7: Lithology (A; see details in Fig. 2), total organic carbon over total nitrogen atomic ratio (B),
810 br GDGT-derived pH (C; based on Tierney et al. (2010b) calibration) and *n*-alkane distribution indices
811 (D-E; ACL and Paq, respectively). The three units discussed in the text are also represented along with
812 their ecological interpretation.

813 Figure 8: (a) Lake Tanganyika TEX₈₆-derived lake surface temperature (LST) of the last 4000 (black
814 contour red dots) and 1500 (red plain dots) years (Tierney et al., 2010a and 2008 respectively) and (b)
815 Lake Malawi TEX₈₆-derived LST of the last 4000 (black contour yellow dots) and 700 (yellow plain
816 dots) years (Powers et al., 2005, 2011) compared to (c) Kyambangunguru br GDGT-derived MAAT
817 (present study). The three units described in the text are also represented (dashed lines).

Figure 9: (a) Lake Edward Mg (mol %; Russell and Johnson, 2005) and (b) Kyambangunguru $\delta^2\text{H}_{\text{wax}}$ records for the last 4000 years (present study). The three units described in the text are also represented (dashed lines).

Table 1: AMS Radiocarbon chronology of the core KYAM12 (2012). Calibrated median age and range (2σ) were obtained using OxCal 4.3 software (Bronk Ramsey and Lee, 2013) with the ShCal 13 atmospheric curve (Hogg et al., 2013) and Bomb13SH3 (Hua et al., 2013).

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Highlights

- 4000-year continuous peat record of the Late Holocene in East Africa (SW Tanzania).
- Multi proxy approach evidences major ecological changes at ca. 2500 yr cal. BP.
- Detailed temperature and hydrology records highlight several cold events.
- More recent climate conditions seem connected to South African climate variability.

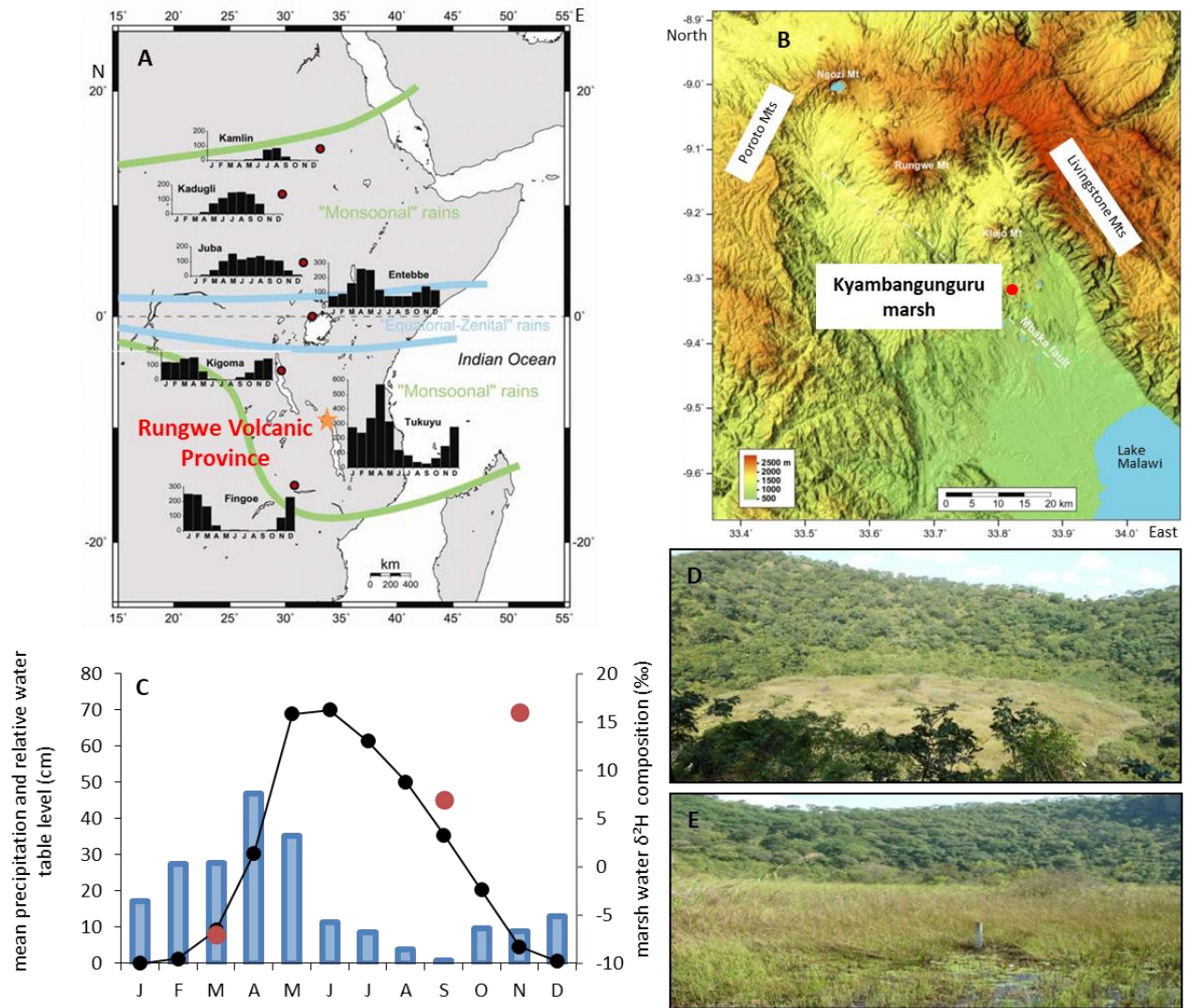


Figure 1.

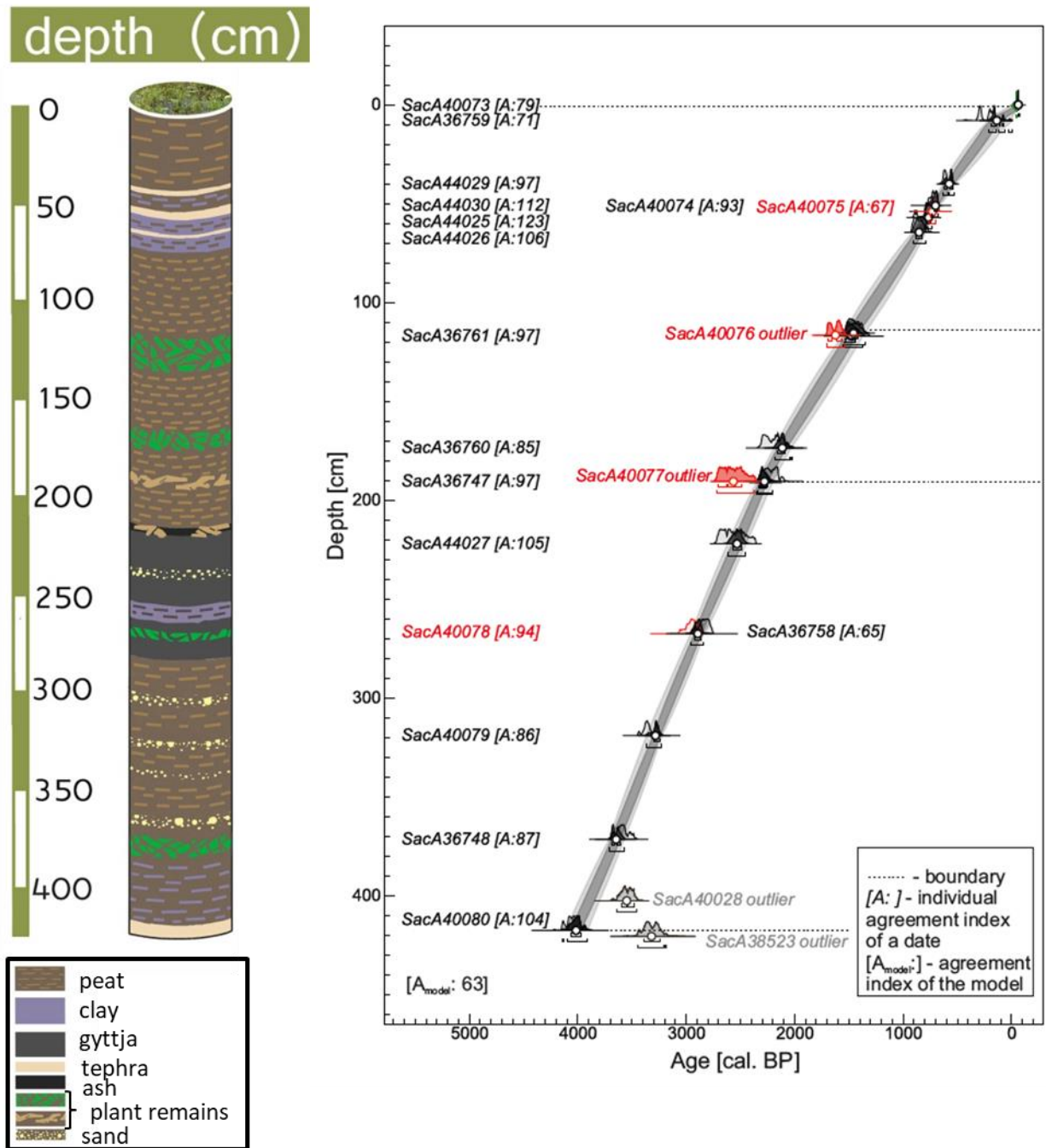


Figure 2.

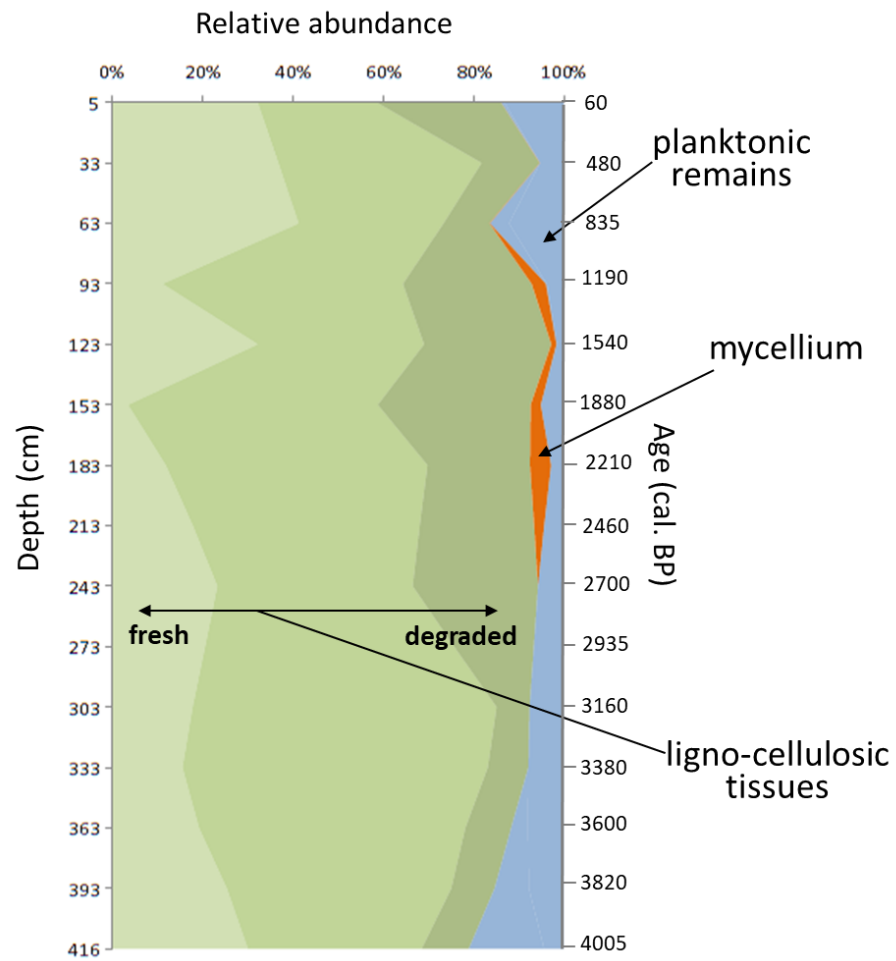


Figure 3.

Figure 4.

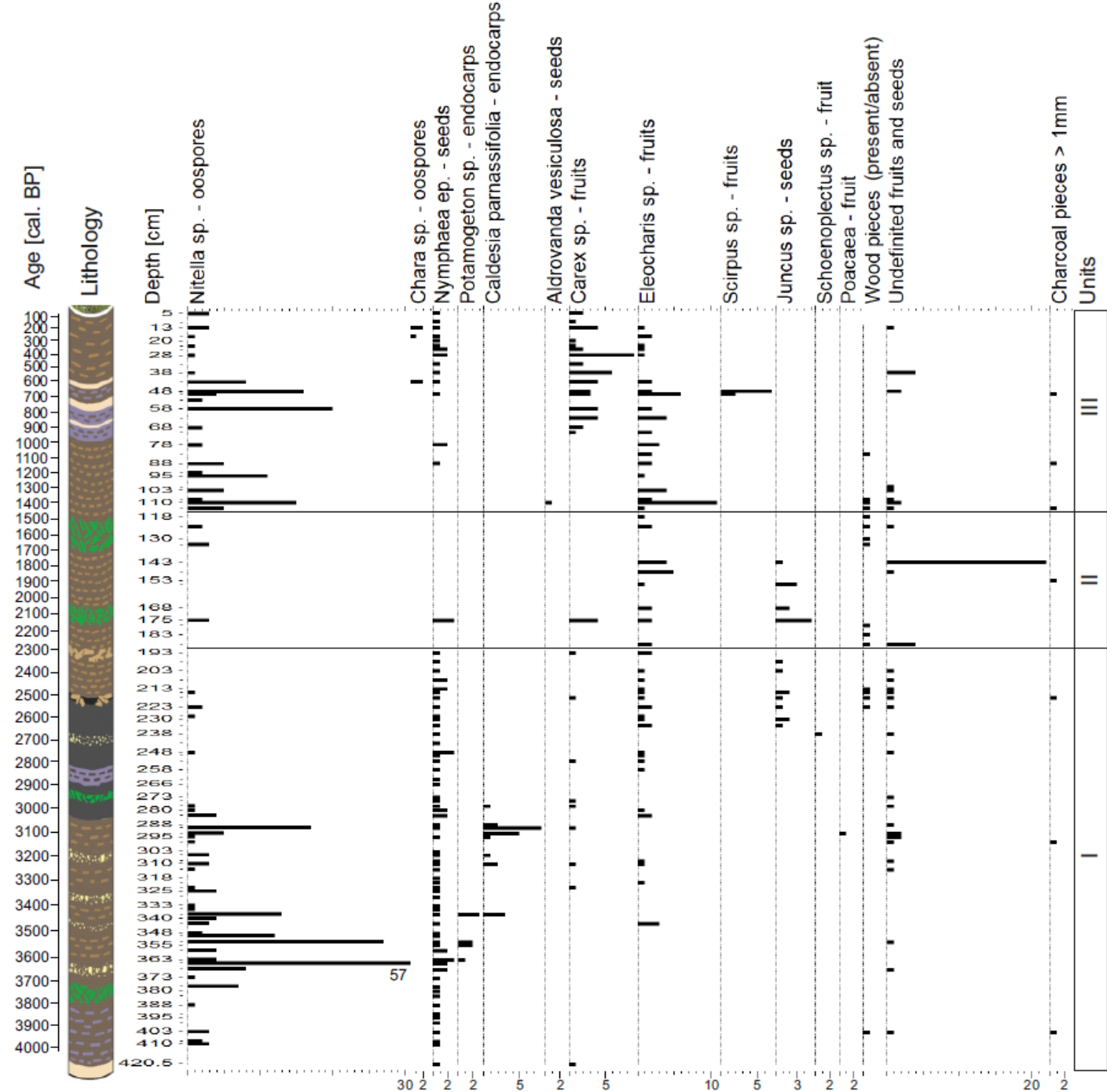


Figure 5.

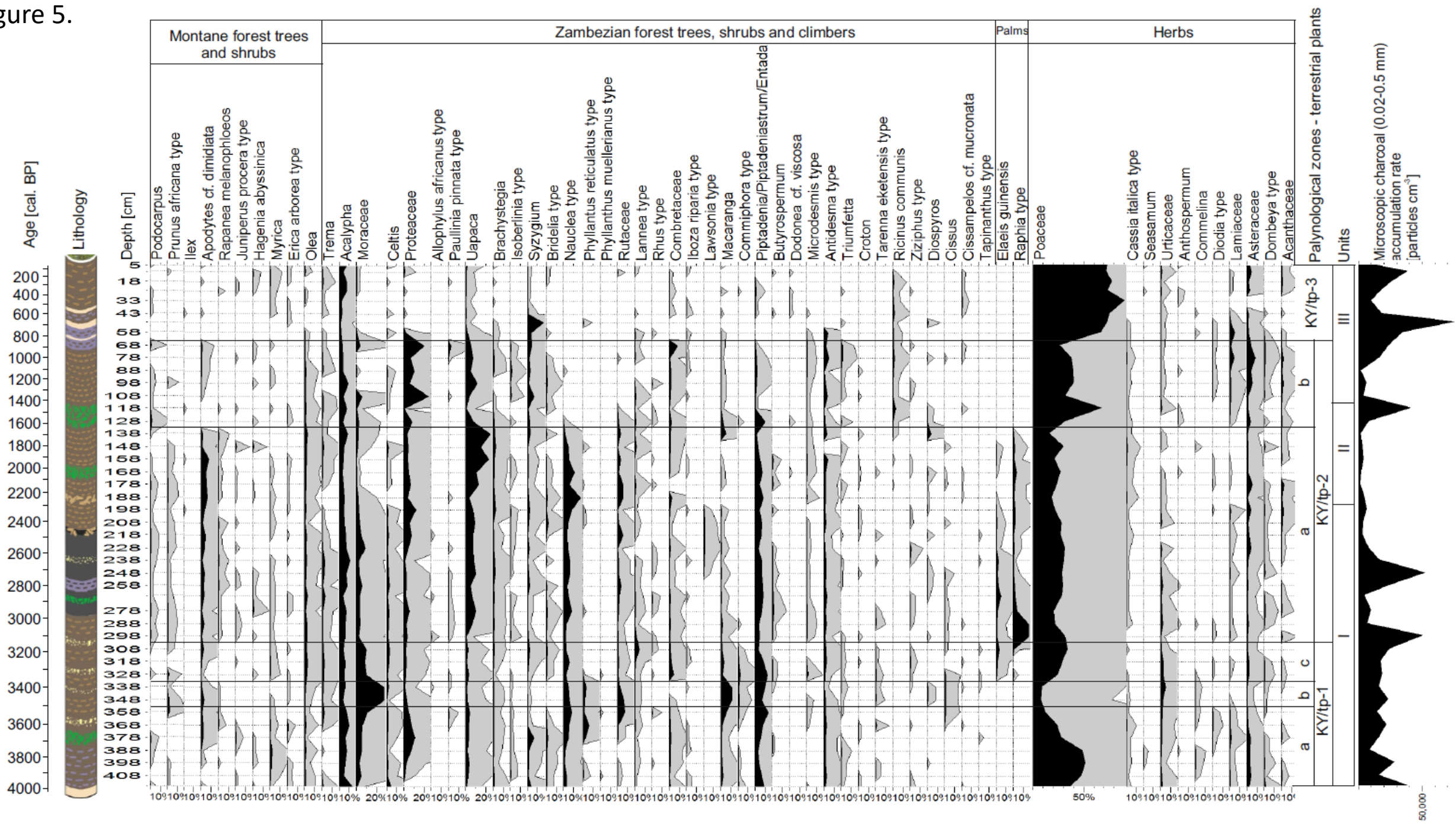
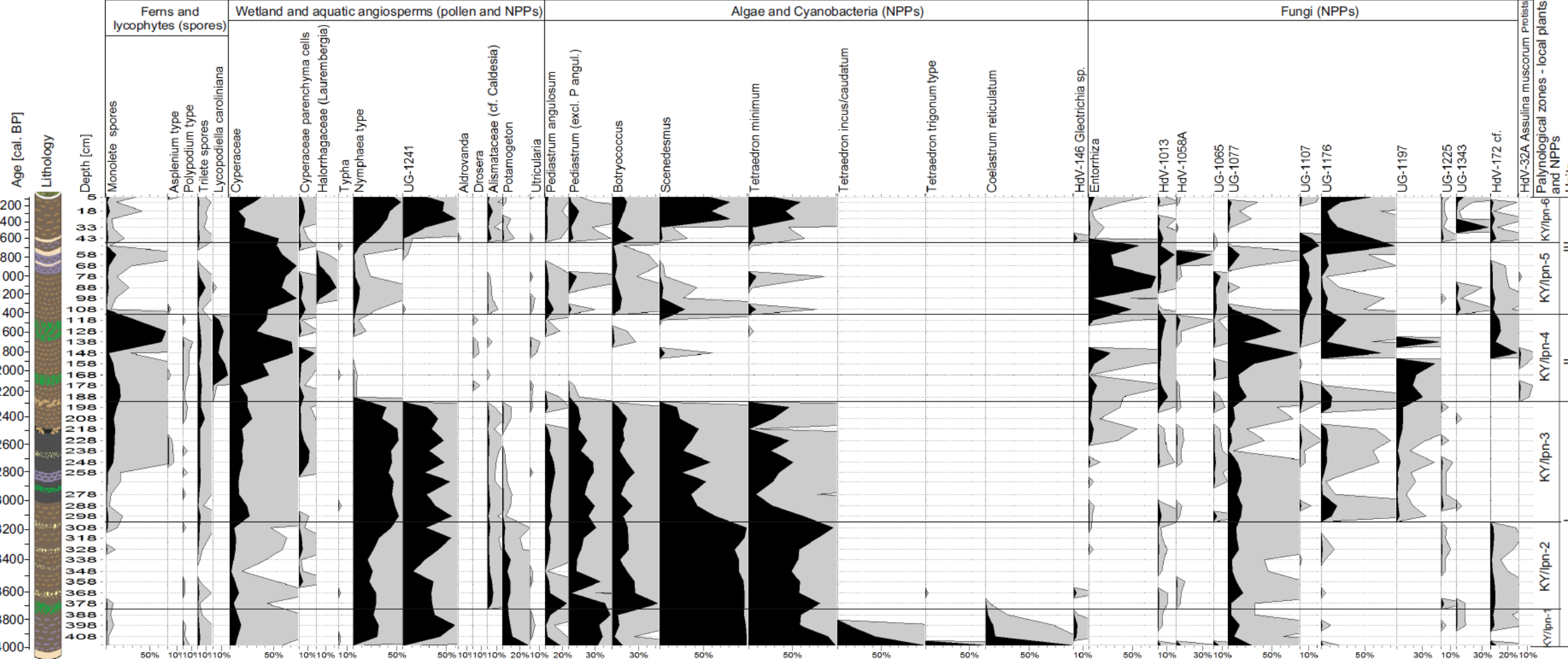


Figure 6.



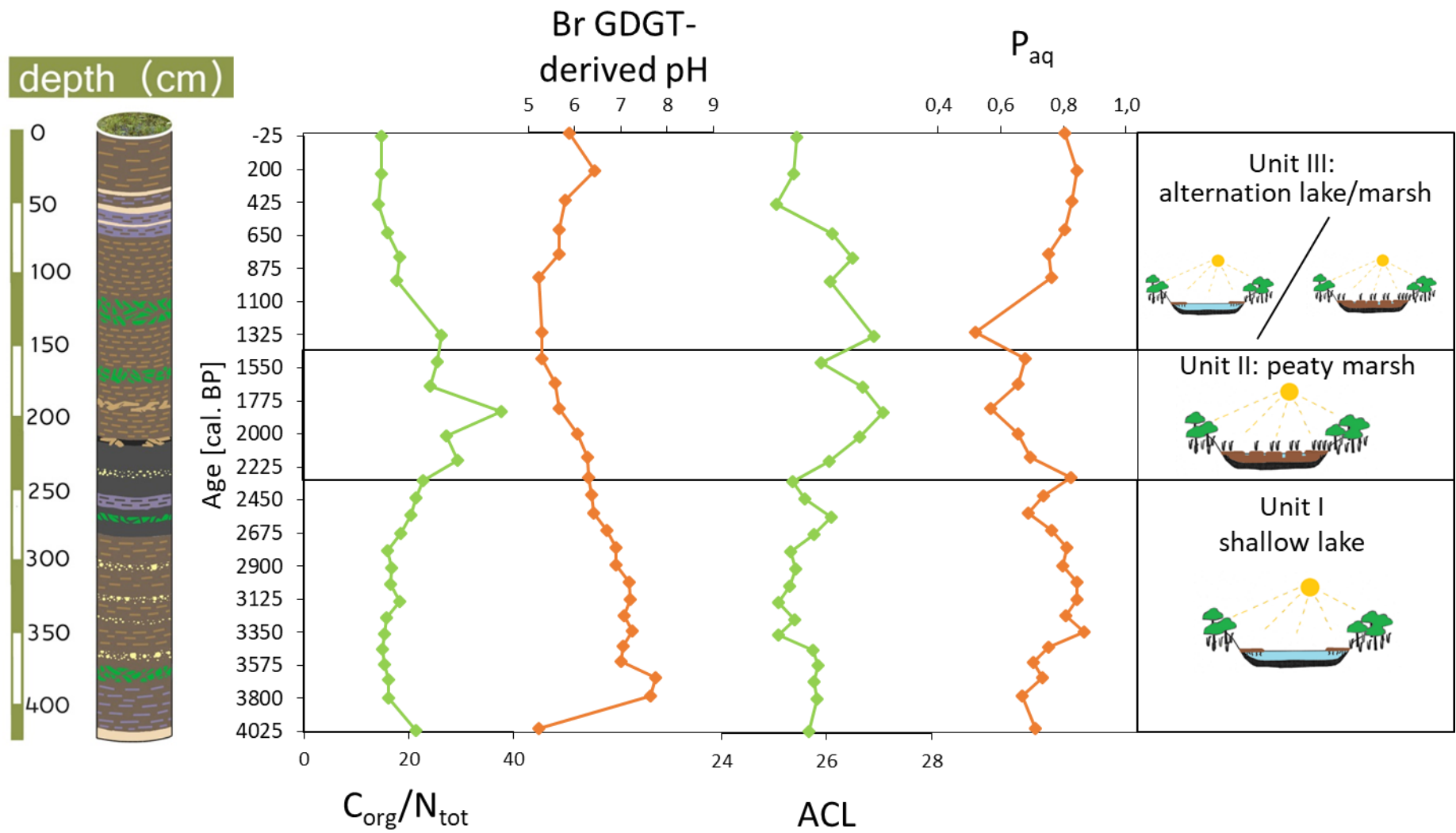


Figure 7.

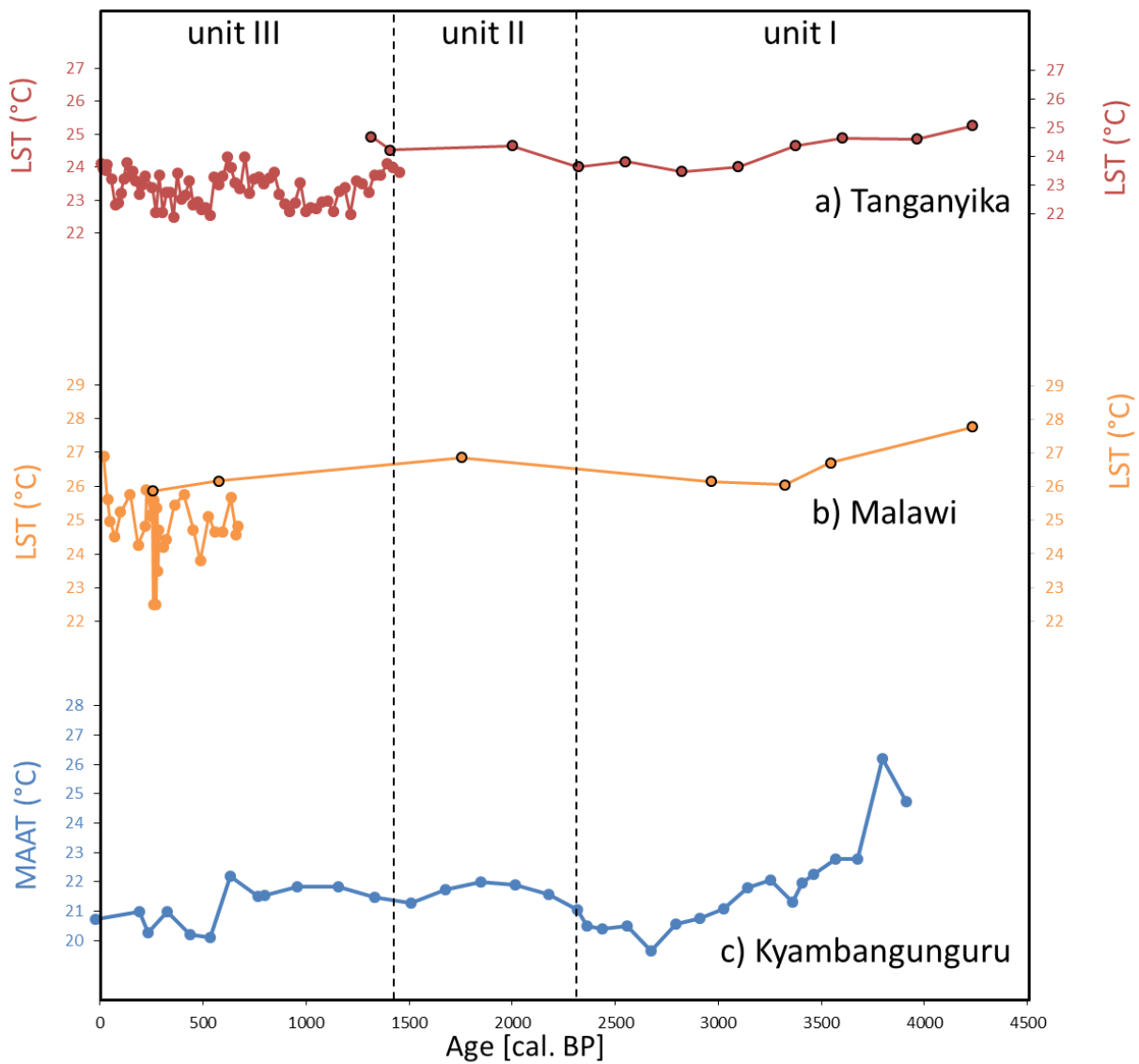


Figure 8.

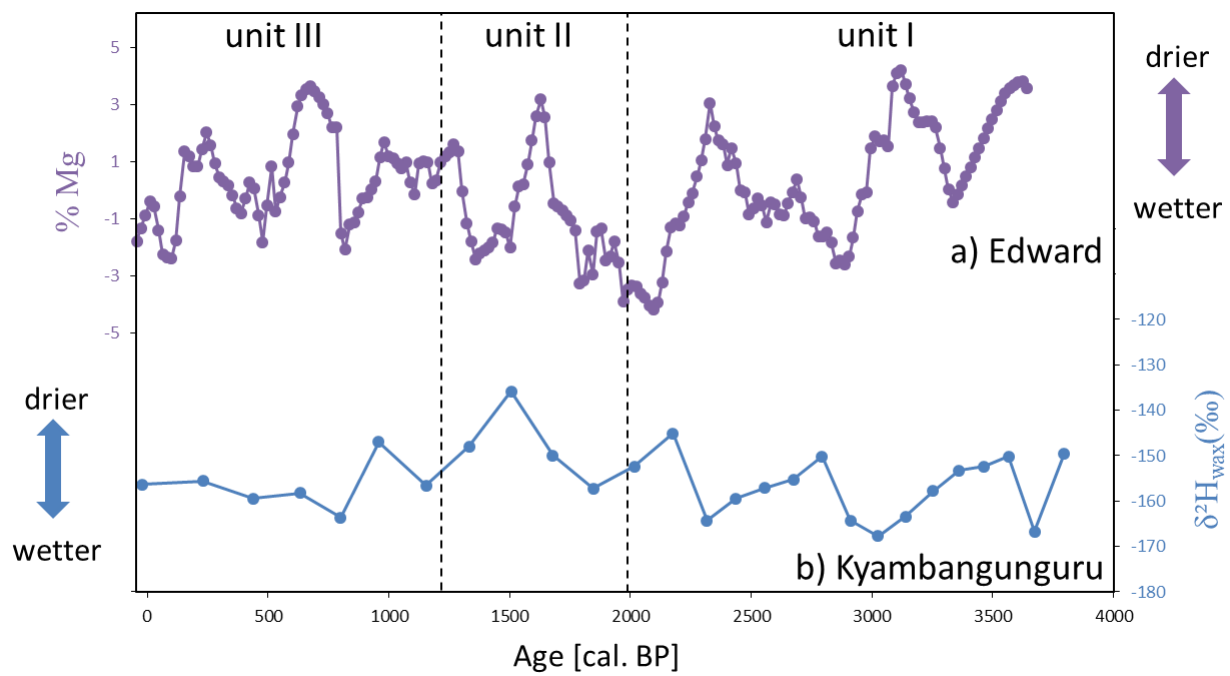


Figure 9.

Table 1.

Depth (cm)	AMS ^{14}C measure ref	Type of material	Carbon mass (mg)	^{14}C activity (pMC)	Measured ^{14}C age (cal. BP)	Calibrated age range [cal. BP; 95.4% (2 σ) range]	Modelled age $\pm \sigma$ error (cal. BP)	$\delta^{13}\text{C}$ (‰)
0.5	SacA40073	TOM	1.82	112.21 ± 0.33	Post 1950	-8–8 (3.3 %) -43–46 (88.4 %) -47–47 (3.7 %)	-45 $\pm$ 5	-29.5
8.0	SacA36759	TOM	0.75	96.86 ± 0.43	255 ± 35	435–408 (1.9 %) 325–254 (39.4 %) 225–143 (54.2 %)	135 $\pm$ 50	-24.5
40.0	SacA44029	TOM	0.75	92.48 ± 0.24	630 ± 30	646–588 (58.7 %) 574–535 (36.7 %)	574 $\pm$ 28	-23.8
51.0	SacA44030	TOM	0.75	89.99 ± 0.27	845 ± 30	763–673	701 $\pm$ 15	-22.9
54.0	SacA40074	TOM	0.55	89.53 ± 0.29	890 ± 30	800–682	730 $\pm$ 14	-28.0
54.0	SacA40075	Wood	0.91	90.27 ± 0.36	825 ± 30	742–666	729 $\pm$ 14	-25.2
57.0	SacA44025	TOM	0.75	89.20 ± 0.23	920 ± 30	905–861 (14.1 %) 842–829 (1.3 %) 820–724 (80 %)	765 $\pm$ 17	-21.4
64.0	SacA44026	TOM	0.75	88.55 ± 0.24	975 ± 30	921–774	852 $\pm$ 30	-20.2
116.5	SacA36761	TOM	0.50	82.03 ± 0.48	1590 ± 45	1538–1348 (94.6 %) 1331–1324 (0.8 %)	1472 $\pm$ 47	-25.5
116.5	SacA40076	Wood	1.37	80.47 ± 0.28	1745 ± 30	1702–1639 (39.3 %) 1633–1542 (56.1 %)	Not applied	-28.2
173.5	SacA36760	TOM	0.75	75.90 ± 0.38	2215 ± 40	2318–2059	2113 $\pm$ 37	-20.1
190.5	SacA36747	TOM	0.75	75.68 ± 0.31	2240 ± 35	2328–2146 (90.1 %) 2131–2103 (5.3 %)	2278 $\pm$ 34	-25.0
190.5	SacA40077	Wood	1.30	73.12 ± 0.26	2515 ± 30	2719–2379	Not applied	-27.5
222.0	SacA44027	TOM	0.75	73.18 ± 0.22	2510 ± 30	2715–2379	2531 $\pm$ 42	-26.8
267.5	SacA36758	TOM	0.75	70.72 ± 0.37	2785 ± 40	2945–2759	2892 $\pm$ 30	-27.9

267.5	SacA40078	Wood	1.40	69.87 ± 0.25	2880 ± 30	3062–2857	2893 $\pm$ 30	-27.9
319.0	SacA40079	TOM	1.11	67.41 ± 0.25	3170 ± 30	3445–3423 (4.8%) 3411–3236 (90.6 %)	3283 $\pm$ 34	-28.6
371.5	SacA36748	TOM	0.75	65.50 ± 0.29	3395 ± 35	3692–3657 (10.4 %) 3651–3478 (85 %)	3646 $\pm$ 37	-28.2
402.0	SacA44028	TOM	0.75	65.76 ± 0.21	3365 ± 30	3639–3456	Not applied	-26.4
417.5	SacA40080	TOM Tephra	1.04	62.75 ± 0.24	3745 ± 30	4151–3960 (91.1 %) 3950–3926 (4.3 %)	4015 $\pm$ 47	-20.0
420.5	SacA38523	TOM Tephra	0.30	67.47 ± 0.42	3160 ± 50	3450–3206 (93.8 %) 3197–3182 (1.6 %)	Not applied	-23.6