Research project PD 128908 – Final report

Stable carbon and oxygen isotope ratios of earthworm biospheroids, as promising paleoenvironmental and paleoclimatic proxies – case studies from loess-paleosol sequences

Dr. Gabriella Barta

1. Introduction, background, research history

My research project has focused on the stable carbon and oxygen isotope ratios of earthworm biospheroids from loess-paleosol sequences. Earthworm biospheroids (EBS, Becze-Deák et al. 1997), also known as earthworm (calcite) granules (Durand et al. 2010; Prud'homme et al. 2016) or calcium carbonate granules (Canti and Piearce 2003), are frequent constituents of loess-paleosol sequences (e.g. Becze-Deák et al. 1997, Pustovoytov and Terhorst 2004, Koeniger et al. 2014, Barta 2016, Prud'homme et al. 2016). During the last decade new possibilities evolved in the research of EBS, as application as a paleoclimate indicator based on its wide temporal and spatial distribution within a sequence (e.g. Versteegh et al. 2013, Prud'homme et al. 2016, 2019).

The aims of my original research plan were based on the findings of these new directions. The expected results and benefits were defined as follows:

- Determination of stable isotope patterns of EBS: 1) in relation to the different stratigraphic units within the same sequence; 2) comparison of patterns with coeval stratigraphic units on a regional scale. Establishment and application of a paleoenvironmental key in order to interpret the stable isotope signatures of EBS.
- Application of cluster- and discriminant function analysis on the stable isotope data of EBS, which helps to determine groups to be interpreted in relation to the different stratigraphic units and study site locations.
- Application of the paleothermometer method provides unique data, as follows: 1) paleotemperature can be determined for the periods of earthworm activity along the individual sequences; 2) correlation of the coeval stratigraphic units allows to compare paleotemperature data and draw regional conclusions.

2. Progress, problems and obstacles encountered

Due to the resource-intensive nature of my research, the first two working years were spent on fieldwork, laboratory work, sample preparation and measurements. The project started originally in 2018, but after the first year it was suspended for 2 years due to maternity leave. The progress report of the second working year already included the preparation of the data interpretation. Certain changes concerning planned collaboration made me restructure the original working plan. My collaboration has ended with the LIAG (Hannover), since the stable isotope IRMS was decommissioned in 2021 during my maternity leave. I received an opportunity from Dr. Attila Demény at the HUN-REN Hungarian Research Network, Institute for Geological and Geochemical Research to proceed with the stable isotope measurements of EBS in a determined amount. Due to the changes in measurement opportunities, the stable isotope analysis of bulk modern soil samples and further complementary samples could not be achieved.

The interpretative directions and methods presented in the working schedule (e.g. cluster analysis) did not confirm the hypotheses formulated in the research plan, therefore I had to turn to new possibilities. My goal was to find the most appropriate statistical method that would allow the evaluation of a large database and provide results that could be interpreted in a sedimentological as well as paleopedological way.

My investigations also revealed that the EBS paleothermometer method applied by PRUD'HOMME ET AL. (2016), which was referred in my research plan and was intended to be used, is not applicable to the studied loess-paleosol sequences of the Carpathian Basin – contrary to international practice. This is due to complex paleoclimatological and paleoenvironmental reasons, which will be presented in this final report (see **subsection 3.a**).

The preparation of manuscripts, exclusively on the EBS research, for peer-reviewed journals with impact factor were not yet completed due to the interpretative challenges, during which I had to start deciphering the data almost from scratch several times. The preparation of the manuscript containing the results of the Basaharc sequence is ongoing – when it is possible, I would like to apply for its inclusion in the final report retrospectively. On the personal side, frequent illnesses of my son made progress difficult during the last year of work. After my project was finished, I was only able to find a job outside the scientific era. This situation together with managing life with a small child made it difficult to proceed with the research.

As biospheroids belong to the group of microscale secondary carbonates in loess-paleosol sequences, it is necessary to gain a better understanding of the evolutionary characteristics of the environment in which they are found, in order to prepare the ground for their micromorphological and geochemical study. I have co-authored several studies during my grant period, adding my work and knowledge to a deeper understanding of the sedimentological, paleopedological, topographical and paleoenvironmental evolutionary processes of the sequences I have been involved in (Basaharc, Hévízgyörk, Malá nad Hronom, Paks, Verőce). These collaborative studies, using different methods, provided complex insights to the understanding of the loess-paleosol sequence development and the climatic and environmental changes during the Pleistocene (as for instance through the understanding of the pedogenesis-erosion-sediment accumulation balance of the paleoenvironment). The information gained from these studies contributed to a deeper insight to the hosting environment of EBS as well. These studies were crucial to put my results and interpretation views in the right context.

As the results of my research exclusively on EBS have not yet been published outside conference proceedings, the criteria for the final report allow for a longer version (max. 25 pages/75000 characters).

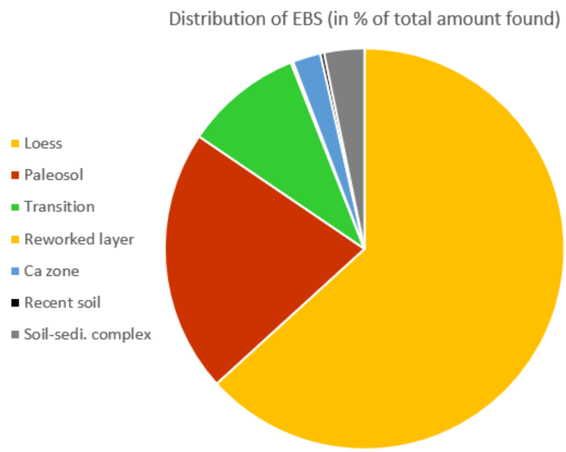
3. Applied methods

a. Field work and morphological investigation

All of the 8 loess-paleosol sequences in the research plan have been examined (the Basaharc sequence consists of 2 parts, with which 9 sequences were investigated in total). Bulk samples (150 g) were collected in 10 cm vertical resolution by using a continuous sampling technique. The wet sieving process (500 µm sieve, as following BARTA 2014) and drying out of samples were followed by the separation of EBS under a binocular microscope. A complete micromorphological description was made for the rest material of each sample and the amount of EBS was counted for each sample. In total 891 bulk samples were taken, from which 50096 pcs of EBS were separated.

The studied loess-paleosol sequences are as follows:

- Basaharc brickyard section: north-facing "classic" wall (142.0 - 164.7 m a.s.l.; 47°48'12"N; 18°50'44"E; after SZEBERÉNYI ET AL. 2018) and south-facing wall (47°48'15"N; 18°50'42"E; after MILINKÓ 2019). The north-facing wall is 7,4 m thick, it contained 6382 pcs of EBS. The south-facing wall is 9,1 m thick, it contained 4552 pcs of EBS in total.
- Süttő section (256 m a.s.l., 47°44.26'N; 18°26.87'E; after NOVOTHNY ET AL. 2009). In total 15,65 m thick sequence, it contained 3657 pcs of EBS.
- Malá nad Hronom section (130 -155 m a.s.l., 47°51'7.8336"N; 18°41'21.9618"E; SZEBERÉNYI ET AL. 2020a,b, BRADÁK ET AL. 2021). 8,3 m thick sequence, it contained 1997 pcs of EBS.
- Verőce section (130 - 155 m a.s.l., 47°49'46.0"N; 19°02'33.6"E; after BRADÁK ET AL. 2014). 9,2 m thick sequence, it contained 369 pcs of EBS.
- Hévízgyörk section (142 m a.s.l., 47°38'25"N; 19°31'36"E; CSONKA ET AL. 2020). 7,8 m thick sequence, it contained 789 pcs of EBS.
- Paks section: North-East facing wall of the former brickyard (135 m a.s.l., 46°38'24"N; 18°52'24"E; following the chronostratigraphic frame of THIEL ET AL. 2014). 10,1 m thick sequence, it contained 1688 pcs of EBS.
- Villánykövesd section (122 m a.s.l., 45°52'47"N; 18°26'16"E; after ÚJVÁRI 2004; DUDÁS 2012). 10,6 m thick sequence, it contained 1785 pcs of EBS.
- Beremend section (126 m a.s.l., 46°50.36' N; 20°05.54' E, NRDIO K119366). 10,9 m thick sequence, it contained 28877(!!!) pcs of EBS in total.



My first intentions concerning the morphological analysis of EBS were limited to the completeness of their appearances and their distribution along the sequence. Incomplete morphologies are connected to changes in the soil-sedimentary environment, since redeposition breaks the granules and strong dissolution erodes the surface of the composing crystals (Becze-Deák et al. 1997). All stable isotope measurements were carried out by solely paying attention to the completeness of the EBS, even though I have already noticed during the sample preparation and counting period, that different appearances are characteristic of EBS (see more details in **subsection 4.**).

b. Stable carbon and oxygen isotope geochemistry

In order to proceed with stable carbon and oxygen isotope analysis, individual EBS were crushed using an agate mortar and pestle (400 to 450 µg of material was prepared, as following Koeniger et al. 2014). Reaction of EBS samples with anhydrous phosphoric acid was performed in Exetainer® vials at 85°C after flushing the headspace gas with helium. CO_2 processing was carried out by a Thermo Finnigan Gasbench II and measurements were subsequently made by using a Thermo Finnigan Delta XP isotope ratio mass spectrometer (IRMS, LIAG, Hannover, Germany) and a Thermo Finnigan Delta V (Institute for Geological and Geochemical Research, Research Center for Astronomy and Earth Sciences, HUN-REN, Budapest, Hungary) in continuous flow mode. Normalization of the measured carbon and oxygen isotope compositions was against international standards (as NBS-19 and L-SVEC). Isotope values are indicated by δ values and are given in per mil (‰) using the equation:

$$\delta = (R_{SA}/R_S - 1) \times 10^3$$

where R is the isotope ratio $^{13}C/^{12}C$ or $^{18}O/^{16}O$ of the sample (R_{SA}) or of the standard (R_S). The δ values are expressed against the V-PDB (Vienna PeeDee Belemnite) scale.

The mean carbon isotopic composition of EBS is **-12.27±1.33‰** and the mean oxygen isotopic composition of EBS is **-7.28±2.17‰** for the complete data set (in total 1513 EBS were measured).

c. Application of the EBS paleothermometer

The research plan aimed to apply the EBS paleothermometer method introduced by Prud'homme et al. (2016). Prud'homme et al. (2016) used the equations introduced by Versteegh

ET AL. (2013) and further developed it as combining the linear relationship between $\delta^{18}O$ values of meteoric water and mean annual air temperatures at mid- to high-latitude locations (excluding areas under Mediterranean climatic regime) and an additional linear equation based on the daily air and ground temperatures under snow-free circumstances in certain locations of Alaska. The above-mentioned combination allowed PRUD'HOMME ET AL. (2016) to determine air temperatures for those periods of the year when earthworm activity allowed the formation of biospheroids.

After the stable isotope measurements of EBS were completed, I have attempted to apply the paleothermometer and run calculations for my complete dataset. The results were not promising and provided such values, which would not reflect the prevailing conditions for the Carpathian Basin (regarding for the periods of dust accumulation and soil development). As PRUD'HOMME ET AL. (2019) pointed out in 2019, EBS were almost exclusively present in tundra gleys and boreal brown soils and not really characteristic of the bracketing loess horizons in Western European loess-paleosol sequences. My findings so far showed a partially opposite picture for various sections of the Carpathian Basin (BRADÁK ET AL. 2014, KOENIGER ET AL. 2014, BARTA 2016, recent project PD 128908). It seemed as the EBS paleothermometer method was fairly applicable for the Western European sites, which were strongly influenced by the effects of the Atlantic Ocean. I have discovered that the equations in the published form are not applicable for the sites in the Carpathian Basin, as the (recent and paleo-) climatic control over this region differs from Western Europe – and also the mesoscale spatial diversity of precipitation supply is relatively large with varied source areas (BOTTYÁN ET AL. 2017). In the case of the southern study sites of the recent project (Villánykövesd and Beremend), the prevailing submediterranean effects (e.g. HUM 2001) also hinder the application of the paleothermometer, which excludes Mediterranean effects (PRUD'HOMME ET AL. 2016). The clarification of these relations and effects has gone beyond the scope of my research project and would need further cooperation with other colleagues to be solved.

d. Experimentally: clumped isotope investigation

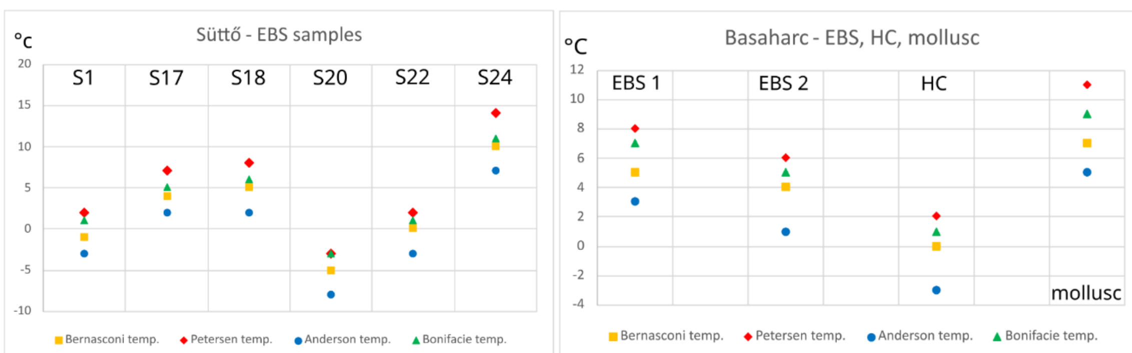
Trial measurements were carried out by a Thermo Scientific 253 Plus 10kV isotope ratio mass spectrometer coupled with Kiel IV automatic carbonate device within the frame of the GINOP-2.3.2-15-2016-00009 – ICER project at the Institute for Nuclear Research, Isotope Climatology and Environmental Research Centre (ICER – Atomki, Debrecen, Hungary) with the contribution of Dr. László Rinyu and Dr. László Palcsu. The measurements and the temperature calibration were made by Dr. László Rinyu.

Samples from two sites were prepared, with two different methods.

- Süttő section: the wet sieved bulk samples were dried out in the oven at 80°C, EBS were separated and cleaned in an ultrasonic bath. 6 EBS were examined from different stratigraphic units (stratigraphy follows NOVOTHNY ET AL. 2011, BARTA 2014): 1) **S1**: 0.75-0.85 m; MIS 2; sandy loess, right below the recent soil; 2) **S17**: 6.55-6.65 m; MIS 3; light brown paleosol; 3) **S18**: 9.15-9.25 m; MIS 5; brown paleosol (P1); 4) **S20**: 9.35-9.45 m; MIS 5; sandy loess (between P1 and P2 paleosols); 5) **S22**: 10.35-10.45 m; MIS 5; brown paleosol (P2); 6) **S24**: 14.15-14.25 m; MIS 6; loess (70 cm below the MIS 5 pedocomplex).

- Basaharc section: the wet sieved bulk samples were dried out at room temperature, EBS were separated and cleaned in an ultrasonic bath. The reasons for drying out the samples at room temperature were to possibly avoid the recrystallization of a potential ACC inclusion. ACC means amorphous calcium carbonate, which precipitates from the milky fluid phase in the oesophagal pouches and transforms to biospheroids (Briones et al. 2008, Gago-Duport et al. 2008, Lee et al. 2008). ACCs tend to recrystallize at 60°C, which may affect the sensitive clumped isotope composition. Two EBS samples were taken from the Basaharc Double 1b paleosol, north-facing wall (MIS7c, stratigraphy follows Horváth et al. 2021), sampling depth 1.20-1.30 m. One hypocoating (HC) and one mollusc sample were also taken from the same level (as these are in-situ and contemporaneous with the sequence built-up, as in Becze-Deák et al. 1997), both complete forms and not reworked.

Until now one conference proceeding was published about an attempt for applying clumped isotope thermometry on earthworm carbonates (Banerjee et al. 2021), but no exact calibration method was available. Therefore, four different calibrations methods were used as Bernasconi et al. (2018) and Petersen et al. (2018) on the CDES25 calibration scale; and Anderson et al. (2021) and Bonifacie et al. (2017) expressed on the CDES90 scale (Bernasconi et al. 2021).



The results are not yet published, since new questions were raised during the data analysis. It is still under clarification which calibration method is the most suitable for the interpretation (Petersen temperatures are the highest).

As an alternative control, results of malacothermometry are available for the MIS 2 and MIS 3 units of Süttő (Krolopp, E. unpublished): mean July temperatures as 11.8-12.9°C for the MIS 2 loess (1.10-5.55 m) and 20.9°C for the MIS 3 paleosol. The S1 and S17 samples show temperatures way below these values. The S24 sample from the MIS 6 loess (70 cm below the pedocomplex) showed surprisingly higher temperature in comparison to the other EBS samples taken from loess (S1 and S20). EBS temperatures were lower from the mollusc clumped isotope temperature in the case of the Basaharc Double 1b paleosol.

The interpretation of the results also requires taking into account other aspects:

- Present-day earthworm activity is seasonally-dependent, with peaks during spring and autumn (Satchell 1967), but it is possible for earthworms that on frost-free winter days they become active again, especially adults and large juveniles (FiBL 2014, Nordström

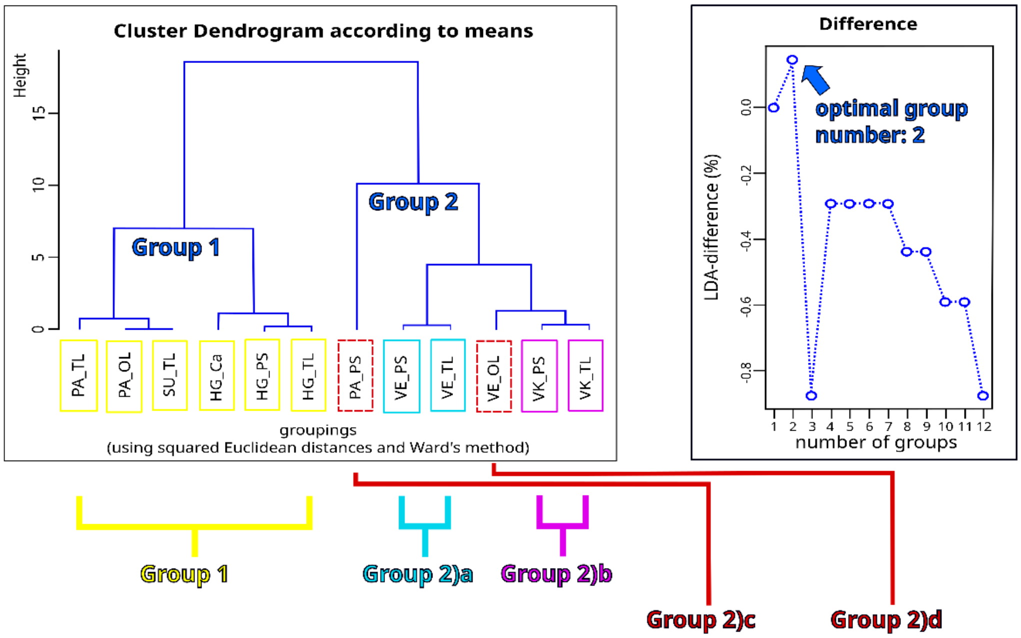
1975). It raises the question, whether EBS produced during winter may be the cause of low clumped isotope temperatures.

- The vital effect for earthworms should be determined, in order to know how accurate the clumped isotope temperatures are and/or how to correct them.
- Other findings of the recent research project (see **subsection 4.**) highlight the possibilities, that 1) EBS from different generations may have been preserved within the same layer – but reflecting different isotopic compositions and thus formation environments; and 2) it has to be taken into consideration, that granule-producer earthworm species have their own characteristic granule morphologies, possibly with effects on the isotopic composition.

New measurements are ongoing recently, considering the above listed factors and with a different preparation and cleaning method in order to rule out impurities on a higher level.

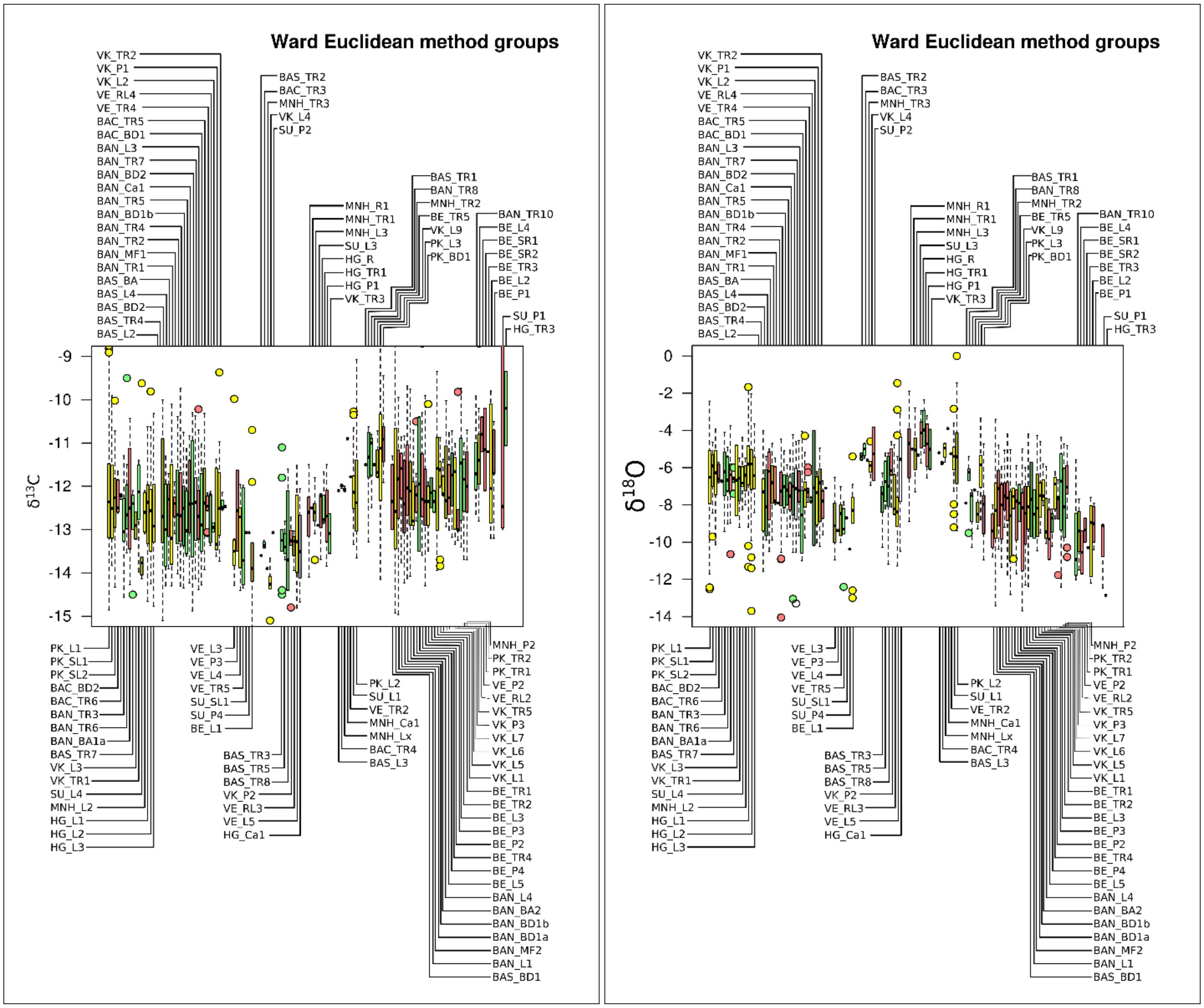
e. Geomathematical methods

Different geomathematical methods were applied on the stable isotope data set. At first the combined cluster and discriminant analysis (CCDA, KOVÁCS ET AL. 2014) was carried out by the help of Dr. József Kovács (ELTE Faculty of Science, Department of Geology) on the Süttő (SU), Verőce (VE), Hévízgyörk (HG), Paks (PA) and Villánykövesd (VK) sections – as these results were prepared in the first phase of the research. The results were presented at the PALEOKLÍMA 2018 conference (BARTA ET AL. 2018) and at the INQUA 2019 (BARTA ET AL. 2019). 471 data pairs ($\delta^{13}C$ and $\delta^{18}O$) were analyzed. Three homogeneous groups originated (*group 1, group 2a and 2b*), which contained samples from loess and paleosol units solely, whereas two inhomogeneous groups (*group 2c and 2d*) included samples from paleosols and stratified, strongly reworked loessic layers. Even though the homogeneous groups contained loess and paleosol units of different age, they indicated that EBS formation may have happened under similar influencing factors. The two inhomogeneous groups were possibly related to regional differences during sequence development, as one group is connected to a mountain foreland area located on a formerly strongly eroding alluvial fan, while the other group was exposed to stronger submediterranean effects.



One result was that the homogeneous groups contained loess and paleosol units of different age and location and despite they indicated EBS formation under similar influencing factors. This finding seemed to be correct from the statistical point of view, but however it pointed out that there should be an explanation for this homogeneity, which is more complex.

After all the stable isotope measurements were ready for each study site, I have received 1513 data pair in total ($\delta^{13}C$ and $\delta^{18}O$). By the help of Dr. József Kovács (ELTE Faculty of Science, Department of Geology), the cluster analysis of the complex database was carried out by using Ward's method with Euclidean distances. Based on the generated clusters, I have visualized the data on box-and-whiskers plots. The main results were that the stable isotope values change in the opposite direction: where the $\delta^{13}C$ values are shifted towards the negative direction, there the $\delta^{18}O$ values are shifted towards the more positive direction – and vice versa. The groups provided by the analysis are still very heterogeneous, they include stratigraphic units of different kinds, different age and different locations. The box-and-whiskers plots were prepared in many different combinations: for each sequence according to depth, for each stratigraphic unit type from all sequences, for co-eval units.



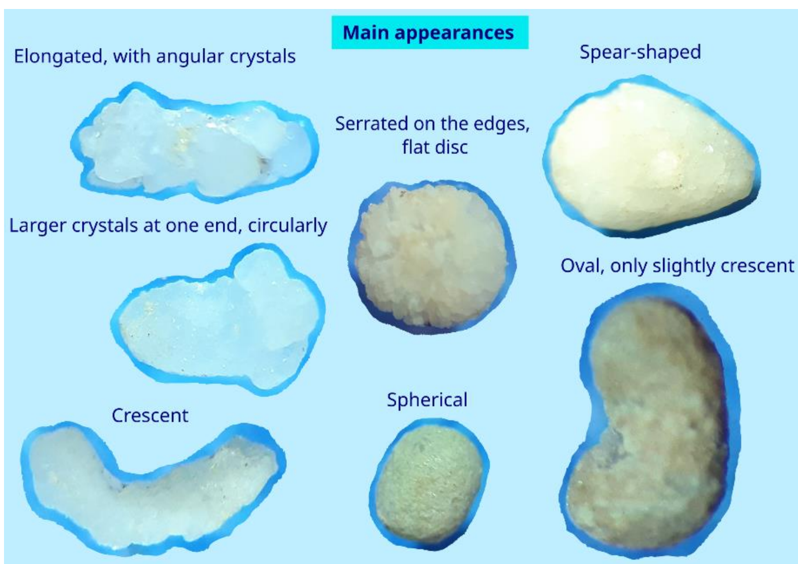
During the last month of the research project, I have applied the t-student test on my EBS data, as following the idea of PRUD'HOMME ET AL. (2018) – but not only for one sequence. The t-student test decides whether the mean values of two populations are statistically different or not at a significance threshold of 95% (PRUD'HOMME ET AL. 2018). By the help of Ágnes Novothny and Tamás Országh we have established a decision tree for the analysis. The method helped to classify the EBS data from the various stratigraphic units, based on the existence of a normal distribution and to choose which statistical analysis should be the next step. The decision tree contained the following methods: Shapiro-Wilk's normality test, t-student test, Welch t-test, Kruskal-Wallis test and Wilcoxon test. The stratigraphic units were tested in different combinations too (within a sequence and between sequences), as e.g. EBS populations of coeval paleosols. The p-value <0.05 refers to different populations, whereas p-value >0.05 validates the null hypothesis and indicates similar populations. The following table shows the main results of the t-test of the whole database (***Table 1.***).

		loess vs. paleosol		loess vs. transition		paleosol vs. transition		paleosol vs recent soi		recent soil vs. loess	
		p-value		p-value		p-value		p-value		p-value	
		δ13C	δ18O	δ13C	δ18O	δ13C	δ18O	δ13C	δ18O	δ13C	δ18O
Basaharc Northern	BAN	0,7102	**0,00025**	**0,04973**	**0,0009811**	0,08744	0,795	-	-	-	-
Basaharc Southern	BAS	0,5912	0,6128	0,08861	0,8662	**0,01128**	0,5242	-	-	-	-
Malá nad Hronom	MNH	**0,02229**	**0,00063**	0,5597	0,129	0,8148	0,874	**0,01975**	**0,01625**	0,4203	0,7553
Beremend	BE	0,2934	0,7793	0,7826	0,3686	0,6591	0,3006	-	-	-	-
Villánykövesd	VK	0,1559	0,7452	0,8069	0,3752	0,283	0,3971	-	-	-	-
Verőce	VE	0,249	0,3396	0,9036	0,6535	0,2709	0,7921	-	-	-	-
Süttő	SU	0,562	**0,029**	-	-	-	-	-	-	-	-
Paks	PK	**4,30E-05**	**0,00481**	0,6271	**0,02673**	**0,00785**	0,5402	-	-	-	-
Hévízgyörk	HG	0,8163	0,1659	0,2138	0,7414	0,2414	0,36	0,9881	0,6522	0,8146	0,138
all profiles together	ALL DATA	0,05444	**2,42E-06**	**0,00993**	0,1085	**0,00021**	**0,03339**	**0,00815**	**6,82E-05**	**0,03994**	**0,00074**

Table 1: Values highlighted in purple indicate different populations (p-values <0.05)

4. Conclusions

The application of the primary geomathematical methods on the dataset (CCDA and hierarchical cluster analysis) resulted in an unclear pattern of the stable isotope signals and led to the maze of other statistical approaches (see details in **subsection 3.e**). By studying the heterogeneity of the clusters, the idea came that the temporal distribution of EBS producing earthworm generations may cause an issue for the habitats of the Carpathian Basin, along with a possibly higher significance of the morphology variance of EBS.



Nevertheless, I have started to catalogize the most frequent appearances. Since I have 50096 pcs of EBS in total, I could not finish to quantify them by morphology. Following the paper of Canti and Piearce (2003), I tried to compare the paleo-EBS types to the published recent ones and I have realized that very probably my samples contained not only *Lumbricus terrestris* EBS, but *Aporrectodea longa* EBS and *Lumbricus rubellus* EBS too. The first two worms are anecic, deep-dwelling species, whereas the latter one is epigeic, leaf litter dwelling species.

At this point only a couple of months were left of my research project and I did not have the capacity to complete a systematic catalogue of the morphologies of the whole data set, and I did not have any opportunity left to carry out new stable isotope measurements and experiment with my theory.

The morphological findings along with stable isotope compositions and the theory concerning the life range of earthworms were examined in detail for the Basaharc section, as a joint study containing comparisons to other methods as soil thin section description, magnetic susceptibility and grain size measurements. The approach was presented on the INQUA Congress 2023 (HORVÁTH ET AL. 2023). *The manuscript is currently being under preparation*. The refined theory and new results will be presented on the LoessFest 2024 (accepted abstract, HORVÁTH ET AL. 2024).

Along with the theory, a new sequence development scheme was prepared (see ***Figure 1.***), which highlights the possibilities of different EBS generations in relation to the built-up process of the sequence. It considers that loess-paleosol sequences have pedosedimentary aspects through formation (e.g. KEMP 1999, 2001) and that during dust accretion the microecosystems remain active and their development follows the surface level according to the velocity of dust accumulation (e.g. CATT 1990) – and thus their biomineralized products remain in the sediment (BECZE-DEÁK ET AL. 1997). The studied sequences of my research project have shown that, in contrast to the Western European sites, loess units of the Carpathian Basin contained EBS in high amount.

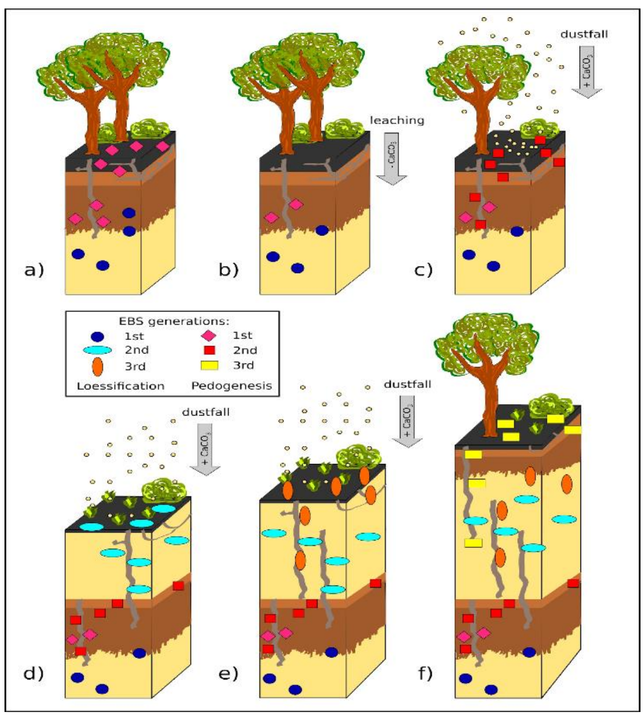


Figure 1: Loess-paleosol sequence development versus EBS generations

When considering the sequence built-up and especially the soil development during the interglacials/interstadials, *<u>the results of my research project draw attention to the following issues</u>*:

1) As the various EBS morphologies have shown, it is possible that different earthworm species were inhabiting that ecological niche. Among the earthworm types there were

co-existing leaf-litter dwelling (as *Lumbricus rubellus*) and deep dwelling (as *Lumbricus terrestris*, *Aporrectodea longa*) species. It is possible that the "release" of EBS happened not solely near the surface, but deeper too (e.g. CANTI AND PIEARCE 2003). Different generations of earthworms could have produced EBS and released them in various depths – as causing a large variability in the stable isotope composition too (and causing difficulties during interpretation of the data, as I have highlighted it formerly).

2) Paleosols undergone significant leaching, which can cause the dissolution of EBS. The fact that paleosols still contain a certain amount of EBS raises the question, that → whether the EBS are connected to the last phase of paleosol development (not complete decalcification of paleosol or extremely good retention of EBS); OR → the EBS derive from the phase of the transition to the next glacial, when dust accretion begins and adds carbonate to the system again. This latter case could justify the merging of generations and the similarities between the statistical populations of loesses, paleosols and transitions (see t-test).

After many detours and interpretation experiments, the results of the project laid the foundations for a deeper understanding of the paleoenvironmental role of EBS. New research possibilities are highlighted, which are intended to reveal the real nature and significance of these features by classifying EBS morphologies based on species and size; and to discover which earthworm species produce the most applicable EBS by preparing special tests (high amounts of samples from the same layer). This approach would be needed in order to discover that depth/level, where only minimal mixing of EBS happened and which would provide clearer paleoenvironmental signals. I hope that my wish for perfection and to achieve the deepest understanding is worth to be appreciated, even though the manuscripts are not yet prepared. I am grateful to have received this research project and I do hope that my contribution is valuable.

5. Participation in collaborations:

- NRDIO K119366 project entitled as *„Genesis and postpedogenic alterations of paleosols in loesses and possibilities for paleoenvironmental reconstruction*" (2016-2020, led by Dr. Erzsébet Horváth);
- ELKH SA-41/2021: Soil carbon budget - from nanoscale organic-inorganic interactions to the global carbon cycle (2022 – 2024, led by Dr. Attila Demény);
- International cooperation with the Leibniz Institute for Applied Geophysics (LIAG, Hannover; Prof. Dr. Manfred Frechen);
- GINOP- 2.3.2-15-2016-00009: Isotope Climatology and Environmental Research Centre (ICER) (2018 – 2020), → clumped isotope measurements (Dr. László Rinyu and Dr. László Palcsu).

6. **Publications**

   a. **Conference abstracts**

1. **<u>BARTA, G.</u>**, BRADÁK, B., KOVÁCS, J., HORVÁTH, E., FRECHEN, M. 2018. Miről mesél a földigiliszta bioszferoidok stabilizotóp összetétele? – Interpretációs lehetőségek a kombinált klaszter és diszkriminancia analízis (CCDA) segítségével. PALEOKLÍMA 2018, 2018-11-27 MTA, Budapest, Hungary
2. **<u>BARTA, G.</u>**, BRADÁK, B., KOVÁCS, J., HORVÁTH, E., FRECHEN, M. 2019. Classification of stable carbon and oxygen isotope composition of earthworm biospheroids from loesses by combined cluster and discriminant analysis. 20th Congress of the International Union for Quaternary Research (INQUA), (2019) P-1870
3. HORVÁTH, E., BRADÁK, B., CSONKA, D., NOVOTHNY, Á., SZEBERÉNYI, J., VÉGH, T., **<u>BARTA, G.</u>** 2023. Joint micromorphological and stable isotope study of Middle Pleistocene paleosols from the European Loess Belt (Basaharc, Hungary). 21st Congress of the International Union for Quaternary Research (INQUA), (2023) P-1555
4. HORVÁTH, E., DEMÉNY, A., RINYU, L., NOVOTHNY, Á., FRECHEN, M., **<u>BARTA, G.</u>** 2024. Combined assay method to determine the environmental significance of earthworm biospheroids in loess-paleosol series. LoessFest 2024, June 19-22, Mainz, Germany (https://www.geomorphologie.uni-mainz.de/loessfest2024/)

   b. **Scientific papers**

1. BRADÁK, B., KERESZTURI, Á., STEINMANN, V., GOMEZ, C., CSONKA, D., HYODO, M., SZEBERÉNYI, J., NOVOTHNY, Á., VÉGH, T., **<u>BARTA, G.</u>**, MEDVEĎOVÁ, A., ROSTINSKY, P., MIHÁLY, E., JÓ, V., HORVÁTH, E. 2022. The magnetic susceptibility of Pleistocene paleosols as a martian paleoenvironment analog. Icarus 387, 115210
2. BRADÁK, B., CSONKA, D., NOVOTHNY, Á., SZEBERÉNYI, J., MEDVEĎOVÁ, A., ROSTINSKY, P., FEHÉR, K., **<u>BARTA, G.</u>**, VÉGH, T., KISS, K., MEGYERI, M. 2021. Late Pleistocene paleosol formation in a dynamic aggradational microenvironment – A case study from the Malá nad Hronom loess succession (Slovakia). Catena 199, 105087
3. CSONKA, D., BRADÁK, B., **<u>BARTA, G.</u>**, SZEBERÉNYI, J., NOVOTHNY, Á., VÉGH, T., SÜLE, G.T., HORVÁTH, E. 2020. A multi-proxy study on polygenetic middle- to late Pleistocene paleosols in the Hévízgyörk loess-paleosol sequence (Hungary). Quaternary International 552, 25-35.
4. HORVÁTH, E., NOVOTHNY, Á., **<u>BARTA, G.</u>**, CSONKA, D., VÉGH, T., BRADÁK, B. 2019. Palaeosoils as indicators of local palaeoenvironmental changes – mosaics from the Hungarian loess studies. In: DEÁK, J., AMPE, C., HINSCH MIKKELSEN, K. (eds.). Soils as records of Past and Present. Raakvlak, Archaeology, Monuments and Landscapes of Bruges and Hinterland, Belgium, 271-278.
5. NOVOTHNY, Á., **<u>BARTA, G.</u>**, VÉGH, T., BRADÁK, B., SURÁNYI, G., HORVÁTH, E. 2020. Correlation of drilling cores and the Paks brickyard key section at the area of Paks, Hungary. Quaternary International 552, 50-61.
6. SZEBERÉNYI, J., MEDVEĎOVÁ, A., ROSTINSKY, P., **<u>BARTA, G.</u>**, NOVOTHNY, Á., CSONKA, D., VICZIÁN, I., HORVÁTH, E., VÉGH, T. 2020. Investigation of spectral properties of different Quaternary paleosols and parent materials. Hungarian Geographical Bulletin, 69, 3-15.

7. SZEBERÉNYI, J., KOVÁCS, J., BRADÁK, B., **BARTA, G.**, CSONKA, D., MEDVEĎOVÁ, A., ROSTINSKY, P., KISS, K., VARGA, GY. 2020. Experiencing new perspectives in the application of reflectance spectroscopy in loess research. Quaternary International 552, 36-49.

Budapest, 25.05.2024

Dr. Barta Gabriella

7. References

ANDERSON, N.T., KELSON, J.R., KELE, S., DAËRON, M., BONIFACIE, M., HORITA, J. ET AL. 2021. A unified clumped isotope thermometer calibration (0.5-1,100°C) using carbonate-based standardization Geophysical Research Letters 48, e2020GL092069

BANERJEE, S., GHOSH, P., THAMIZHARASAN, S., VERSTEEGH, E.A.A., BLACK, S., HODSON, M.E. 2021. Clumped isotope thermometry in earthworm carbonates. Goldschmidt 2021 Abstract, https://doi.org/10.7185/gold2021.6679

BARTA, G. 2014. Paleoenvironmental reconstruction based on the morphology and distribution of secondary carbonates of the loess-paleosol sequence at Süttő, Hungary. Quaternary International 319, 64-75.

BARTA, G. 2016. Analysis of secondary carbonates from the young loess-paleosol sequences of the Carpathian Basin – especially regarding their paleoenvironmental role. PhD dissertation, Eötvös Loránd University, Budapest, 207 p., DOI: 10.15476/ELTE.2016.044.

BARTA, G., BRADÁK, B., KOVÁCS, J., HORVÁTH, E., FRECHEN, M. 2018. Miről mesél a földigiliszta bioszferoidok stabilizotóp összetétele? – Interpretációs lehetőségek a kombinált klaszter és diszkriminancia analízis (CCDA) segítségével. PALEOKLÍMA 2018, 2018-11-27 MTA, Budapest, Hungary

BARTA, G., BRADÁK, B., KOVÁCS, J., HORVÁTH, E., FRECHEN, M. 2019. Classification of stable carbon and oxygen isotope composition of earthworm biospheroids from loesses by combined cluster and discriminant analysis. 20th Congress of the International Union for Quaternary Research (INQUA), (2019) P-1870

BECZE-DEÁK, J., LANGOHR, R., VERRECCHIA, E.P. 1997. Small scale secondary $CaCO_3$ accumulations in selected sections of the European loess belt. Morphological forms and potential for paleoenvironmental reconstruction. Geoderma 76, 221-252.

BERNASCONI, S.M., MÜLLER, I.A., BERGMANN, K.D., BREITENBACH, S.F.M., FERNANDEZ, A., HODELL, D.A. ET AL. 2018. Reducing uncertainties in carbonate clumped isotope analysis through consistent carbonate-based standardization. Geochemistry, Geophysics, Geosystems 19, 2895-2914.

BERNASCONI, S.M., DAËRON, M., BERGMANN, K.D., BONIFACIE, M., MECKLER, A.N., AFFEK, H.P. ET AL. 2021. InterCarb: A community effort to improve interlaboratory standardization of the carbonate clumped isotope thermometer using carbonate standards. Geochemistry, Geophysics, Geosystems 22, e2020GC009588

Bonifacie, M., Calmels, D., Eiler, J.M., Horita, J., Chaduteau, C., Vasconcelos, C., Agrinier, P., Katz, A., Passey, B.H., Ferry, J.M., Bourrand, J.-J. 2017. Calibration of the dolomite clumped isotope thermometer from 25 to 350°C, and implications for a universal calibration for all (Ca, Mg, Fe)CO_3 carbonates. Geochimica et Cosmochimica Acta 200, 255-279.

Bottyán, E., Czuppon, Gy., Weidinger, T., Haszpra, L., Kármán, K. 2017. Moisture source diagnostics and isotope characteristics for precipitation in east Hungary: implications for their relationship. Hydrological Sciences Journal, 62/12, 2049-2060.

Bradák, B., Kiss, K., Barta, G., Varga, Gy., Szeberényi, J., Józsa, S., Novothny, Á., Kovács, J., Markó, A., Mészáros, E., Szalai, Z. 2014. Different paleoenvironments of Late Pleistocene age identified in Verőce outcrop, Hungary: Preliminary results. Quaternary International 319, 119-136.

Bradák, B., Csonka, D., Novothny, Á., Szeberényi, J., Medveďová, A., Rostinsky, P., Fehér, K., Barta, G., Végh, T., Kiss, K., Megyeri, M. 2021. Late Pleistocene paleosol formation in a dynamic aggradational microenvironment – A case study from the Malá nad Hronom loess succession (Slovakia). Catena 199, 105087

Briones, M.J.I., López, E., Méndez, J., Rodrígez, J.B., Gago-Duport, L. 2008. Biological control over the formation and storage of amorphous calcium carbonate by earthworms. Mineralogical Magazine 72/1, 227-231.

Canti, M.G., Piearce, T.G. 2003. Morphology and dynamics of calcium carbonate granules produced by different earthworm species. Pedobiologia 47, 511-521.

Catt, J.A. 1990. Paleopedology manual. Quaternary International 6, 1-95.

Csonka, D., Bradák, B., Barta, G., Szeberényi, J., Novothny, Á., Végh, T., Süle, G.T., Horváth, E. 2020. Quaternary International 552, 25-35.

Dudás, N. 2012. Villánykövesdi téglagyár lösz-paleotalaj sorozatának vizsgálata. MSc thesis, ELTE TTK, Budapest (in Hungarian)

Durand, N., Monger, H.C., Canti, M.G. 2010. Calcium carbonate features. In: Stoops, G., Marcelino, V., Mees, F. (Eds.), Interpretation of micromorphological features of soils and regoliths. Elsevier 2010, 149-194. (DOI: 10.1016/B978-0-444-53156-8.00009-X)

Gago-Duport, L., Briones, M.J.I., Rodríguez, J.B., Covelo, B. 2008. Amorphous calcium carbonate biomineralization in the earthworm's calciferous gland: Pathways to the formation of crystalline phases. Journal of Structural Biology 162, 422-435.

Horváth, E., Bradák, B., Szeberényi, J., Végh, T., Csonka, D., Novothny, Á. 2021. Toward the understanding of (post)pedogenic processes behind the peaks and lows of commonly used climate proxies. 2nd International Conference on Quaternary Sciences, Gorgan, Iran 2021, Paper 46406

HORVÁTH, E., BRADÁK, B., CSONKA, D., NOVOTHNY, Á., SZEBERÉNYI, J., VÉGH, T., BARTA, G. 2023. Joint micromorphological and stable isotope study of Middle Pleistocene paleosols from the European Loess Belt (Basaharc, Hungary). 21st Congress of the International Union for Quaternary Research (INQUA), (2023) P-1555

HORVÁTH, E., DEMÉNY, A., RINYU, L., NOVOTHNY, Á., FRECHEN, M., BARTA, G. 2024. Combined assay method to determine the environmental significance of earthworm biospheroids in loess-paleosol series. LoessFest 2024, June 19-22, Mainz, Germany (https://www.geomorphologie.uni-mainz.de/loessfest2024/)

HUM, L. 2001. Délkelet-Dunántúli lösz-paleotalaj sorozatok keletkezésének rekonstrukciója őslénytani vizsgálatok alapján. Földrajzi Közlöny 131/1-2, pp. 235-251. (in Hungarian with English abstract)

KEMP, R.A. 1999. Micromorphology of loess-paleosol sequences: a record of paleoenvironmental change. Catena 35, 179-196.

KEMP, R.A. 2001. Pedogenic modification of loess: significance for palaeoclimatic reconstructions. Earth-Science Reviews 54, 145-156.

KOENIGER, P., BARTA, G., THIEL, C., BAJNÓCZI, B., NOVOTHNY, Á., HORVÁTH, E., TECHMER, A., FRECHEN, M. 2014. Stable isotope composition of bulk and secondary carbonates from the Quaternary loess-paleosol sequence in Süttő, Hungary. Quaternary International 319, 38-49.

KOVÁCS, J., KOVÁCS, S., MAGYAR, N., TANOS, P., HATVANI, I.G., ANDA, A. 2014. Classification into homogeneous groups using combined cluster and discriminant analysis. Environmental Modelling & Software 57, 52-59.

KROLOPP, E. 2008. Süttő, Haraszti bánya, malakológiai analízis. – unpublished manuscript, in Hungarian.

LEE, M.R., HODSON, M.E., LANGWORTHY, G. 2008. Earthworms produce granules of intricately zoned calcite. Geology 36/12, 943-946.

MILINKÓ, I. 2019. A basaharci lösz-paleotalaj szelvény mészdinamikája. MSc thesis, ELTE Eötvös Loránd University, Faculty of Science, Budapest, 73 p. (in Hungarian)

NOVOTHNY, Á., FRECHEN, M., HORVÁTH, E., BRADÁK, B., OCHES, E.A., MCCOY, W.D., STEVENS, T. 2009. Luminescence and amino acid racemization chronology of the loess-paleosol sequence at Süttő, Hungary. Quaternary International 198, 62-76.

NOVOTHNY, Á., FRECHEN, M., HORVÁTH, E., WACHA, L., ROLF, C. 2011. Investigating the penultimate and last glacial cycles of the Süttő loess section (Hungary) using luminescence dating, high-resolution grain size, and magnetic susceptibility data. Quaternary International 234, 75-85.

NORDSTRÖM, S. 1975. Seasonal activity of lumbricids in southern Sweden. Oikos 26, 307-315.

PETERSEN, S.V., DEFLIESE, W.F., SAENGER, C., DAËRON, M., HUNTINGTON, K.W., JOHN, C.M. ET AL. 2019. Effects of improved ^{17}O correction on interlaboratory agreement in clumped isotope calibrations, estimates of mineral-specific offsets, and temperature dependence of acid digestion fractionation. Geochemistry, Geophysics, Geosystems 20, 3495-3519.

Prud'homme, C., Lécuyer, C., Antoine, P., Moine, O., Hatté, C., Fourel, F., Martineau, F., Rousseau, D.-D. 2016. Paleotemperature reconstruction during the Last Glacial from $\delta^{18}O$ of earthworm calcite granules from Nussloch loess sequence, Germany. Earth and Planetary Science Letters 442, 13-20.

Prud'homme, C., Lécuyer, C., Antoine, P., Hatté, C., Moine, O., Fourel, F., Amiot, R., Martineau, F., Rousseau, D.-D. 2018. $\delta^{13}C$ signal of earthworm calcite granules: A new proxy for palaeoprecipitation reconsructions during the Last Glacial in western Europe. Quaternary Science Reviews 179, 158-166.

Prud'homme, C., Moine, O., Mathieu, J., Saulnier-Copard, S., Antoine, P. 2019. High resolution quantification of earthworm calcite granules from western European loess sequences reveals stadial-interstadial climatic variability during the Last Glacial. Boreas 48, 257-268.

Pustovoytov, K., Terhorst, B. 2004. An isotopic study of a Late Quaternary loess-paleosol sequence in SW Germany. Revista Mexicana de Ciencias Geológicas 20/001, 88-93.

Technical Guide on Earthworms, Order no. 1629, International edition; FiBL 2014 (www.tilman-org.net, www.shop.fibl.org)

Thiel, C., Horváth, E., Frechen, M. 2014. Revisiting the loess/palaeosol sequence in Paks, Hungary: A post-IR IRSL based chronology for the 'Young Loess Series'. Quaternary International 319, 88-98.

Satchell, J. E. 1967. Lumbricidae. In Soil Biology (eds. A. Burges and F. Raw), Academic Press, London and New York, 259–322.

Szeberényi, J., Barta, G., Bradák, B., Csonka, D., Horváth, E., Novothny, Á., Végh, T. 2018. Reflektancia mérésekből származtatott színindexek alkalmazása a basaharci feltárás szelvényein. In: Fazekas, I., Kiss, E., Lázár, I. (eds.) Földrajzi tanulmányok 2018 Debrecen, Hungary, MTA DAB Földtudományi Szakbizottság, 95-99.

Szeberényi, J., Medveďová, A., Rostinsky, P., Barta, G., Novothny, Á., Csonka, D., Viczián, I., Horváth, E., Végh, T. 2020a. Investigation of spectral properties of different Quaternary paleosols and parent materials. Hungarian Geographical Bulletin 69 (1), 3-15.

Szeberényi, J., Kovács, J., Bradák, B., Barta, G., Csonka, D., Medveďová, A., Roštínský, P., Kiss, K., Varga, G. 2020b. Experiencing new perspectives in the application of reflectance spectroscopy in loess research. Quaternary International 552, 36-49.

Újvári, G. 2004. A Villánykövesdi Téglagyár rétegsorának malakofaunája. Malakológiai Tájékoztató 22, 39-49. (in Hungarian with English abstract)

Versteegh, E.A.A., Black, S., Canti, M.G., Hodson, M.E. 2013. Earthworm-produced calcite granules: A new terrestrial palaeothermometer? Geochimica et Cosmochimica Acta 123, 351-357.