Theses of Doctoral (PhD) Dissertation

Environmental changes as recorded in the sediment of peat bogs and glacial lakes

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1. Introduction and objectives

Climate change is gaining increasing public attention and its effects can be seen in everyday life. Climate models help us to predict the extent and direction of future changes [1]. This knowledge makes it possible to prepare for changes and help us further reduce anthropogenic impact. More information may be available to predict climate change and improve models with understanding past processes [2].

Local and regional environmental processes were preserved in the lake and bog sediments, so they can provide valuable information about the environmental and climatic changes since their formation. Paleoenvironmental studies use biotic (pollen, micro- and macrocharcoal, macrofossils, diatoms) and abiotic (magnetic susceptibility, grain size, geochemical) sediment compositions to detect environmental changes in the past. Fundamental points of these studies are the 1) coring location, 2) the coring technique, 3) the chronology of the sediment and 4) finding the ideal proxy for climatic reconstruction.

For a better understanding of current environmental changes two significant periods are worth taking into account. The time interval between the Late Glacial and early Holocene period about 20 thousand and 9 thousand years ago [3]. This period was characterized by similar changes (warming and cooling) as climate models forecast. The second period is in the last 9 thousand years. In this period anthropogenic effects already are intensively present and well observable. Studies from several parts of Europe are known in this topic (mainly in the Alps, 4-7), but radiocarbon supported paleoenvironmental research done from lake sediments is scarce from the Southern Carpathians [8, 9]. The PROLONG (Providing long environmental records of Late Quaternary climate oscillations in the Retezat Mountains) project, starting in 2007, intended to fill this gap with radiocarbon base paleoenvironmental reconstructions from Retezat Mts. [10-17].

In my thesis I study how the changes of chemical element concentration in the sediment can indicate environmental changes, climate variations and human effects. Our aim was to develop analytical methods, which may complement the methodology of routinely applied paleoenvironmental methods and can be used to identify environmental changes in the past and help us reconstruct local and regional processes.

For these reasons, we aimed to:

1. Develop a large diameter sampling tool for peat coring to provide undisturbed and uncompressed samples with adequate quality and quantity for wide-ranging paleoecological studies.
2. Develop a fossil selection strategy and sample preparation methods for radiocarbon age determination of peat and lake sediments. Comparison of different age-depth models and sedimentation rate estimation methods.
3. Develop elemental analysis and sample preparation methods for high resolution sediment analysis. Elemental analysis and determination of organic matter content of four lake (Brazi, Gales, Lia and Bucura) sediments from the Southern Carpathians and peat samples from Mohos peat bog (Eastern Carpathians). Multivariate statistical evaluation of geochemical results and reconstruction of detectable environmental and climatic changes.
4. Identify local and regional events and global changes in the sediment. Testing climate history reconstructions based on age-depth models and high-resolution geochemical composition. Examining the potential of elemental component information to reconstruct environmental change. We can take into account the paleoecological and paleolimnological results from other analysis of the same core. Comparison of results with climate reconstructions from other areas (e.g. Greenland, Alps).

2. Materials and methods

2.1. Study site and sampling

The lava dome complex of the Ciomadul volcano (Eastern Carpathians) is an exciting area in many aspects, both in the study of environmental changes of the Late Glacial and Holocene and in volcanology. It is mentioned as the youngest volcano in the Carpathian-Pannonian region [18, 19], where Mohos peat bog was formed in the older explosion crater. In the area of the present peat bog a lake was formed which was gradually transformed into a peat bog, therefor under several meters of continuous peat layer the former lake sediment can be found. The peat section provides an opportunity to study climatic changes in high resolution.

A previous study from Tantau et al. (2003) discusses in detail sampling and radiocarbon dating of the Mohos peat bog [20]. In this study four radiocarbon dates were set as outlier as younger samples may have taken deeper position during the sampling with a Russian-type corer.

Errors of this kind consume unnecessary resources, both in time and financially, and the sorting of outliers involves subjective factors to building age-depth models.

For peat bog sampling we previously used a Russian-type corer, which has many disadvantages. Based on our previous experience with peat bog sampling, a modified piston corer was built, that allows undisturbed and uncompressed sampling of peat, and it works great for lake sediment sampling under the peat layer as well. The first coring with the modified piston corer from the Mohos peat bog (East Carpathians, Romania) was carried out in summer 2013. There was a second sampling action in 2018, with a sampling point being close to the first one.

The second sampling site is in the Retezat Mts., Southern Carpathians, where more than hundred glacial lakes were formed after the last glaciation. These glacial lakes are paleoecologically significant because they are characterized by continuous sedimentation since their origin to the present. We extended our study to four lakes in the Retezat Mts, located at approximately the same altitude. Lake Gales (1990 m asl) and Lake Brazi (1740 m asl) is located on the northern side of a mountain in Gales valley where Lake Bucura (2040 m asl) and Lake Lia (1910 m asl) is situated on the southern side of the Retezat Mts in Bucura-valley.

The Retezat Mts. is a natural reserve area, there are no roads for traffic and aerial tramway. Lakes can be reached on relatively difficult hiking trails. Another factor that may have limited the research so far is the depth of the water, the deepest lake is 24 m. Sampling in such terrain required the development of a special mobile drill rig that could be picked up in pieces in backpacks. A specially modified Livingstone-type sampler and Kullenberg corer were developed to solve this problem. The first coring from the Gales valley (Gales and Brazi lakes) was carried out in summer 2007, while the other two lakes (Bucura and Lia lakes) were sampled in 2008. Surface sampling was also carried out in the area.

2.2. Experimental methods

Samples were placed in PVC tubes during sampling, and samples were stored at +4°C until processing. Photographic documentation and sediment lithology were described in the laboratory. Sediment cores were subsampled for radiocarbon, elemental analysis and organic matter content.

Before the radiocarbon measurements samples were subjected to mechanical and chemical preparation. The surface of the samples were examined under a microscope and photographed. The required fractions were separated with wet sieving. Lake sediment samples were prepared with traditional acid-base-acid (ABA) method. Samples were then combusted to prepare carbon dioxide, the resulting gas was cryogenically purified and graphitized in a closed tube system in the presence of zinc and titanium hydride reagents. The radiocarbon content of the samples was determined by an accelerator mass spectrometer (EnvironMICADAS AMS) at the ICER laboratory in Debrecen. A number of international reference samples (IAEA C9 fossil wood) with known C-14 activity were prepared and measured in the same way as the real samples to check the conditions of preparations and measurements.

For chemical analysis first the samples were dried at 105°C. The determination of organic matter and water content was carried out with the traditional loss-on-ignition (LOI_{550}) method [21], samples were combusted in a porcelain crucible for 4 hours at 550°C.

The sample preparation of plant, soil, rock and sediment samples prior to the elemental analysis was carried out by digestion. The samples dried at 105°C were dissolved in a solution by wet digestion at atmospheric pressure. In another procedure the organic matter was removed at 550°C and the ash was digested with acids (36% (m/m) HCl, 38% (m/m) HF, 4% (m/m) H_3BO_3) in a closed plastic vessel. The elemental analysis of samples was carried out by inductively coupled plasma optical emission spectrometry (ICP-OES, IRIS Intrepid II XDuo, Thermo Fisher Scientific) and by microwave plasma atomic emission spectrometry (MP-AES 4100, Agilent Technologies)

2.3. Data analysis

All the radiocarbon dates refer to calibrated radiocarbon years before present (cal yr BP, 0 yr BP = AD 1950). For the calibration of the measured conventional radiocarbon ages, CALIB 7.1 was used. The age-depth models were obtained using CLAM v 2.1 [22] and BACON [23] software.

The concentrations of elements are shown as oxides (Al_2O_3, TiO_2, CaO, MgO, K_2O, Na_2O, Fe_2O_3, MnO and SiO_2). Numerical zonation of the geochemical data is based on stratigraphically-constrained cluster analysis calculated with CONISS, using Psimpoll (version 4.27). Statistical calculations were performed using the SPSS/PC+ and PAST statistical software packages.

3. New scientific results

3.1. Development peat sampling technique

A peat sampler was developed that provides adequate quantity and quality of samples required for paleoecological and geochemical investigations, which is also suitable for the extraction of peat layers with considerable thickness (> 10 m). The sampler is robust enough to be able to sample the lake sediment under the peat with the same coring head. It was tested on the Mohos peat bog, where 9.30 m continuous peat was extracted. Drilling continued in the lake sediment, reaching 19.5 m depth.

The cutting of the fibrous parts of the peat was ensured by the cutting edge and the use of electric hammer. The hammer-induced vibration helps the sampler enter to the peat without compaction. PVC casing prevented peat fall down from the coring wall.

Radiocarbon measurements proved that there was no age reversal during the sampling, and the previously experienced problems did not occur. The developed sampler is in use in our research team, and since then it has been used successfully on several peat bog.

The upper 10-meter-long peat section of the Mohos peat bog was cored in 2018 again assisted with the same electric hammer and PVC casing technology. The two coring campaigns were compared with paired sample t-test. Based on the t-test ($t = -0{,}968$; $df = 9$, $p = 0{,}358$) it can be seen that there is no significant difference between the two coring at the same time with the same equipment. So, the earlier drilling is well reproducible.

3.2. Method development for radiocarbon analysis of the cellulose content extracted from peat

In the radiocarbon age determination of peat, it is common that a specific volume of sample is taken from the core and its total carbon content is measured. During these “bulk” analyses many problems may arise (e.g. rejuvenation effect caused by modern roots).

To eliminate these errors, I did the following during the sample preparation for radiocarbon dating: 1) I tried to isolate only the leaves of the peat by wet sieving, instead of using the “bulk” sample; 2) I extracted the specific cellulose fraction of peat for the radiocarbon measurements.

Methodological developments were carried out to prepare cellulose from the peat fractionated by sieving. The BABAB (base-acid-base-acid-base) method is well applicable for cellulose preparation from wood samples [24]. I adapted this method to extract peat cellulose.

Using cellulose for measurements along the entire length of the sediment has the advantage that all ages were determined from the same special material. There were no outliers in the data, thanks to proper sampling and radiocarbon sample preparation.

3.3. High resolution chronology based on peat cellulose

As chronology constitutes the backbone of all paleoenvironmental study, our aim was to build a high-resolution radiocarbon chronology for the peat-bog sediment. In total, 36 ^{14}C samples were measured for the age-depth model.

The 930-cm long continuous peat core dates back to c. 11 770 cal yr BP. Fine peat accumulation rate change was observed along the profile. 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.

3.4. The first radiocarbon based multi-proxy studies from the Retezate Mts.

Radiocarbon dating of high-mountain lake sediment could sometimes be challenging, mainly due to very low organic matter content (<4%) characteristic for the late glacial and Holocene. For radiocarbon dating, the most reliable materials are terrestrial macrofossils. Due to lack of this material in glacial lake sediments from the Retezat Mts. other fossil and sediment fractions were used for dating.

The aquatic animal and terrestrial plant macrofossil components in the Holocene section of Lake Gales were carefully compared. On the whole, wet sieved sediment fractions and terrestrial macrofossils both gave consistent and useful dating results for the age-depth models. This suggests that in the cases where individual terrestrial plant macrofossils are scarce in the sediment which anyway is rich in organic matter, this bulk organic fraction provides reliable dates for the Retezat glacial lake sediments.

As a first regional absolute chronological study on lake sediment deposition rates and times in the Retezat Mts., this study discusses four sediment profiles dating back to the late glacial (LG) period. The sediment of Lake Brazi dated back to 15,750 cal yr BP. This is the best dated core from the four lake sediments, with 21 radiocarbon ages. The Gales core spans the interval between 1770 and 15,250 cal yr BP. According to the age-depth model the Bucura core ranges from 695 to 10,350 cal yr BP. The longest sediment sequence was obtained from Lake Lia, the continuous core dates back to c. 17,130 cal yr BP.

3.5. New paleolimnological reconstruction based on sediment element composition

3.5.1. Reconstructing local and regional environmental changes from Lake Bucura and Gales

In the case of Lake Gales, the period between 1744 and 10,370 cal yr BP is represented by 94 subsamples down to 196 cm. The Lake Bucura sediment record discussed here is 494 cm long and covers the time interval between 740 and 10,320 cal yr BP period, which is represented by 250 subsamples. As expected, given the chemical homogeneity of the geological background, the down-core chemical profiles of both lakes show relatively small variability within the Holocene. Changes in the geochemical composition are relatively weak in most major oxide profiles, therefore the principal components are not much variable either. However, when the enrichment factors are taken into consideration, both chemical records show significant compositional changes. Given the homogenous lithology the variability seen in the chemical data likely indicates changes brought about by external factors, such as vegetation change and soil formation processes throughout the Holocene in the catchment areas and within the water columns.

In this study we use multi-proxy approach to document the contrasting geochemical evolution of two alpine lakes in the Retezat Mts. during the Holocene. We compare the trends in the pollen, microcharcoal, siliceous algae, major oxide and loss-on-ignition records of the two lakes characterized by similar geological setting. For the time period discussed in this work, there are a few robust changes in the geochemical data, although their amplitude is lower than the significant variations seen in the Younger Dryas to Early Holocene transition in Lake Brazi. Certain synchronous changes and events were observed, among which the most conspicuous is the onset of human impact around the studied lakes, and other Holocene environmental changes that have been also identified with other proxies (pollen, $LOI_{550°C}$, microcharcoal, plant macrofossils, chironomids, diatoms). Main changes have been observed at ca. 10,670-9000, 6500 and 3000 cal yr BP. Catchment soil stabilization was apparent from ~9000 cal yr BP onwards, in-lake and terrestrial productivity was the highest between 9000 and 6500 cal yr BP, and human impact became the dominant driver in both terrestrial vegetation and sediment geochemical changes and in-lake processes over the last ~3400 years.

The geochemical, pollen and silicious algae data also suggested that the northern and southern slopes underwent slightly different vegetation and soil development trajectories, which in a large extent originate from the different human impact histories of the two slopes. Whether such differences are really northern and southern slope specific must be verified through further work.

3.5.2. Geochemical investigation and paleoclimate reconstruction of the Late Glacial and early Holocene sediments from Lake Brazi and Lia

In the case of Lake Brazi, the period between 9950 and 15,800 cal yr BP is represented by 101 subsamples. The Lake Lia sediment record is 263 cm long and covers the time interval between 9950 and 15,700 cal yr BP period, which is represented by 130 subsamples. Both sediment profiles show significant differences in the geochemical composition during the late Glacial and Holocene periods.

Paleoclimate reconstruction was prepared based on the age-depth models and high-resolution geochemical elemental composition data of the Brazi and Lia sediments. Results were compared with Greenland ice core $\delta^{18}O$ record (NGRIP). Sediment samples were ordered *a priori* into “cold” and “warm” representative groups based on their inferred ages. Groups were compared with classic linear discriminant analysis (LDA).

The calculated discriminant values are good indicators of changes in sediment caused by climate change, as their values give the cold and warm directions. Thus, negative values indicate cooling, while positive values indicate warming of the climate. The “*a posteriori”* groups can be used to determine the period during which local changes differed from the climate changes in the North Atlantic region. The chemical composition of sediments deposited during the “cold” and “warm” periods shows differences in both sediments. There is no significant difference in CaO concentration between “cold” and “warm” groups. The organic matter content ($LOI_{550°C}$) was significantly higher in the “warm” group, while the other elements showed higher concentrations in the “cold” group. Considering that the canonical correlation shows significant relationship between the discriminant values and the two formed groups (“cold”, “warm”), the discriminant function is a good measure of the chemical composition of sediments formed during cooling and warming.

Significant environmental changes such as the Younger Dryas cooling event and the significant warming period of Holocene can be recognized by the elemental composition and organic matter content of the sediments. However, shorter periods of cooling and warming indicated by isotope studies [25, 26] don’t always affect the geochemical and biotic composition, and also the resolution plays a major role in studying such changes.

We found that analysing element and biotic composition changes together with reconstructions from other regions can be used to describe regional and local changes from the Southern Carpathians. The importance of these regional climate reconstructions is that they can be used to fine-tune the regional parts of global forecasts and contribute to local forecasting of the expected impacts of global climate change.

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List of publications related to the dissertation

Foreign language scientific articles in international journals (4)

1. **Hubay, K.**, Molnár, M., Orbán, I., Braun, M., Bíró, T., Magyari, E.: Age-depth relationship and accumulation rates in four sediment sequences from the Retezat Mts, South Carpathians (Romania).
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