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The chronology of the Šarengrad II loess-palaeosol section (Eastern Croatia)

Lara Wacha*[1, 2], Lidija Galović[1], László Koloszár[3], Árpád Magyari[3, 4], Géza Chikán[3] and István Marsi[3]

[1] Department of Geology, Croatian Geological Survey, Sachsova 2, HR-10000 Zagreb, Croatia; (*corresponding author: lwacha@hgi-cgs.hr)

[2] S3 Geochronology and Isotope Hydrology Section, Leibniz Institute for Applied Geophysics (LIAG), Stilleweg 2, D-30655 Hannover, Germany

[3] Geological and Geophysical Institute of Hungary, Stefániaút 14, HU-1143 Budapest, Hungary

[4] MOL Plc, Októberhuszonharmadika u. 18, HU-1117 Budapest, Hungary

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ABSTRACT

A 23m thick succession of Quaternary deposits was investigated at Šarengrad, Eastern Croatia. A geochronological study was performed on loess samples using luminescence dating. An elevated temperature post-IR infrared stimulated luminescence (post-IR IRSL) dating protocol was applied. Performance tests showed that the protocol is suitable for the samples under study. The post-IR IRSL based chronology implies that, contradictory to previous correlations, a Middle Pleistocene loess-palaeosol sequence is exposed at Šarengrad II and not the Last Glacial – Interglacial cycle. Based on ages, the lowermost loess is correlated to oxygen isotope stage (OIS) 9-10 and the double palaeosol at the top of the section most likely correlates with OIS7. The Last Glacial record is exposed at the nearby Šarengrad I section. Furthermore, a heavy and light mineral analysis was performed on the investigated section as well as on the previously investigated Šarengrad I section, showing that the mineral association is typical for Quaternary deposits of the Pannonian basin.

Keywords: loess-palaeosol, Šarengrad, geochronology, luminescence dating, Middle Pleistocene

1. INTRODUCTION

In Croatia (Fig. 1a) two major loess regions can be distinguished: the North Adriatic loess region related to the river Po in Northern Italy and its tributaries (WACHA et al., 2011 a, b; MIKULČIĆ PAVLAKOVIĆ et al., 2011), and the Danube loess region in the eastern part of the country (Fig. 1b) (GALOVIĆ et al., 2009, 2011; WACHA & FRECHEN, 2011). In eastern Croatia, the sedimentary record is more or less continuous, from the Neogene to the Quaternary, and is related to the sedimentary evolution of the Pannonian basin (PAVELIĆ, 2001) and influenced by active tectonics during the Quaternary (PRELOGOVIĆ et al., 1998). The Pleistocene sediment succession is closely related to the major rivers of the area (the Sava, Danube and Drava Rivers) and their tributaries, and mainly consists of alluvial sediments and thick loess deposits along the Danube River. The thickness of loess and loess-like sediments in eastern Croatia ranges from 0.5 to 60 m (BOGNAR, 1979). Recently, several loess-palaeosol sequences were described and investigated in more detail along the Danube river in Croatia; the Erdut,

Figure 1: a, b and c: Location maps of the investigated Šarengrad II loess-palaeosol section in eastern Croatia and its relationship to the Šarengrad I section referred to in this study (https://maps.google.hr/maps?hl=hr&tab=wl).

Šarengrad and Zmajevac sections (GALOVIĆ et al., 2009, 2011; HUPUCZI et al., 2010; MOLNÁR et al., 2010; BANAK et al., 2012) and the Gorjanović loess section in Vukovar (WACHA & FRECHEN, 2011). A detailed stratigraphy of the Danube loess from the nearby area in Vojvodina (Serbia) was presented by MARKOVIĆ et al. (2012, and references therein). This stratigraphy follows the Chinese loess stratigraphic system (KUKLA, 1987) and is supported by detailed geochronological, palaeomagnetic and other data (MARKOVIĆ et al., 2012; FITZSIMMONS et al., 2012).

The first luminescence data for dating the investigated area were presented by SINGHVI et al. (1989). They applied thermoluminescence (TL) dating to several loess-palaeosol sequences in the Carpathian basin, one of which was the Erdut section in Croatia. Based on the ages, SINGHVI et al. (1989) provided a correlation of loess-palaeosol sequences in the Pannonian basin following the pedostratigraphy provided by BRONGER (1975, 1976), and concluded that the Mende-Base (MB) soil from the Hungarian loess stratigraphy (the F_5 palaeosol after BRONGER, 1975, 1976, 2003), represents the last interglacial, since it is much older than previously assumed. GALOVIĆ et al. (2009), applied the infrared stimulated luminescence (IRSL) dating on three loess-palaeosol sequences from Eastern Croatia; the Zmajevac, Erdut and Šarengrad (referred to as Šarengrad I in this study) sections. The age estimates presented in GALOVIĆ et al. (2009) from the loess above the uppermost palaeosol at Zmajevac and Erdut, are in good agreement with the TL results of SINGHVI et al. (1989), indicating that most of the investigated loess-palaeosol sections correlate to the last glacial period. GALOVIĆ et al. (2009) measured the feldspar signal from polymineral fine grains, usinga multiple aliquot additive dose (MAAD) protocol. Recently, WACHA & FRECHEN (2011) provided a detailed geochronological framework for the Gorjanović loess section in Vukovar, using a newly developed post-IR IRSL dating protocol (BUYLAERT et al., 2009, 2012; THIEL et al., 2011a,b).

In this study, luminescence dating results from the Šarengrad II loess-palaeosol section, (a newly exposed section at the town of Šarengrad), are presented and a more robust geochronological framework of the exposed sequence is proposed. For this purpose, the elevated temperature post-IR infrared stimulated luminescence dating protocol, (post-IR IRSL), was applied to loess samples (e.g. THIEL et al., 2011a, b; WACHA & FRECHEN, 2011). Furthermore, a comparison with the previously investigated Šarengrad I section (GALOVIĆ et al., 2009, 2011) is presented, based on both the geochronological and mineralogical investigations. This study presents a contribution to the unravelling of the loess-palaeosol evolution in eastern Croatia, and is also another step forward to establishing a more reliable stratigraphy of loess therein. These results present the first dating attempt of older loess-palaeosol sequences in Croatia and therefore can be used to apply high-resolution proxy studies.

2. GEOLOGICAL SETTING AND THE SEDIMENT SUCCESSIONS

Eastern Croatia (Fig 1b, 2) is mostly covered by Quaternary deposits, which were deposited on Mesozoic, Miocene and Pliocene sediments, as well as magmatic and metamorphic rocks. Pliocene clastites transgressively overlie Middle Triassic and Miocene sediments. Since the end of the Miocene, the slow uplift continues in the central part of the Fruška gora (gora = mountain). Subsidence of its northern and southern slopes has resulted in increased sedimentation during the Pliocene and the Quaternary (Fig. 2). Lake and

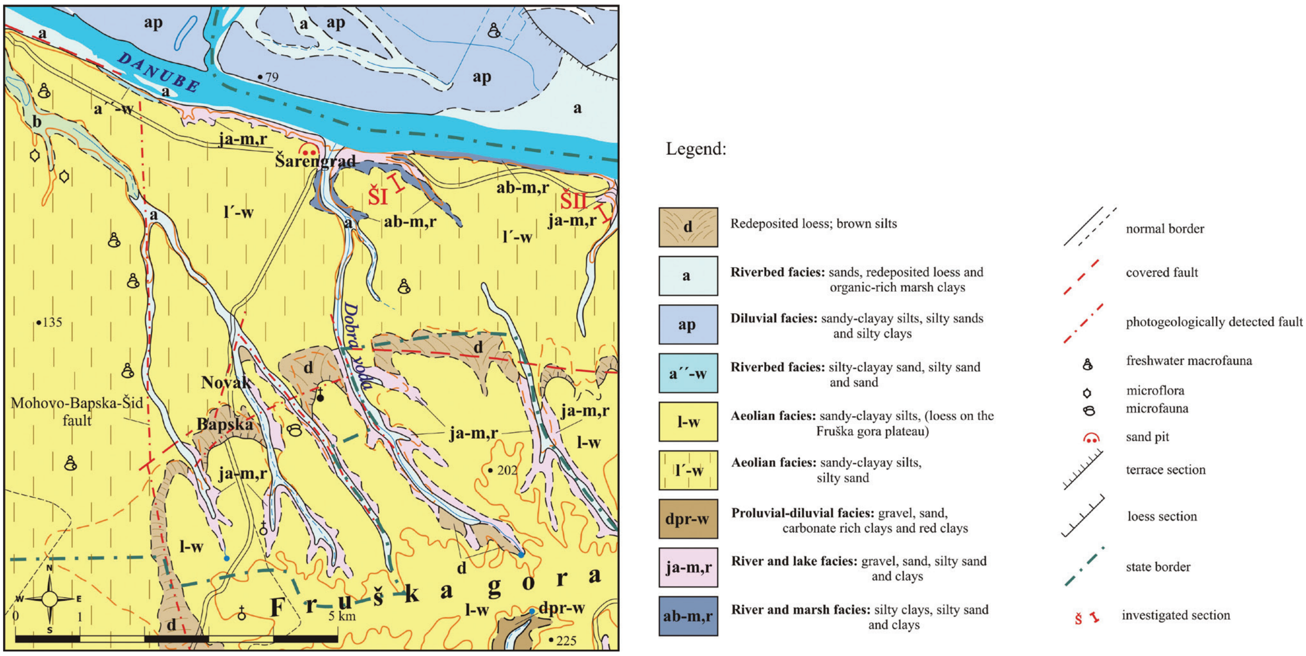

Figure 2: Geological map of the investigated area with the Šarengrad I and II sections indicated, (modified after ČIČULIĆ-TRIFUNOVIĆ & GALOVIĆ,1983).

marsh sediments were deposited during the Lower and Middle Pleistocene (ČIČULIĆ-TRIFUNOVIĆ & GALOVIĆ, 1983, 1985). These sediments were covered by Middle and Upper Pleistocene loess (BOGNAR, 1979). The loess originates from the Danube River and its tributaries; there it forms plateaus and covers the slopes of the Fruška gora (Fig. 2). The Vukovar loess plateau continues to the Fruška gora.They are separated by the N – S spreading Mohovo – Bapska – Šid fault (ČIČULIĆ-TRIFUNOVIĆ & GALOVIĆ, 1983, 1985). The Danube River undercuts and erodes the northern slopes, creating steep loess exposures. Numerous loess-palaeosol sections have been described in this area (MARKOVIĆ et al., 2004, 2006, 2007, 2011; ANTOINE at al., 2009; GALOVIĆ et al., 2009, 2011). The town of Šarengrad is located on the Danube River, between the towns of Vukovar and Ilok, in the easternmost part of Croatia and the Fruška gora to the Southeast (Fig. 1 a, b, c; 2).

The **Šarengrad I** section (45°13’21” N, 19°17’50” E) is situated in a road cut, about one kilometre east of the centre of Šarengrad (Fig. 1c). The elevation is about 110 m above sea level (a.s.l.). The 16 m thick loess-palaeosol sequence has been described in detail by GALOVIĆ et al. (2009) including a geochronological framework. At the **Šarengrad I** section (Fig. 3) the oldest hydromorphic palaeosol contains Fe/Mn-precipitates. The lowermost loess horizon has a thickness of 140 cm and gave an IRSL age estimate of 86.6 ± 8.6 ka (GALOVIĆ et al., 2009). This layer is truncated and covered by laminated sediment with wave-ripple marks in the lower part. The well-developed argillic dark brown palaeosol is characterised by rubification and illuviation processes. The loess on top of that palaeosol is covered by a brown palaeosol. The uppermost palaeosol is weakly developed and covered by loess which is influenced by recent pedogenesis. An IRSL age of 55.3 ± 5.5 ka for the uppermost loess can be correlated to the oxygen isotope stage (OIS) 3 – 4. However, due to the methodological limitations of the previous study, an older age estimate is not entirely excluded and the data as well as the interpretation should be revised. For a detailed lithostratigraphical subdivision of the profile see GALOVIĆ et al. (2009).

The **Šarengrad II** section (45°13’54’’N, 19°17’50’’E) is located near the main road, at the eastern town exit and about 2 km away from the Šarengrad I section. The elevation is about 115 m a.s.l (Fig 1c).

The base of the investigated **Šarengrad II** section consists of about 1 m of yellow to light-brown micaceous, fine-grained sand, and weakly cemented sand of fluvial origin (Figs. 3, 4). The sand fines upwards into a 3 m thick greyish sandy-silty clay, and clay with red hue due to dispersed limonite (Fig. 3, 4). This clay is overlain by a greyish, variegated clay with red and brown stripes, and patches caused by enrichment in dispersed limonite and manganese precipitates. The lower part of this horizon is intercalated with a thin layer of carbonate precipitates. The clays represent marshy sediments (HUPUCZI et al., 2010) and are overlain by greyish-yellow clayey silt containing calcareous nodules, which represents a transitional infusion loess layer between swamp deposits and loess. The transition between these two units is gradual. The homogenous, unstratified, slightly micaceous yellow silt, with numerous gastropod remains, covers the marshy sediments and represents the oldest loess, which is the parent material of the overlying 2 m thick palaeosol (Figs. 3 and 4). The palaeosol consists of a transitional horizon on the top, an AB-horizon in the middle and a thin C-horizon at the bottom. The AB-horizon is composed of red-brown silty clay and becomes darker with depth. The fossil soil level is strongly compacted, homogenous and unstratified. Below the AB-horizon, a 0.2 m thick horizon of carbonate accumu-

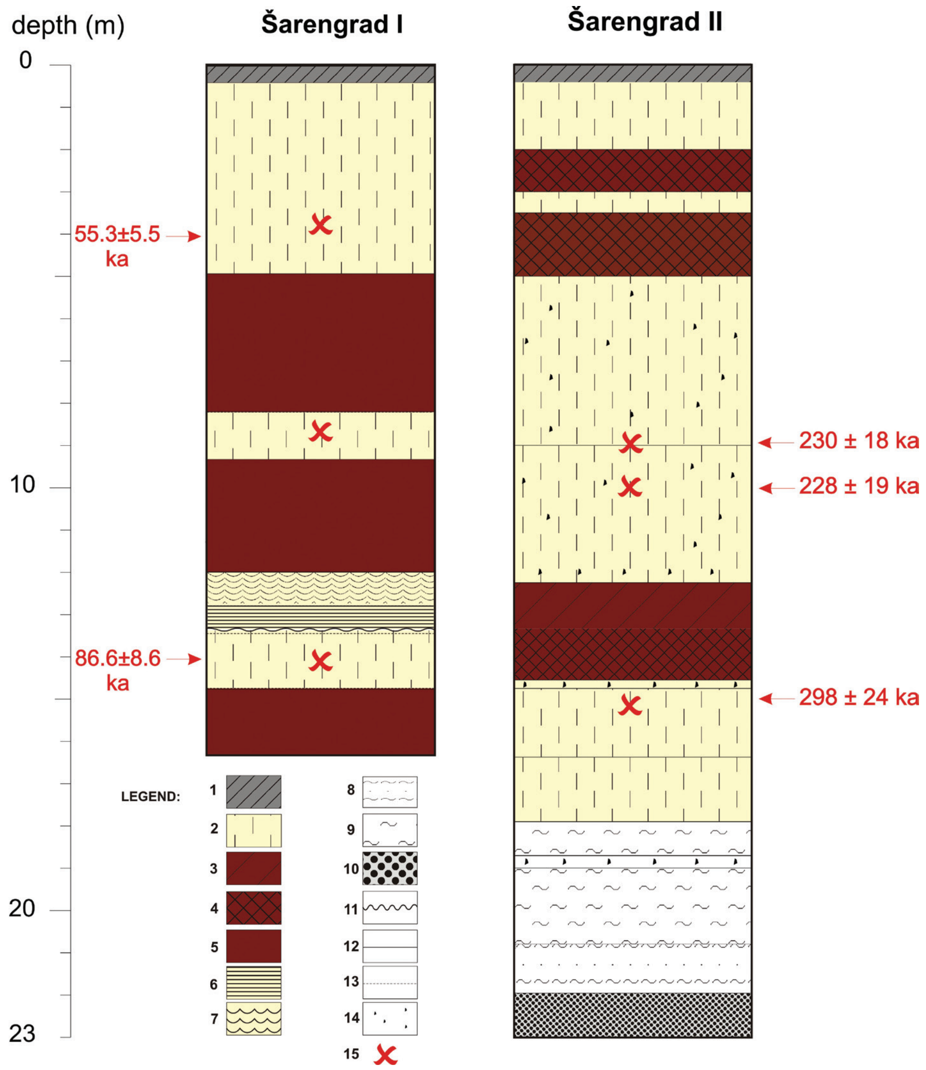

Figure 3: The simplified sketch of the loess-palaeosol sequence of Šarengrad I (GALOVIĆ et al., 2009) and the Šarengrad II section. Legend: 1. modern soil; 2. loess; 3. transitional layer; 4. AB-horizon; 5. palaeosol; 6. horizontally laminated sediment; 7. wave-ripple marks; 8. silty clay; 9. clay; 10. sand; 11. erosional border; 12. sharp border; 13. transitional border; 14. carbonate concretions; 15. mineralogy sample location.

lation and cementation has been recorded. This palaeosol is a chernozem-brown forest type fossil soil, and is covered by a loess horizon about 7 m thick. The loess is well-sorted, homogenous, unstratified, and pale-yellow to greyish-yellow with dark brown manganese dots and white calcareous veins and flakes. It consists of slightly- to medium-micaceous silt. The calcareous nodules are dispersed throughout the entire loess layer, occasionally forming layer-like enrichments. The nodules range in size from 1 to 10 cm. Above this thick loess horizon is a double fossil soil, containing two palaeosol subhorizons. It is overlain by a loess layer seen at the uppermost, inaccessible part of the section (Figs. 3 and 4).

3. METHODS AND SAMPLING

3.1. Luminescence dating

Luminescence dating is the method of choice for establishing a reliable geochronological framework for loess-palaeosol sequences. It is a radiation exposure dating method, based on measuring the cumulative effect of natural radioactivity to which minerals in the sediment were exposed during burial. Its basic principle is that the larger the number of trapped electrons or crystal damage in a grain, the longer the time of exposure to natural radiation, and hence the older the material being dated (AITKEN, 1998). Luminescence dating determines the time since mineral grains in the sediment were last exposed to direct sunlight, which removes the latent luminescence signal in minerals (WINTLE & HUNTLEY, 1979). Wind induced sediment transportation causes bleaching of the latent luminescence signal in the dosimeters. Quartz and feldspar are the two most dominant constituents of loess and both are used as dosimeters for dating. Feldspar has the advantage over quartz in that it saturates at higher doses (e.g. at ~ 2000 Gy; THIEL et al., 2011c), and hence is more appropriate for dating older sediments. However, feldspar suffers from anomalous fading (WINTLE, 1973; SPOONER, 1994), an athermal loss of signal which can cause age underestimation; a major disadvantage when

Figure 4: The Šarengrad II loess-palaeosol section at the town of Šarengred.

using feldspars as dosimeters. A common procedure for overcoming this problem is the application of fading corrections (HUNTLEY & LAMOTHE, 2001), after performing fading tests (AUCLAIR et al., 2003), or measuring more stable luminescence signals (THOMSEN et al., 2008). Hence, in recent years a lot of effort has been invested in studying the properties of more stable feldspar signals obtained at higher temperatures, and the fading properties of feldspar grains (THOMSEN et al., 2008; BUYLAERT et al., 2009, 2011, 2012; REIMANN et al., 2011; THIEL et al., 2011a, b, c, d; TSUKAMOTO et al., 2012). THOMSEN et al. (2008) showed that stimulation at elevated temperatures (IRSL at 110°C, 170°C and 225°C after an IR bleach at 50°C) reduces the fading rate and gives more stable signals. THIEL et al. (2011c) applied a measurement protocol which involved even higher temperatures, (a preheat at 320°C, followed by an IR stimulation at 290°C after an IR bleach at 50°C), showing that there is negligible fading. Hence, the elevated temperature post-IR IRSL measuring approach is widely accepted as appropriate for the dating of Middle Pleistocene deposits.

3.1.1. Sampling and sample preparation

Three loess samples were collected from the freshly cleaned loess-palaeosol section exposed in Šarengrad II for luminescence dating using metal cylinders. Sample Š1 was taken at a depth of 15m, Š2 at 10m and Š3 at 9m below present day surface. Laboratory treatments were performed under subdued red light. The material was treated with hydrochloric acid to remove the carbonates, sodium oxalate against coagulation and hydrogen peroxide to remove organic material. The 4-11 μm fraction was separated from the treated material following FRECHEN et al. (1996). The fine-grained aliquots were prepared by dispensing and settling the material onto aluminium discs using acetone.

3.1.2. Dose rate determination

The U, Th and K content were measured by high-resolution gamma spectrometry equipped with a high-purity germanium detector, in the laboratory at the Leibniz Institute for Applied Geophysics (LIAG) in Hannover, Germany. For the measurements, 700 g of dried and homogenised sample was placed in Marinelli-type beakers, sealed and stored for at least four weeks to re-establish the equilibrium between radon and its daughter nuclides prior to measurement. Radioactive equilibrium was assumed for the decay chain. Cosmic dose rates were corrected for the altitude and sediment thickness following PRESCOTT & HUTTON (1994). The alpha efficiency was estimated to be a mean value of 0.08 ± 0.02 (REES-JONES, 1995). The water content was assumed to be 15 ± 5% depending on depth (PÉCSI, 1990) to allow time dependant changes of the water content. An equipment dependant systematic error of 5% is included for the results of gamma counting.The conversion factors by GUERIN et al. (2011) were used for dose rate calculations. An error of 10%, which accounts for all uncertainties related to burial

Table 1: Dosimetry results. The total dose rate is the sum of alpha, beta, gamma and cosmic radiation.

Sample name	Sample ID (LUM No)	Depth below surface (m)	U (ppm)	Th (ppm)	K (%)	Cosmic dose rate (mGy/a)	Total doserate (mGy/a)
Š1	1661	15	2.79 ± 0.03	11.66 ± 0.07	1.34 ± 0.02	0.030 ± 0.003	3.18 ± 0.19
Š2	1662	10	3.04 ± 0.04	12.44 ± 0.11	1.61 ± 0.02	0.049 ± 0.005	3.59 ± 0.21
Š3	1663	9	2.94 ± 0.02	11.84 ± 0.06	1.53 ± 0.01	0.055 ± 0.006	3.43 ± 0.20

depth, is assumed for the cosmic dose. The uranium, thorium and potassium concentrations, as well as the cosmic doses and the calculated total dose rates, are given in Table 1.

3.1.3. Equivalent dose measurements and luminescence characteristics

Luminescence measurements were performed using an automated Risø TL/OSL-DA15 reader (BØTTER-JENSEN et al., 2000) equipped with a $^{90}Sr/^{90}Y$ β-source (delivering~ 0.094 Gy/s to fine-grain material on aluminium discs) at the LIAG in 2010. Infrared light emitting diodes emitting at 875 nm were used for luminescence stimulation, and the luminescence signals were detected in the blue-violet wavelength region (320 – 460 nm) using the Schott BG39/Corning7-59 filter combination.

The equivalent dose (D_e) is a measure of the past radiation and, when divided by the environmental dose rate, gives the age of the sediment.The D_e's were determined using a modified elevated temperature post-IR IRSL protocol as proposed by BUYLAERT et al. (2009; coarse grains) and THIEL et al. (2011a,b; polymineral fine grains). A similar protocol was applied on the Gorjanović loess section in Vukovar by WACHA & FRECHEN (2011). For the measurements a preheat of 250°C (10 s) was applied followed by IR stimulations at 50°C (IR_{50}) and 225°C ($pIRIR_{225}$). The response to the test dose (~ 50Gy) was measured in the same way. A high-temperature signal clean-out at the end of each cycle was not applied because it showed no significant difference during test measurements. The D_e's were calculated using the middle part of the decay curve (5-20 s) because previous studies show (e.g. WACHA & FRECHEN, 2011; NOVOTHNY et al., 2010; TSUKAMOTO et al., 2006) that this part of the decay curve showed a lower fading rate than the initial part. The last 50 s of the decay curve were used for background subtraction for the IR_{50}. For the $pIRIR_{225}$ the middle 20 s of the curve were used for background subtraction. The intensity of this part of the $pIRIR_{225}$ signal curve was similar to the background intensity of the IR_{50} signal and was therefore selected for subtraction. Dose response and decay curves of sample 1662 are presented in Figure 5. The signal intensity of the IR_{50} is significantly stronger than the $pIRIR_{225}$ signal (Fig. 5a). This could be the result of the dominance of Na-feldspars in the polymineral fraction as proposed by TSUKAMOTO et al. (2012). The same was also observed by WACHA & FRECHEN (2011) and THIEL et al. (2011a).

The protocol was tested by checking the recycling ratios, recuperation and by performing the dose recovery tests. The recycling ratios show that the measuring protocol can reproduce the response to a given laboratory dose after it has been repeatedly heated. Recycling ratios are 0.99 ± 0.04 (n=3, 1661) and 0.98 ± 0.04 (n=3, 1662) for both the IR_{50} and the $pIRIR_{225}$ signals. For the sample 1663 the recycling ratios are 0.95 ± 0.04 for the IR_{50} and 0.96 for the $pIRIR_{225}$ signal (n=3). Recuperation is below 1% for all samples and both signals showing that the signals were reset completely during measurement.

The dose recovery test shows the quality of successfully recovering a known laboratory dose (WALLINGA et al., 2000). For the dose recovery test; six aliquots (sample 1663) were bleached for 4 h in a Hönle SOL 2 solar simulator. Three aliquots were irradiated with a known dose similar to the natural dose, and then measured using the protocol described above. The remaining three aliquots were used to measure residual signals after bleaching. The dose recovery ratio for sample 1663 is 1.5 ± 0.2 and 1.2 ± 0.1 for the IR_{50} and the $pIRIR_{225}$ respectively, showing overestimation especially for the IR_{50} signal. Such poor dose recovery results have been reported in several studies (e.g. THIEL et al., 2011d; BUYLAERT et al., 2012), even though the results were in accordance with independent age control data. In the case of BUYLAERT et al. (2012), the dose recoveries showed underestimation for the IR_{50} signal. Furthermore, they compared the results of dose recoveries for the IR_{50} signals during a single IR stimulation SAR protocol and during a post-IR IRSL protocol. They concluded that the IR_{50} D_e values measured as part of the post-IR IRSL protocol could underestimate the IRSL signal of the single IR stimulation SAR protocol which uses lower preheat conditions. Furthermore, they concluded that a poor dose recovery ratio is not a good predictor of inaccurate D_e measurement and that the dose recovery should only be used as a qualitative indicator of the performance of SAR (BUYLAERT et al., 2012). For comparison, in this study, a dose recovery test was performed with a single IR stimulation SAR protocol while using various preheat temperatures (sample 1661). The dose recovery ratios were all ~ 1.0 which seems to be satisfactory (data not shown). Another reason for poor dose recovery results could be the size of the test dose applied for the measurements (~ 50 Gy) which might be too small. QIN & ZHOU (2012) showed that measured D_e-s decrease by ~ 6% with the test dose increasing from 12 Gy to 60 Gy, while an increase is observed when the test dose is further increased (measured for the $pIRIRSL_{295}$ signal). This could be the result of D_e overestimation due to a thermally transferred post-$IRIRSL_{295}$ signal and hence poor dose recoveries (QIN & ZHOU, 2012).

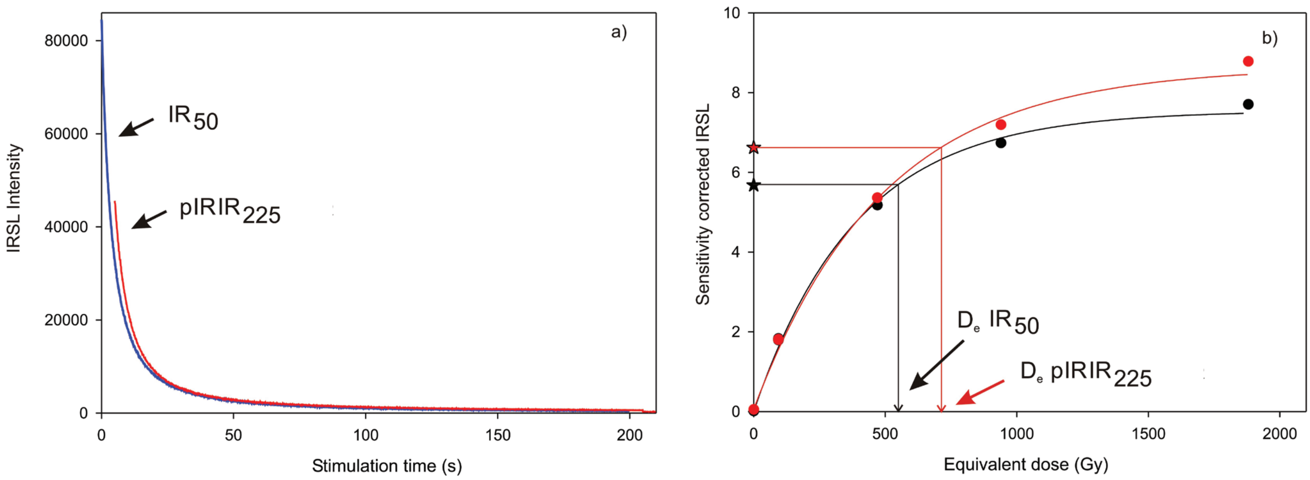

Figure 5: Luminescence characteristics of the measured IRSL signals of sample 1662. a) Signal intensity (decay curves) and b) dose response curves with indicated D_e values for both the IR_{50} and $pIRIR_{225}$ signals. Dose response curves were fitted using exponential and linear curve fitting.

Nevertheless, we acknowledge that we are unsure of the significance of our overestimated dose recovery results. SOHBATI et al. (2011) showed that residual doses appear to be dependant on the corresponding natural doses; the larger the natural dose, the larger the residual. They also showed that there may be a small, apparently unbleachable component present. Our measured residual doses (~ 2Gy for IR_{50} and ~ 6Gy for $pIRIR_{225}$) are negligible and do not influence the quality of the dose recoveries nor the D_e-s.

3.1.4. Fading tests and fading corrections

In this study fading tests and fading corrections were performed following AUCLAIR et al. (2003) and HUNTLEY & LAMOTHE (2001), respectively. According to HUNTLEY & LAMOTHE (2001), fading corrections are applicable only for the linear part of the growth curve i.e. for samples up to 50 ka. BUYLAERT et al. (2011) applied fading corrections on K-feldspars from a MIS5e site in Denmark for which independent age control was available and concluded that fading corrected IRSL ages using K-feldspar may be both precise and accurate over a greater age range than proposed by HUNTLEY & LAMOTHE (2001). In this study fading tests were performed for both IR_{50} and $pIRIR_{225}$ signals and fading corrections were applied for the IR_{50} signal. Fading tests were performed on the same aliquots as for the D_e measurements. The aliquots were irradiated with a beta-dose similar to the natural D_e, and then measured using the above mentioned protocol, but with inserted different storage times, up to ~10 hours, immediately after irradiation and preheat (AUCLAIR et al., 2003). The fading rates (g-values) were normalised to a measurement time delay of 2 days after irradiation. The mean g-values calculated from three aliquots per sample and their standard errors were used for fading corrections. The g-values are 5.0 ± 0.3, 3.9 ± 0.4 and 4.4 ± 0.3 for the IR_{50} and 1.4 ± 0.7, 0.3 ± 1.0 and 1.8 ± 0.7 for $pIRIR_{225}$ signals, for samples Š1, Š2 and Š3 respectively. The results of the fading tests are presented in Table 2.

3.2. Heavy and light mineral analysis of loess samples

The samples for the modal analyses of loess were collected at the same depths as the samples for luminescence dating. For correlation and comparison between the sections, three samples were also analysed from the Šarengrad I section. In addition to samples from the location for luminescence dating, one further sample was collected from the middle loess layer (Fig. 3).

To determine the qualitative and semi-quantitative mineral composition of heavy and light mineral associations, the samples were extracted after disaggregation in an ultrasound bath and sieved to the 0.09–0.16 mm size fraction, followed by dissolution of calcite with hydrochloric acid (5%). This fraction was selected for the analysis, because it includes all virtual mineral species in proportions representative of the bulk sample. The heavy mineral fraction (HMF) was separated from the light mineral fraction (LMF) using bromoform ($CHBr_3$) at a density of 2.85-2.88 gcm^{-3}. Canada balsam was used as the mounting medium, and the slides of heavy and light mineral fractions were examined in polarized light (MANGE & MAURER, 1992). Qualitative and semi-quantitative composition of each sample was established after the determination of 300–400 grains and the percentage of each mineral was calculated. The composition of transparent minerals was presented in ranges in the case of less than 1% of HMF per sample due to the lack of grains required for calculating the reliable semi-quantitative mineral composition.

4. RESULTS AND DISCUSSION

4.1. The chronology of the Middle Pleistocene loess section Šarengrad II

The dose rates are 3.18 ± 0.19, 3.59 ± 0.21 and 3.43 ± 0.20 mGy/a of samples Š1, Š2 and Š3 (Table 1), respectively, which is in accordance with other loess in Croatia (WACHA et al., 2011a, b; WACHA & FRECHEN, 2011; GALOVIĆ et al., 2009) and European loess (e.g. FRECHEN et al., 1997; NOVOTHNY et al., 2011; THIEL et al., 2011c, in press).

Table 2: Summary of the IR_{50} and $pIRIR_{225}$ results (3 aliquots per sample). Fading-corrected ages for the IR_{50} signal were calculated after HUNTLEY & LAMOTHE (2001).

Sample	Depth below surface (m)	De (Gy)		g-value (%/decade)		Uncorrected age (ka)		Fading corrected age (ka)
		IR_{50}	$pIRIR_{225}$	IR_{50}	$pIRIR_{225}$	IR_{50}	$pIRIR_{225}$	IR_{50}
Š1 (1661)	15	598.4 ± 48.8	946.5 ± 51.2	5.0 ± 0.3	1.4 ± 0.7	188 ± 19	298 ± 24	324 ± 36
Š2 (1662)	10	548.7 ± 29.6	816.9 ± 49.5	3.9 ± 0.4	0.3 ± 1.0	153 ± 12	228 ± 19	228 ± 21
Š3 (1663)	9	539.8 ± 42.3	788.6 ± 43.0	4.4 ± 0.3	1.8 ± 0.7	157 ± 15	230 ± 18	250 ± 26

The complete age dataset obtained is presented in Table 2. The D_e values are in stratigraphic order. The measured residuals were ~ 2Gy for the IR_{50} signal and ~ 6Gy for the $pIRIR_{225}$ signal which is negligible and was hence not subtracted from the D_e's and did not play an important role in the results of the dose recovery test. The somewhat high dose recovery ratios were not considered to be inadequate; therefore the D_e results were not discarded. The ratio of the sensitivity-corrected natural signal to the laboratory saturation level was checked. According to WINTLE & MURRAY (2006), reliable equivalent doses for quartz OSL can be obtained up to a dose value of $2D_0$ which corresponds to 86% saturation. The same principle was applied to evaluate the D_0 values of our dose response curves. The values of 0.79 ± 0.08, 0.76 ± 0.07 and 0.77 ± 0.10 for the IR_{50} signal curve and the values of 0.86 ± 0.04, 0.82 ± 0.05 and 0.81 ± 0.05 for the $pIRIR_{225}$ signal for samples Š1, Š2 and Š3, respectively, were calculated. Our data show that the D_e values obtained for the IR_{50} are less than $2D_0$, while the D_e values for the $pIRIR_{225}$ signal are a little higher than D_0 for Š1 sample, equal to the $2D_0$ for Š2 sample and less than $2D_0$ for Š3 sample indicating that the samples are very close to saturation, or in the case of sample Š1 at saturation.

The g-values are given in Table 2 where it can be seen that the highest fading rate measured for the IR_{50} signal is 5.0 ± 0.3%/decade (sample Š1). For the $pIRIR_{225}$ signal the highest value calculated was 1.8 ± 0.7 %/decade (sample Š3) showing that fading for the $pIRIR_{225}$ signal indeed is significantly lower than from the IR_{50} signal. These values are similar to other data from the area e.g. from the Gorjanović loess section in Vukovar (e.g. WACHA & FRECHEN, 2011) where the same protocols and conditions were used for data calculations. Furthermore, they calculated data using different integration limits to find the most stable part of the curve with the lowest fading. They noticed that the lowest fading can be seen in the middle part of the curve. Their observations were applied in this study. Fading corrections using the method by HUNTLEY & LAMOTHE (2001) were applied for the IR_{50} signal. It has been shown that $pIRIR_{225}$ signals show lower to negligible fading, and that measured g-values might not be representative for the fading rates over geological time (e.g. THIEL et al., 2011b). Even though we measured g-values of up to 2%/decade for our samples, we did not apply any fading correction, but acknowledge that this may result in an underestimate of the age. The results of the fading corrected IR_{50} and the $pIRIR_{225}$ are in very good agreement within the uncertainties (Table 2).

The first detailed investigation of loess exposures at the town of Šarengrad were presented by GALOVIĆ et al. (2009), where a geochronological framework of the Šarengrad I section was presented based on luminescence dating. The ages were calculated using the MAAD protocol, which in a way, is an outdated protocol. The IRSL ages of 55.3 ± 5.5 ka and 86.6 ± 8.6 ka can be correlated to the last glacial – last interglacial period, but it is probable that the ages show some underestimation due to the lack of fading corrections on the samples. More measurements using up-to-date measurement protocols are mandatory to re-evaluate the existing data. The presented chronology of the Šarengrad II section, which is in the vicinity of the Šarengrad I section (Fig 1c), is based on the $pIRIR_{225}$ data (Fig. 6) and is considered to represent minimum ages due to the measured g-values of around 2%/decade (Table 2) and the signal being close to saturation. Furthermore, the results were correlated, (in addition to the older Šarengrad I section), with other loess sections in the area (GALOVIĆ et al., 2009; WACHA & FRECHEN, 2011; THIEL et al., in press), which has allowed compilation of a chronostratigraphy of the area based on numerical dating.

At the bottom of the Šarengrad II section, sand and clay horizons are exposed (Fig. 3, 4, 6). These deposits were already registered as being widespread in this area in the base of loess by GORJANOVIĆ KRAMBERGER (1922), who conducted the first detailed investigation on loess deposits covering the sand. Furthermore, he made a detailed investigation of the steep outcrop at the bank of the Danube at Šarengrad, where he described a 2.5 m thick loess package covering yellow and grey sands and sandstones. Based on *Viviparidae* remains observed in the sands, he assumed that the sediment was deposited as early as the Late Pliocene or later (GORJANOVIĆ KRAMBERGER, 1922). POJE (1986) produced a detailed palaeoclimatic reconstruction of the Vukovar loess plateau based on detailed malacological investigations. In her study, among others, a loess section at Šarengrad (the exact location was not indicated),was described consisting of three loess layers intercalated with two palaeosols. The mollusc assemblages investigated there indicated cold climatic conditions; comparison with mollusc data and radiocarbon ages from carbonate concretions found in loess at the section at Vukovar (GALOVIĆ & MUTIĆ, 1984) led to correlation with the Last Glacial (POJE, 1986). HUPUCZI et al. (2010)

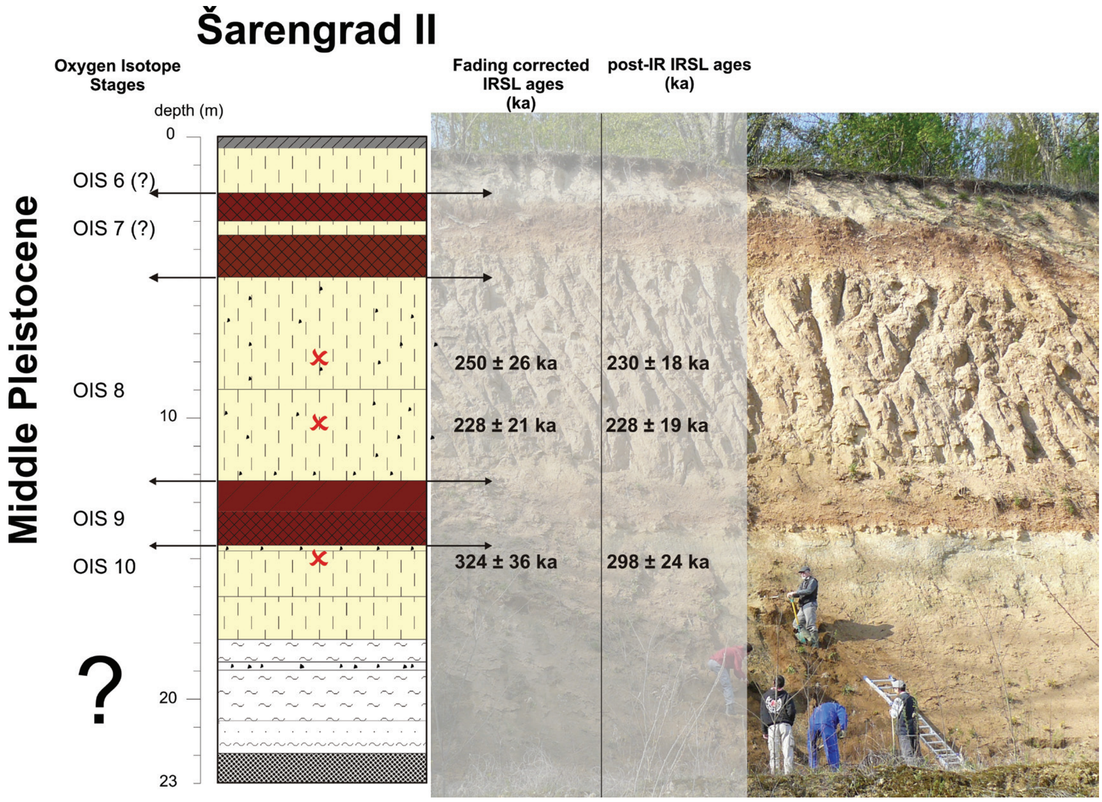

Figure 6: Luminescence dating results and interpretation of the data from the Šarengrad II section. Fading corrected IR_{50} and $pIRIR_{225}$ results are presented for comparison as well as the correlation to oxygen isotope stages (LITT et al., 2008). The lowermost sample (Š1) shows signals which are very close to saturation and should hence be considered as minimum age.

performed malacological investigations at the Šarengrad II section, where they separated five malacological horizons. Based on their results and the correlation with data from the Šarengrad I section (GALOVIĆ et al., 2009), they assumed a Last Glacial-Interglacial age for the section. They also provided palaeoenvironmental reconstructions based on their mollusc assemblages. For the sand and clay layers from the bottom of the Šarengrad II section, they recognized a freshwater environment with warm climatic conditions (HUPUCZI et al., 2010), which grades into a fluvial setting and was eventually covered by loess representing cold climatic conditions. Based on the results of IRSL dating applied in this study, the loess package covering the sand and clay gave a $pIRIR_{225}$ age estimate of 298 ± 24 ka which (if the fading corrected IR_{50} age estimate of 324 ± 36 ka is considered), correlates well with the end of the oxygen isotope stage (OIS) 10 and the beginning of OIS9 (e.g. LITT et al., 2008). The upper loess package showed $pIRIR_{225}$ ages of 228 ± 19 ka and 230 ± 18 ka; this correlates with OIS8. This loess package is covered by a double palaeosol and another loess layer. Based on our results we conclude that the double palaeosol correlates to OIS7, but more dating is necessary for providing further evidence for such a statement.

Similar double palaeosols were described in the Vojvodina region and in Hungary. THIEL et al. (in press) applied the post-IR IRSL to the Paks loess section in Hungary. In the upper part of the section, a soil complex is exposed, (the so called BD_1-BD_2 complex – see THIEL et al., in press). The luminescence ages from above this soil complex pointed to OIS6, and thus the BD soil complex correlates to OIS7. The loess below the soil complex can be correlated to OIS8 based on the data presented by THIEL et al. (in press). The same interpretation, based on magnetic susceptibility measurements and a detailed lithostratigraphy, but unfortunately lacking numerical age estimates, from the Udvari-2A borehole from Hungary was presented by KOLOSZAR & MARSI (2010) and KOLOSZAR (2010). MARKOVIĆ et al. (2011) correlated the double soil complex (the V-S2 soil from the Serbian stratigraphy; the BD_1-BD_2 soil complex from Hungary), with the double peak of OIS7. Recently, dating of the Middle Pleistocene loess from Stari Slankamen was performed (MURRAY et al., in press), using an elevated temperature post-IR IRSL dating protocol. They suggested that the loess unit V-L2 accumulated during OIS6 and that the V-S1 pedocomplex can be correlated with the complete OIS5 stage. They also proposed minimum ages of ~230 ka for the loess unit V-L3 (OIS8) and palaeosols V-S3, V-S4 and V-S5 exposed at Stari Slankamen. This interpretation is supported by magnetostratigraphic and aminostratigraphic evidence (MARKOVIĆ et al., 2011). These observations are in very good agreement with our study; however, more sampling of the uppermost loess horizon is necessary to clarify whether both soils belong to OIS 7. We conclude that OIS7 is represented by a double palaeosol complex in the wider region (the Pannonian basin).

Table 3: Modal composition of the heavy and light mineral fractions (0.09–0.16 mm) of loess horizons from the Šarengrad I and II sections. LMF – light mineral fraction, q – quartz, m – muscovite, f – feldspar, lf – lithic fragments; HMF – heavy mineral fraction, op – opaque minerals, ch – chlorite; b – biotite, THM – transparent heavy minerals; ep-zt – epidote – zoisite, am – amphibole, py – pyroxene, g – garnet, ky – kyanite, st – staurolite, tu – tourmaline, zr – zircon, rt – rutile, ti – titanite, ap – apatite, chr – chromite.

SAMPLE	LMF				% of HMF	HMF				Transparent heavy minerals											
	q	m	f	lf		op	ch	b	THM	ep zt	am	py	g	ky	st	tu	zr	rt	ti	ap	chr
Šarengrad I																					
Š-1	21	72	2	4	5	13	43	16	28	26	16	5	34	1	2	2	3	3	5	1	1
Š-12	35	61	2	1	3	15	35	15	35	25	9	2	33	5	4	2	3	6	5	0	6
Š-22	47	40	11	2	6	51	24	3	23	18	14	3	33	3	5	3	2	1	7	3	7
Šarengrad II																					
Š1	58	27	8	7	5	25	9	6	65	26	12	9	32	1	4	2	2	5	3	0	5
Š2	52	30	14	4	5	17	7	2	67	22	2	6	43	2	3	7	2	9	2	3	0
Š3	76	2	18	3	< 1	14	1	1	61	22	4	9	44	1	3	3	2	9	1	1	0

Our results suggest the conclusion that loess accumulation in this area started during the Antepenultimate Glacial, or even earlier. Based on the data from GALOVIĆ et al. (2009), it can be concluded that it was also deposited during the Last Glacial, although these data should be interpreted with caution. The data of the Šarengrad I section should be re-evaluated using modern dating techniques to confirm such premises. In the investigated area, loess deposition was interrupted by pedogenesis at least six times. Based on the development stages of the palaeosol types and their chronology, Šarengrad I and II cannot be correlated. Nevertheless, the two lowermost palaeosols at Šarengrad I (the hydromorphic and the well-developed argillic dark brown palaeosol with rubification – GALOVIĆ et al., 2009), indicate at least two intensive and relatively longer periods of warmer climate. Various palaeosols exposed at the studied sections are related to interglacial and/or interstadial conditions and provide evidence of abrupt climate changes.

4.2. Mineralogical data and the provenance of the material

The bulk mineral composition of TMF of all investigated samples is typical for loess-palaeosol sequences of the area (MUTIĆ 1993; BANAK et al., 2012; THAMÓ-BOZSÓ et al., in press).There is a significant difference in the light mineral fraction between the studied sections. The dominant constituent of the light mineral fraction of the Šarengrad II samples is quartz (21 – 76%) while in the Šarengrad I samples muscovite (2 - 72%) predominates; followed by feldspars (<18%). Lithic fragments are present in all samples (< 7%). The amount of heavy minerals ranges from < 1% in sample Š3 to 6% in sample Š-22. Among them the transparent heavy minerals predominate (23–67%) over opaque minerals (13–51%). Biotite and chlorite are present in all samples, but they are more abundant in the samples from the Šarengrad I section. The most abundant transparent heavy minerals are resistant garnet grains (32 – 44%) followed by epidote (18–26%). Less abundant are amphiboles (2 – 16%). Pyroxenes, kyanite, staurolite, tourmaline, zircon, rutile and titanite occur regularly. Chromite is present in all samples except Š2 and Š3 from the Šarengrad II section. The results of the modal analysis are presented in Table 3.

THAMÓ-BOZSÓ et al. (in press, fig. 4) showed that NNW winds could supply the wider Transdanubian area, and hence eastern Croatia, with aeolian material of such mineral composition. The chlorite- and/or biotite-rich heavy mineral association of loess from South-Transdanubia is similar to the composition of recent fluvial sediments of the Danube and other Transdanubian rivers (THAMÓ-BOZSÓ et al., in press), and they could also be the source area for (re)sedimentation. Alternatively, the base of the Šarengrad II sectionis composed of fine-grained micaceous fluvial sand (see Section 4 above; Figs. 3, 4), which probably extends over a wider area, and could also be the local source of the material. Chlorite and biotite are flaky minerals, together with muscovite which is the dominant mineral in the samples from the Šarengrad I section, and their behaviour is very similar during transport (alluvial or aeolian). Due to the higher mobility of flaky minerals, (greater than that of the isometric or elongated ones), it is very likely that they were transported farther and from a more distal source (THAMÓ-BOZSÓ et al. (in press). Domination of flaky minerals is observed in several horizons in the Zmajevac, Erdut and Šarengrad I sections which correlate to the Last Glacial period (GALOVIĆ et al., 2009) and in fluvial micaceous fine grained sand in the base of loess in the area (macroscopically determined at Šarengrad II and the Ilok sandpit).

The predominance of garnet and epidote indicate the Danube flood plain region to be the main source of the material (THAMÓ-BOZSÓ & KOVÁCS, 2007), and loess from Hungary (THAMÓ-BOZSÓ et al., in press). The amphiboles, could also originate from the nearby Slavonian Mountains and/or from the Dinaride Ophiolite Zone in Bosnia in addition to the Danube flood plain (PAMIĆ et al., 2002; ROBERTSON et al., 2009). Chromite is present in all samples except Š2 and Š3 from the Šarengrad II section and indicates an ultramafic magmatic source, most probably the Dinaride Ophiolite Zone, eroded and transported by the Vrbas and Bosnia Rivers in the south to the Pannonian basin in the north. This is supported by simulations of palaeowind patterns in the Carpathian Basin during the Last Glacial Period proposed by ÚJVÁRI et al. (2010), showing west-southwesterly (WSW) wind directions. In general, such a heavy mineral association as presented here is similar to the Danube flood plain sediments (THAMÓ-BOZSÓ & KOVÁCS, 2007) and loess from Hungary (THAMÓ-BOZSÓ et al., in press), but the southern edge of the Carpathian basin was probably influenced by the nearby Dinaride Ophiolite Zone which makes a slight contribution to the difference in the mineral composition.

5. CONCLUSION

At the Šarengrad II section, an excellent transition from sediments deposited in a waterlain marshy environment, to sediments deposited under subaerial conditions is exposed. An elevated temperature post-IR IRSL protocol was applied on polymineral fine-grains separated from loess from the Šarengrad II section for equivalent dose (D_e) estimation. This method proved to be applicable for our samples. Our interpretation is based on the $pIRIR_{225}$ signal, even though the doses were near saturation and fading around 2%/decade was measured for the samples.The dating results from the Šarengrad II section indicate ages much older than the Last Glacial-Last Interglacial cycle, discarding previous interpretations; clearly, the section is of Middle Pleistocene age. The lowermost loess can be correlated with oxygen isotope stage (OIS) 9-10, and the lowermost palaeosol can be correlated to OIS9. Due to the measured fading of the $pIRIR_{225}$ signal, an even older age cannot be entirely excluded. The middle loess section can be correlated to OIS8 and the double palaeosol to OIS7. Similar Penultimate Interglacial double palaeosols have commonly been described in the European loess stratigraphy (the BD_1-BD_2 palaeosol from Hungary and the V-S2 from Serbia). The Šarengrad II succession, together with the nearby Šarengrad I section, reflect environmental changes since at least OIS 10 and can be correlated to the last three glacial-interglacial cycles. The Penultimate and Last Glacial loess exposed at Šarengrad is intercalated with at least six palaeosols. Our study is the first to date older (antepenultimate glacial) loess in Croatia.

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