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Initial results of the colorimetric indices of the oldest exposed pedocomplex (Titel loess plateau, Serbia)

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Abstract

In this study we present an in-depth description of the colorimetric values for the lowest section of the Dukatar Loess Palaeosol Sequence (LPS) pedocomplex S5. Formed during the Marine Isotope Stage (MIS) 13-15, it represents the oldest pedocomplex exposed at the base of the Titel loess plateau (TLP), near the confluence of the Tisa and Danube rivers in Vojvodina (northern Serbia). The results of low-field magnetic susceptibility measurements (χ_{lf}) were compared to colour properties (obtained by conventional methods as well as instrumental measuring) and quantified Soil Development Indices (SDI). Of these measurements we found that the Redness Index (*RI1)* yielded the most useful results, as this index appears most sensitive to lithological changes and soil development intensity. It was also observed that a high level of correlation existed between χ_{lf}, and *a** chromaticity. The initial results of this study highlight the utility of colorimetric methods as an interdisciplinary tool when evaluating the presence of ferromagnetics, and the application of rock magnetism to the Middle and Upper Pleistocene LPS of the Middle Danube Basin. The presented approach can be used to observe the evolution of climatic and ecological conditions in the given study area, and for establishing correlations between sites extending over the Eurasian LPS provinces.

1. INTRODUCTION

Soil pedocomplexes are unique, highly sensitive natural multi-component systems capable of forming valuable archives for numerous signals that are indicative of intense palaeoclimatic and palaeoenvironmental changes over time (BRONGER et al., 1998; BRONGER, 2003). Soil memory preserves diverse components which vary due to the type of evolutionary processes that have taken place during the time since deposition. Therefore, combining studies of these complex physical and geo-chemical components allows detailed reconstruction of geoecological trends that drive the naturally induced soil transformation over different spatio-temporal scales (e.g. VASILJEVIĆ et al., 2011; 2014). Unlike modern soils, palaeosols are fossil relics which have undergone pedogenic processes in the geological past, capturing palaeoclimate components of ancient landscapes (NETTLETON et al., 2000). The Danube watershed encompasses complex geological terrains comprised of diverse geomorphic units, such as the Alps, Bohemian Massif, Carpathians, Dinarides, and the Balkan Mountains. The catchment has been a valuable source of thick loess deposits for study, which are especially well preserved within the Middle Danube basin, with sections dating back until at least the mid-Quaternary (e.g. FENN et al., 2022; WACHA et al., 2021; BANAK et al., 2016; GALOVIĆ & PEH, 2016; SÜMEGI et al., 2019; MARKOVIĆ et al., 2013). Therefore, Danubian river catchment loess deposits preserve some of the longest terrestrial records of palaeoenvironmental and palaeoclimatic changes in Central and Southeastern Europe, and possess great palaeoecological significance (e.g. RUBINIĆ et al., 2018; GALOVIĆ, 2014, 2016; BANAK et al., 2013; MARKOVIĆ et al., 2015). In Serbia, loess-palaeosol sequences (LPS) have been documented predominantly in the northern part of the country (MARKOVIĆ et al., 2006; MARKOVIĆ et al., 2008, MARKOVIĆ et al., 2015; and references therein), but with a few exceptions occurring in southern parts of the Carpathian basin (BASARIN et al., 2014; OBREHT et al., 2014; BOESKEN et al., 2017; MARKOVIĆ et al., 2021b). Palaeosols developed on loess can be traced over extensive parts of Europe and Asia (KUKLA, 1987), and it is possible to correlate their chronostratigraphy based on different proxies and thus reconstruct pan-continental climate fluctuations. The most common proxies encompass: magnetic properties of soils and loess (RADAKOVIĆ et al., 2019), grain size or grain size indices (VANDENBERGHE, 2013), luminescence dating (BERGER et al., 1992; PERIĆ et al., 2019), stable isotopes (SCHATZ et al., 2011), chemical analysis (POTTER et al., 2021;

FENN et al., 2022), tephrochronology (TIMAR-GABOR et al., 2017), palynology (ZHANG et al., 2017), malacology (LUDWIG et al., 2021), and colour indices (SPRAFKE et al., 2020). VASILJEVIĆ et al. (2014) asserts that the aforementioned features of this widely spread sediment and pedocomplexes have proven to be of the utmost scientific, archaeological and agricultural significance within the Eurasian loess belt. This study focuses on the colour indices proxy.

Different colours of LPS were recorded by the pioneering loess researcher, Aloisius Ferdinando Marsigli in his "*Danubius Pannonico Mysicus*" (MARSIGLI, 1726). One and a half centuries later, the geologist Aleksandar Popovics (1847-1877) wrote about the transition of yellow colour of the loess sediments on the Fruška Gora Mountain in Serbia. This study recorded the appearance of a red colour in loess, which is today known as a feature of the regions palaeosols (POPOVICS, 1876).

The main topic of our study is the colour of the Dukatar LPS, which was described primarily by JELENA MARKOVIĆ-MARJANOVIĆ (1950), who noted the strong red colour visible at the bottom of the profile. In Danubian loess stratigraphy, this layer is referred to as S5; where "S" stands for soil and 5 means that it was formed in the fifth interglacial period, counting from the present (MARKOVIĆ et al., 2015). Before the above-mentioned proxies were applied to loess research, the colour intensity was used to make connections between multiple loess profiles in Serbia and allude to certain aspects of loess plateau geomorphological evolution. Counting of the loess (yellow) or palaeosol (red) layers was utilised in around 70 papers in the former Yugoslavia in an attempt to correlate loess profiles in Serbia. As time passed, the more detailed the descriptions became, and the more differentiated the LPS became (MARKOVIĆ-MARJANOVIĆ, 1965, and references therein).

Presently, many LPS in Serbia still do not have determined colorimetric perspectives. The work of LUKIĆ et al. (2014) was the first detailed colour investigation, describing the Orlovat LPS (Fig. 1), in the Banat region of Serbia. A strong correlation between frequency-dependent magnetic susceptibility and Redness Index was discovered. The Orlovat LPS spans the last glacial cycle, with the lowest palaeosol belonging to Marine Isotope Stage (MIS) 5, or S1 in Danube loess stratigraphy. The second LPS in Serbia to be characterized in terms of its colorimetric properties was the profile at Stalać (OBREHT et al., 2016). This archive is a valuable record of the transition from the Mediterranean to continental climate in the central Balkan region, which occurred between MIS 9 and MIS 1. The most recent profile to be described in terms of its colour proxies is the Zemun LPS, on the right bank of the Danube (LAAG et al., 2021). The respective authors expanded the search to MIS 11, or palaeosol S4. In general, it has been found that the luminance of the LPS sediments follows glacial cycles; although in Zemun the S4, which is the palest of all palaeosols, the signal is not as distinguishable as in other palaeosols. Fortunately, there are other parameters of colour such as redness and blueness, with indices derived from them, that assist our interpretations of the LPS, and pedogenetic processes (ZYKINA et al., 2021). Interestingly, coloured soils with shades of red and brown are already recognized as touristic attractions, for example in Chamarel, Mauritius (SHETH, et al., 2010). High-resolution colour measurements provide detailed palaeoenvironmental markers, which can even be linked to the migration of anatomically modern humans (SPRAFKE et al., 2020).

This study aims to characterize the oldest pedocomplex of the Titel Loess Plateau, in terms of its magnetic and colorimetric properties, and the palaeoecological features that outline the scientific and educational values of the given LPS. These sites are somewhat endangered by various factors, as presently they remain for the most part unprotected.

2. MATERIAL AND METHODS

2.1. Study area

The Dukatar site (45°16'59" N, 20°14'33" E) is located 500 m downstream from the settlement of the same name and represents the most complete series of loess-palaeosol sequences of the TLP (Fig. 1). It is situated at the foot of a loess section and is available for sampling only during the low water level of the Tisa River. The sampled S5 pedocomplex part of the Dukatar sequence, is approximately 2.7 m thick and was formed during the Early Middle Pleistocene (e.g. MARKOVIĆ et al., 2012). This site belongs to the highest protected zone of the Special Nature Reserve TLP, as indicated with a thick red line in Fig. 1 (SGRS, 2012). The site is situated in the contact zone of the Tisa River and TLP. After building the dam at Novi Bečej, north of the TLP, the Tisa River has a controlled regime. When the water level is high (April-May), it is not possible to reach the Dukatar LPS, while in September and October it is accessible. The maximum recorded amplitude of the Tisa river at the TLP is 876 cm (PAVIĆ, 2006). In the north of Dukatar there are two famous LPS: Veliki Surduk (Big Gully) (PERIĆ et al., 2019) and Feudvar (NAMIER et al., 2021). In the south, three more exist: Rogulić Surduk, Stara ciglana (Old brickyard) and Kalvarija. The list of protected LPS in Serbia is quite short: Surduk (VUJIČIĆ et al., 2011), Batajnica (VIŠNJIĆ et al., 2016), Stari Slankamen (VASILJEVIĆ et al., 2011), and Zemun (ŠARIĆ, 2008).

2.2. Sampling strategy

A field survey was undertaken prior to the onset of sampling the selected profiles for palaeoclimatic and palaeoecological reconstruction using colorimetric parameters. The field survey, carried out in 2013, involved a detailed description of LPS, only then were samples collected for laboratory analysis. Efforts were complicated by erosion and rockfall over the surface of the profile, resulting in colluvial deposits of considerable thickness; this greatly affected the consistency of the laboratory results. Initial steps included the removal of colluvial deposits and recent vegetation in order to expose the undisturbed surface of the loess section. The next step involved cutting a vertical trench and sampling of the profile. On the TLP, at the site located near the Dukatar settlement, a strongly rubified S5 pedocomplex was exposed and sampled. Sampling of the S5 pedocomplex on the Dukatar profile was undertaken at a high resolution of 3 cm from the bottom part of the L5 unit, across the upper part of the A horizon, on into the Ck horizon in the basal part of this strongly rubified palaeosol. A total of 100 bulk samples were taken for palaeopedological research (Fig. 2A). In addition, during sampling of this pedocomplex particular care was taken to avoid contact with widespread carbonate concretions in order to obtain valid results of the laboratory analysis.

2.3. Magnetic and colorimetric measurements

Measurement of the χ_{lf} of palaeosol samples from the Dukatar profile were undertaken at the laboratory of the Loess Research Group at the Department of Geography, Tourism and Hotel Management, Faculty of Sciences, University of Novi Sad, using a portable Bartington susceptibility meter set to 300 Hz. Determin-

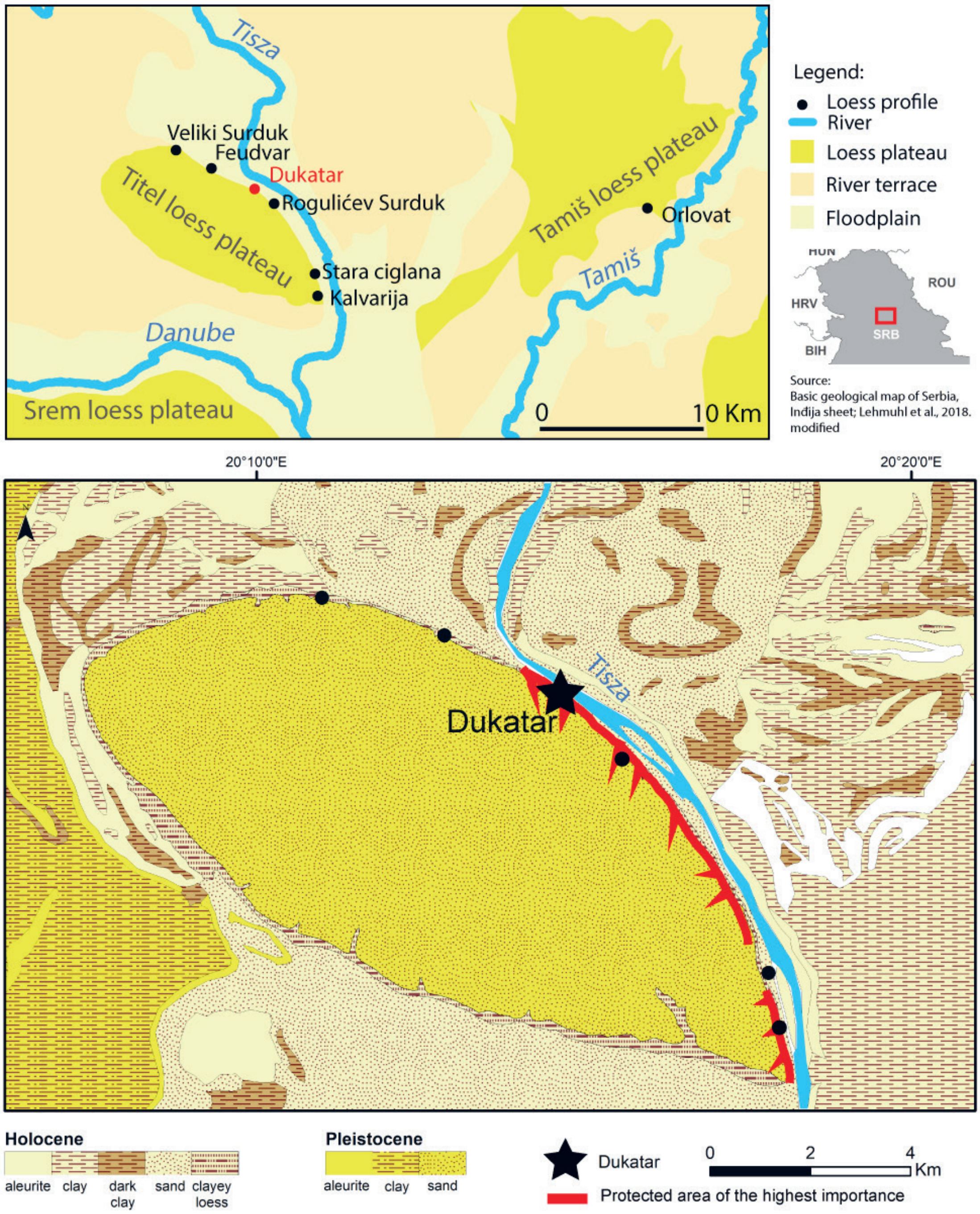

Figure 1. The upper panel shows the wider region of northern Serbia and the higher resolution part covers the Tisa and Danube confluence, with the loess palaeosol sequences over the TLP; the lower panel shows the geological map and protected area of the highest importance indicated with a red line.

ing the colour of samples in wet conditions, drying of the samples, and re-examination of the colour in dry conditions was carried out during March and April 2014 at the Laboratory for Paleoecological Reconstruction (LAPER), Department of Biology and Ecology, Faculty of Sciences, University of Novi Sad. Following the approach of LUKIĆ et al. (2014), the samples were dried in the oven for at least 12 h at 39 °C. Instrumental measurement of loess-palaeosol sample colour involved the use of a tristimulus colorimeter, and expressing the obtained values in the CIE *L a* b** colorimetric system (Hünter Lab, 2001). This was undertaken at the Department of Food Preservation Technology, Faculty of Technology, University of Novi Sad. The calculation of Rubification (*RI*) and Melanization (*MI*) Indices was performed using the following the approaches provided by HARDEN (1982), HARDEN and TAYLOR (1983), VIDIĆ et al. (2004) and VIDIĆ and LOBNIK (1997). This methodology was based on the Lotus Template by TAYLOR (1988), as it possesses robust approaches for quantification procedures and index calculations. The intensity of rubification indicates the formation of haematite crystals suspended in the soil matrix, which are responsible for the distinctive bright red colour of the soil. The *MI* indicates accumulation of humus and humic substances in the soil, and the *RI* quantifies the degree of soil reddening relative to the parent material. *MI* is used to calculate the degree of darkening (VIDIĆ et al., 2004, SCHAETZL, and ANDERSON, 2005).

The Redness Index (*RI1*), proposed by HARDEN (1982), can be seen as a proxy for semi-quantitative soil reddening, and is indicative of the proportion of haematite in the pedocomplex. The

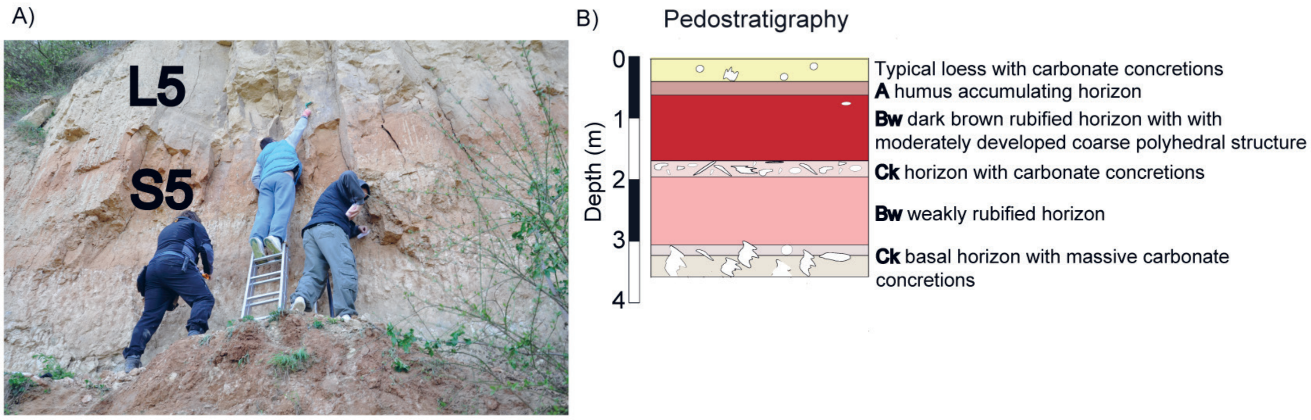

Figure 2. A) The picture of Dukatar LPS (photo by Lukić, T., September 2014) with indicated lithostratigraphy; B) Pedostratigraphy of Dukatar LPS.

Redness Rating Index (*RR*) translates the increase in redness between a soil or palaeosol and its parent material into a numerical value by comparing the changes in hue and chroma (BUGGLE et al., 2014). Based on the obtained parameters, *RI1* (BARRON & TORRENT, 1986; ROSSEL et al., 2006) (Equation 1) and *RR* were calculated (TORRENT et al., 1983), (Equation 2):

$$RI1 = \frac{L(a^{*2} + b^{*2})^{0.5} \times 10^{8.2}}{b^{*} \times L^{6}}, \qquad (1)$$

$$RR = \frac{(10 - H) \times C}{V}, \qquad (2)$$

where *L* stands for lightness, *a** for green-red colour, *b** for blue-yellow colour calculated by Konica Minolta Chroma meter CR-400, *H* for hue, *C* for chroma, and *V* for value in the Munsell colour chart. As previously mentioned, the colour was determined in both dry and wet conditions. Their difference was used in the calculation, following the approach by Buggle et al. (2014). The indices described above belong to the group of "true" index values, presented by the non-dimensional number.

3. RESULTS

3.1. Pedostratigraphy and Chronostratigraphy

The Dukatar profile is a section where the oldest sediments on the TLP were discovered and which has a thickness of 10 m (e.g. MARKOVIĆ et al., 2012, 2015). Both the oldest loess unit (L5) and the oldest exposed pedocomplex (S5) of TLP are observed within this site. For the purposes of this study, the lowest part of the L5 loess unit and the S5 pedocomplex, with a total thickness of approximately 2.7 m, were sampled. The sampled sequence (300 cm thick) extends from the contact between the upper part of the humus accumulating horizon A (~ 20 cm thick) and the basal part of the L5 loess unit. Beneath the A horizon there is a well-developed dark brown rubified cambic Bw horizon, with moderately developed coarse polyhedral structure that is approximately 110 cm thick (Fig. 2B). In the central part of the pedocomplex, S5 is a Ck horizon containing many carbonate concretions, and bioturbation caused by roots that are now preserved as rhizoliths. The lower Bw is a weakly rubified horizon disturbed by many hydromorphic features. In the bottom part of the pedocomplex S5 there is a horizon containing massive carbonate con-

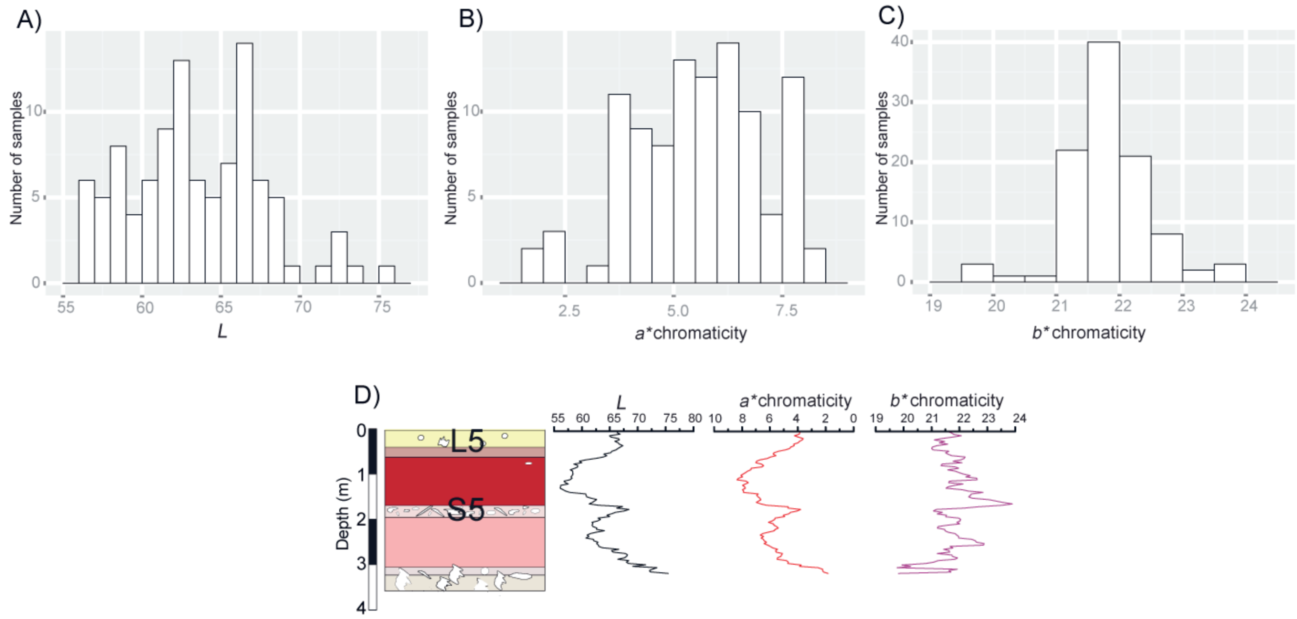

Figure 3. Histograms indicating A) *L*; B) *a** chromaticity; C) *b** chromaticity; D) The same parameters plotted on the depth scale of Dukatar LPS.

cretions overlying loessoid material of alluvial origin (also with numerous hydromorphic forms). The description presented here corresponds well to the previous palaeopedological interpretations put forward by BRONGER, (1976, 2003), MARKOVIĆ et al., 2012, 2015; BASARIN et al., 2014). Chronostratigraphic models of the LPSs in Serbia, presented by MARKOVIĆ et al. (2012, 2015) and BASARIN et al. (2014), provide correlation between the pedocomplex S5 at the Dukatar profile (TLP) with the accepted MIS 13-15 S5. MARKOVIĆ et al. (2015) indicated a remarkable accordance between Danubian and Chinese loess records, thus opening up the possibility for a transcontinental correlation of European, Central Asian and Chinese LPSs, using a standardised nomenclature and chronostratigraphic model.

3.2. Colorimetric and magnetic interpretation

The histograms in Fig. 3 illustrates the distribution of results for the colorimetric measurements *L*, *a**, and *b** parameters from the Dukatar profile (S5). Based on instrumental colour measurements, marked variations in *L* colorimetric values were observed. These variations displayed the following ranges: between the bottom part of the L5 unit, and the upper part of the A horizon within the S5 pedocomplex (64-66 units), and the bottom part of the Bw horizon, and its contact with Ck basal horizon (66-75 units) (Fig. 3A and Fig. 3D). The lowest values (56) are associated with the Bw dark brown horizon, at 150 cm depth. Variations are particularly noticeable between 180 and 195 cm from the upper part of the examined section (values vary between 61 and 68 units) and result from the presence of large diameter carbonate concretions that are characteristic of this part of the pedocomplex. Again, the values of *L* increase towards the calcified Ck horizon in the basal part of the S5 pedocomplex.

The values of *a** chromaticity (Fig. 3B and Fig. 3D) display variation that correspond to the detected variations in *L* colorimetric values. Note that the *a** values are mirrored to symmetrically match the *L*. They range from 3.4 units to 8.2 units at a depth of 15 cm to 120 cm. Subsequently, the values gradually decline to a depth of 192 cm, where the lowest value (3.6) was recorded. The *a** chromaticity gradually increases to the value of 6.5 units at a depth of 252 cm, and then begins to continuously decline to a depth of 270 cm (2.4), which is the lowest value in the pedocomplex. The highest measured *a** values are 8.2 and were recorded in the upper part of the examined section, in the central part of the Bw rubified horizon.

Observations of *b** chromaticity (Fig. 3C and Fig. 3D) display fluctuating values that vary slightly starting from the top to the middle of the examined profile. These values range from 19.5 to 23.9 units. In the transitional part between the L5 unit, and the upper part of the A horizon within the S5 pedocomplex the values rise from 21.6 to 19.8 units (between the bottom part of the Bw horizon and its contact with the Ck basal horizon). As noted in the previously mentioned colorimetric parameters values, stronger fluctuations in *b** chromaticity values are observed in the central part of the S5 pedocomplex. In general, these values gradually increase from the upper part of the pedocomplex to 171 cm, ranging from 21.5 units to 23.9 units, respectively. After this, *b** chromaticity values start to gradually decrease towards the value of 21.2 units observed at a Ck horizon, at the depth of 192 cm. The *b** chromaticity values from this point display continuous fluctuations that are most likely to be the result of the presence of hydromorphic features and carbonate concretions that are common at the basal part of the Bw horizon and its contact with the Ck horizon. The depth plot of these parameters is presented in Fig. 3D.

Results of the χ_{lf} measurements indicate that χ_{lf} variations are in good agreement with the pedostratigraphy (Fig. 4A). The histogram Fig. 4B illustrates the results of χ_{lf} for the Dukatar profile (S5). Values of χ_{lf} range from 40×10^{-8} $m^3 kg^{-1}$ in the upper part up to 132×10^{-8} $m^3 kg^{-1}$ at a depth of 114 cm. Thereafter, the values gradually decline to 43×10^{-8} $m^3 kg^{-1}$ at a depth of 171 cm, after which they begin to progressively increase to the value of 93×10^{-8} $m^3 kg^{-1}$, observed at 229 cm from the top of the examined section (Fig. 4A). A decrease in the χ_{lf} value was identified in the deeper parts of the horizon, with a sharp transition detected at a depth of 270 cm (61×10^{-8} $m^3 kg^{-1}$) and continuing to the basal part of the profile, where the values of 18×10^{-8} $m^3 kg^{-1}$ were recorded. The values represent the absolute minimum found on the examined profile. These low χ_{lf} values are probably the result of gleization due to variation in underground and surface water levels. The results display very similar variations to those recorded during previous research of LPSs in Serbia; also to those on the Eastern and

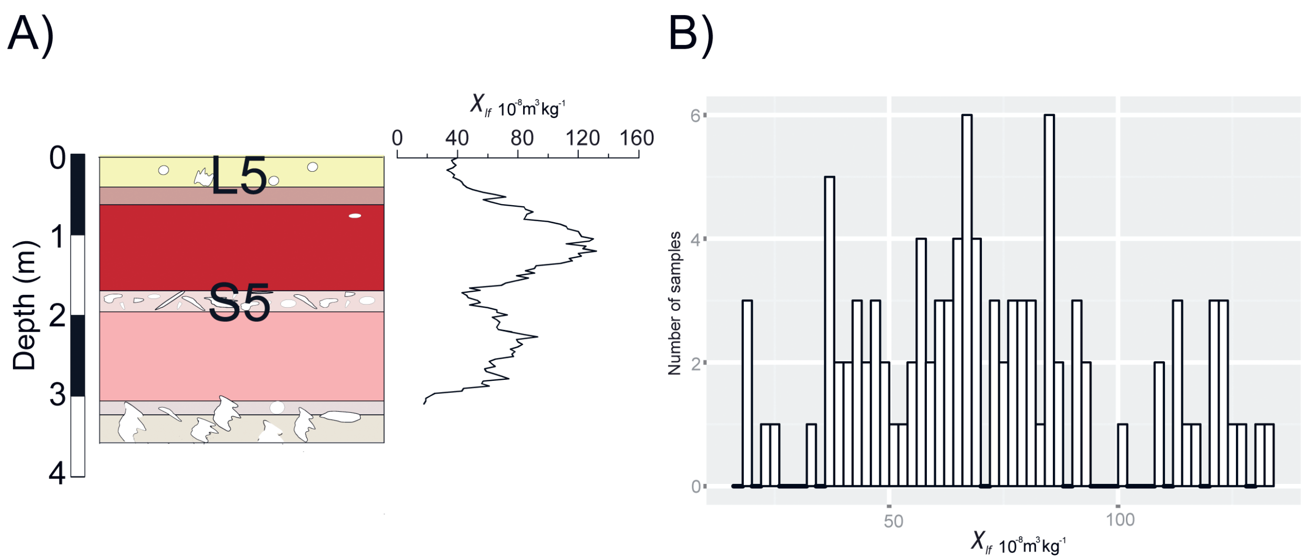

Figure 4. A) The low-frequency magnetic susceptibility measurements (χ_{lf}) for the Dukatar profile; B) Histogram for χ_{lf}.

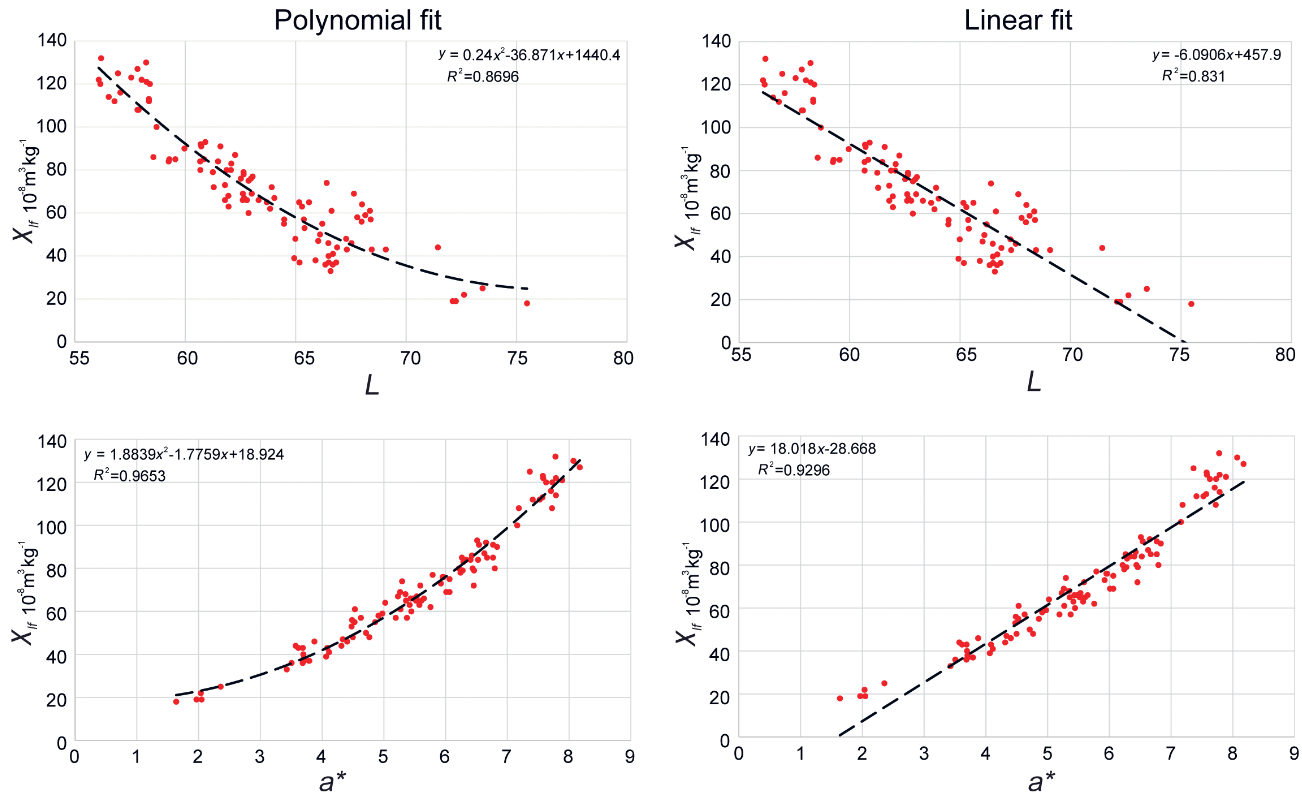

Figure 5. Polynomial amd linear fit of the magnetic susceptibility data χ_{lf}, *L*, and *a** for Dukatar LPS. The y and x in the regression equalions refer to the mentioned parameters.

Western coast of the Black Sea and in Central and East Asia (China) (e.g. HELLER & EVANS, 1995; BUGGLE et al., 2009; BUGGLE et al., 2014; MARKOVIĆ et al., 2009, 2012; BASARIN et al., 2014).

In Fig. 5 the four panels present the linear and polynomial fit of *L* and *a** with magnetic properties of the S5 pedocomplex. The correlation between the χ_{lf} and *L* of the samples in Dukatar is very strong; high values of *L* correlate to the low values of χ_{lf}. This can be explained by the higher concentrations of calcium carbonate ($CaCO_3$) along some horizons of the pedocomplex, which do not possess Fe minerals, and provides the white colour. The coefficient of determination R^2 is best expressed as the polynomial fit, and has the value of 0.87. In contrast, the higher *a** values correspond to higher χ_{lf}, and the trend is steadily rising. The high values of *a** present the red colour which most likely comes from ferromagnetic haematite. The R^2 value is also fitted better in polynomial case, with the value of 0.97.

3.3. Soil Developed Indices (SDI): Rubification (*RI*) and melanization (*MI*) indices

In moist conditions, the hues determined using a Munsell Soil Colour Chart for the first 66 cm of the examined sequence hues, range between 10YR3/6 and 10YR5/6. In the next 90 cm the hues vary from 7.5YR3/4 to 7.5YR4/6; for a depth from 159 cm to 288 cm hues range from 10YR3/4 and 10YR5/6. In the remaining part of the profile, the hue has the value of 2,5Y5/6. At the same depths in dry conditions, the values ranged from 2.5Y6/4 to 10YR7/4, then from 10YR6/6 to 7.5YR5/6, from 10YR4/6 to 10YR7/4 and 2.5Y7/4 respectively.

Quantified soil development indices (SDI), rubification (*RI*) and melanization (*MI*) are shown in Fig. 6. The *RI* shows relatively low variability in the first 57 cm from the upper part of the sampled section. Calculated values vary from 10 to 40, with only one extreme value observed at 24 cm (10). From 60 cm to 150 cm

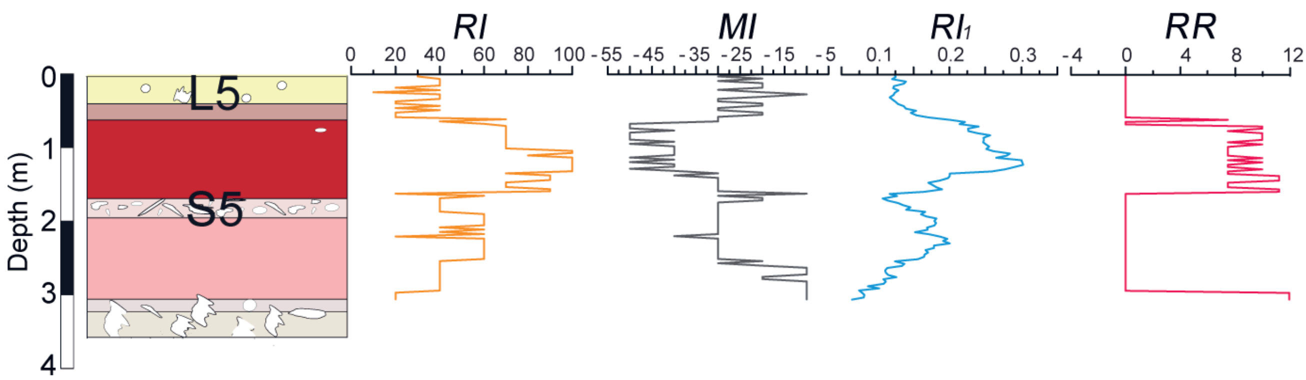

Figure 6. The Rubification index (*RI*), Melanization index (*MI*), Redness Index (*RI1*), and the Redness Rating index (*RR*) for the Dukatar LPS.

of the section, *RI* values progressively increase from 40 to 100. A pronounced decline in values was observed at a depth between 165 cm and 170 cm (90 to 40) which is consistent with measured *a** chromaticity values. At a depth of 216 cm of the examined section, *RI* values gradually decline from 60 to 20.

Melanization index (*MI*) in the first 63 cm of the profile is characterized by moderate variations ranging from –10 to –30. Thereafter, index values vary from –30 to –50 to a depth of 84 cm. Then a progressive decline in values occurs, with –10 being the most extreme one, detected at 171 cm from the top of the profile. At a depth between 171 cm and 258 cm relatively stable *MI* values (–30) are present. In this part of the section, only one value of –40 was noted (at a depth of 222 cm). From 258 cm, a gradual decrease in values ranging from –30 to –10 was apparent. *RI* and *MI* values, obtained through soil sample analysis using a Munsell Soil Colour Chart (observed with the naked eye) also largely correspond to the results from measurement of *a** chromaticity and *L* lightness values using a tristimulus colorimeter.

3.4. Soil colour proxies: Redness (*RI1*) and Redness rating (*RR*) indices

The calculated redness index values (*RI1*) are shown in Fig. 6. Values of *RI1* that may relate to relative changes in haematite content (BARRON & TORRENT, 1986) in the examined pedocomplex, appear to correspond to the detected variations recorded in the signal of χ_{lf} (Fig. 4A). These values remain relatively low in the top 30 cm of the upper part of the section, and vary between 0.12 and 0.11, presumably indicating a slightly lower haematite content. After this depth, the possible haematite concentration gradually increases to a depth of 126 cm, with a maximum value of 0.30. Thereafter, the *RI1* values decrease to a depth of 189 cm, where the minimum value was recorded (0.10). The values then gradually increase to a depth of 234 cm from the upper part (0.21), before decreasing again towards the basal part of the sequence. Consequently, it can be noted that *RI1* values generally correspond to the measured values of *L*, *a**, and *b** colorimetric indicators and SDI *RI* and *MI*.

Soil redness rating index (*RR*) values, associated with the pedostratigraphy of the section are also shown in Fig. 6. Therefore, *RI1* as well as *RR* indices could reflect the haematite content in the examined Dukatar S5 pedocomplex in a semi-quantitative manner. The values appear relatively uniform for the first 78 cm from the upper part of the section and amount to 0, with one exception, observed at 72 cm (7.5). Thereafter, the index values increase to a depth of 156 cm, ranging from 7.5 to 11.25. At a depth of 171 cm the values again drop to zero. The basal part of this site is characterized by the highest *RR* values of 12. Soil *RR* values correspond to a large degree to the values of *L*, *a**, and *b** colorimetric indicators, SDI *RI* and *MI* and χ_{lf} values for the first 156 cm from the top of the examined section. Values detected in the second half of the Dukatar section at the contact between horizons Bw and Ck show slightly higher deviations, especially in the basal part. This can be explained by the presence of massive carbonate concretions.

4. DISCUSSION

4.1. Magnetic susceptibility and colour as indicators of palaeoclimatic and palaeoecological processes at the Dukatar pedocomplex S5

Studies focused solely on instrumental colour determination of palaeosols are relatively scarce in Serbia. The results presented in this study represent the first palaeoclimatic/palaeoecological reconstruction based on colorimetric values of the strongly rubified interglacial S5 pedocomplex, formed in the time frame between MIS 13-15 at the Dukatar site.

The observed changes, from yellowish-brown to reddish-brown, along with the corresponding indices, most likely indicate variations between haematite and goethite concentrations (JI et al., 2004; SCHEINOST & SCHWERTMANN, 1999). Studies undertaken in China indicate that on the loess plateaus, where soils are generally well drained, haematite and goethite concentration is closely associated with variations in soil temperature and precipitation/humidity. Magnetic susceptibility measurement of palaeosols from the Chinese Loess Plateau (CLP) indicate palaeo-precipitation variability between 590-1120 mm^{-1} (SARTORI et al., 2005). Comparable palaeo-precipitation data for the Southeastern and Central Pannonian Basin indicates variations of 580 to 650 mm a^{-1} over a similar timescale (PANAIOTU et al., 2001; BRADÁK et al., 2011).

Palaeoclimatic forcing affects the production of pedogenically formed haematite and goethite. KÄMPF & SCHWERTMANN (1983) assert that pedogenic ferrihydrite conversion to haematite is initiated by dehydration in the soil, increasing aridity and/or a sustained increase in temperature. The same research states that unlike haematite, goethite production from ferrihydrite does not display a high degree of sensitivity to temperature change, with its formation being initiated under more humid conditions. Therefore, haematite prevalence in relation to goethite rises with an increase in mean annual air temperature, while goethite prevalence is associated with increased soil moisture. Based on these characteristics, the utilization of SDI along with magnetic measurements for detection of iron-bearing minerals can serve as a useful tool in LPS research. Increases in the haematite/goethite ratio (Hm/Gt) indicate arid conditions, while lower values are an indicator of more humid conditions (LIU et al., 2006).

BUGGLE et al. (2014) in their study on Quaternary climate change in LPSs from South-East Europe, presented results obtained by using diffuse reflectance spectroscopy (DRS) and colorimetric proxies. These were based on predetermined values from the Munsell Soil Colour Chart such as *RI1* and *RR*. The *RI1* and the *RR* rely upon the positive relationship between chroma, and the chroma to lightness ratio, to the total iron oxide content, as well as the power of hue to discriminate between the relative proportions of haematite and goethite (HURST, 1977; BUGGLE et al., 2014). The aforementioned colour proxies in correlation with the χ_{lf} values were used to determine the variation in the presence of ferromagnetic minerals in the LPSs of Batajnica and Stari Slankamen in Serbia, and Mircea Vodă in Romania, as well as the initial results for this study.

Due to a general lack of absolute dating of LPSs, chronostratigraphic models based on the correlation of χ_{lf} values on a regional scale, along with deep-sea sediment records, are crucial for the reconstruction of the palaeoclimatic change dynamics (BUGGLE et al., 2009). The stratigraphic model developed for the Vojvodina loess, while based upon the Chinese loess-stratigraphic system, actually resulted from the systematically conducted research of MARKOVIĆ et al. (2004a, b, 2005, 2006, 2007, 2008, 2009, 2011, 2012, 2014, 2015, 2021a). This stratigraphic model, enabled loess units and pedocomplexes to be linked with the corresponding MIS for the first time. In general, cyclic fluctuations that occur in the χ_{lf} signal correlate well with variations of oxygen isotopes in deep-sea sediments. European stratigraphic members from S0 to S5 are in good agreement with

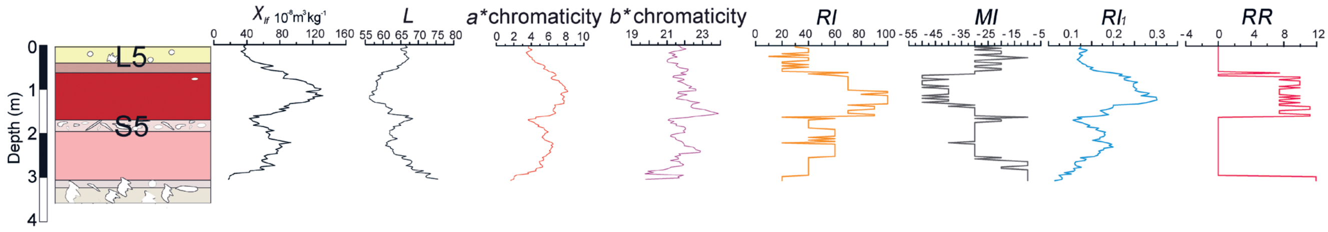

Figure 7. All of the calculated parameters and indices for Dukatar LPS.

stratigraphic members from the CLP (LU et al., 1999), corresponding to the MIS 1, 5, 7, 9 and 11 interglacials.

The thick S5 pedocomplex at Dukatar correlates well with MIS 13 to 15 (MARKOVIĆ et al., 2015). According to its palaeopedological characteristics, this stratigraphic unit must have formed over a longer period of warm and humid climate as it displays elements of more advanced pedogenetic modification than can be found in more recent soils from the region (BRONGER et al., 1998; BRONGER, 2003). The results of BUGGLE et al., (2014) also support this assertion.

Samples from the Dukatar S5 pedocomplex display a general increase in the χ_{lf} value. This pedogenically conditioned increase in magnetic susceptibility indicates the formation of ferromagnetic minerals. Consequently, more investigation is required to clarify pedogenic and palaeoclimatic background of the investigated pedocomplex. A similar increase in S5 pedocomplex χ_{lf} values was also observed in the Batajnica (MARKOVIĆ et al., 2009) and Stari Slankamen LPSs (MARKOVIĆ et al., 2011).

In Fig. 7, all of the parameters and indices from this study are plotted with depth. Sampled at a resolution of 3 cm, the obtained χ_{lf} values display variations with increasing depth: from the contact between the basal part of loess unit L5 and the upper part of the A horizon, through to the contact between the bottom part of the Ck horizon and loessoid material at the base of the exposed part of the profile. The SDIs *RI* and *MI*, as well as *L*, *a**, and *b** colorimetric indicators co vary with the χ_{lf} values. Measured values of *a** chromaticity could be considered as an indicator of the presence of pedogenetic haematite. In addition, these values show a strong correlation with the values of the *RI1* (R^2=0.91, not shown) at the significance level p<0.01.

4.2. Possible relationship between colorimtric indices and ferromagnetic composition

4.2.1. The haematite/goethite model problem

Iron minerals such as haematite and goethite are significant constituents of most soils, and serve as proxy records of climatic and ecological changes. As has been previously asserted, warm and dry conditions are favourable for the formation of haematite, and lead to its separation from ferrihydrite (Fh), while humid conditions are more favourable for the formation of goethite, and lead to its formation by direct excretion from any Fe source (JI et al., 2004). Soils containing only, or almost exclusively goethite, have yellow colour, whereas in soils rich in haematite the colour is usually reddish, due to the fact that red haematite successfully masks yellow goethite (SCHEINOST & SCHWERTMANN, 1999). Unfortunately, the haematite/goethite model in loess (e.g. GUO et al., 2009; LUKIĆ et al., 2014), is not uniformly applicable in all LPSs across Europe. Contrary to this model, ZEEDEN et al. (2015) found haematite present in a loess unit at Krems-Wachtberg LPS. Furthermore, at the Paks LPS the well developed MB and PD2 palaeosols contain significant amounts of pedogenic magnetite and maghemite, but no haematite (BRADAK et al., 2019). Haematite was found both in loess and palaeosols in Czech Republic (OCHES & BANERJEE, 1996). However, in Serbia, the study of LAAG et al. (2021) suggested that tracking the magnetite/maghemite content on its own may not provide a clear enough interpretation of pedogenic processes during the interglacials. They advocated the use of colorimetric data, as it provides direct insight into haematite and goethite content. The same authors revealed that between MIS2 and MIS11 palaeosols contain relatively higher amounts of haematite compared to goethite.

4.2.2. The significance of rubified fossil soils in LPSs

As pointed out by numerous international studies colour variations have been used as a proxy for mineral concentrations in LPSs (e.g. CHEN et al., 2002; VIDIĆ et al., 2004; LUKIĆ et al., 2014; BUGGLE et al., 2014). The reddening of palaeosols is shown to depend of presence of the amount of ferromagnetic minerals such as maghaemite (SCHWERTMANN, 1993) and haematite (CHEN et al., 2002; BUGGLE et al., 2014). LUKIĆ et al. (2014) and BUGGLE et al. (2014) pointed out that *a** chromaticity values can be considered to be a proxy for relative abundance of pedogenic haematite similar to *RI1* and *RR*. This pedogenic haematite may also be responsible for the darkening of fossil soils (as shown on CLP by CHEN et al. (2002)) affecting the *MI* and the *L* chromaticity values, regardless of soil organic matter content. VIDIĆ and LOBNIK (1997) emphasized that the decrease in *MI* depends on time, nature and quality of soil organic matter. There are a few cases where the *MI* obtained in this study does not fully correspond with the other quantified indices at some depths, such as the slight increase in *MI* in weakly rubified Bw horizon. This feature may be attributed to soil components other than organic matter such as Mn-mottles. Based on these initial findings, soil colour indices represent additional sensitive palaeoenvironmental proxies, which, therefore, should be more widely applied in similar investigations including rock magnetic

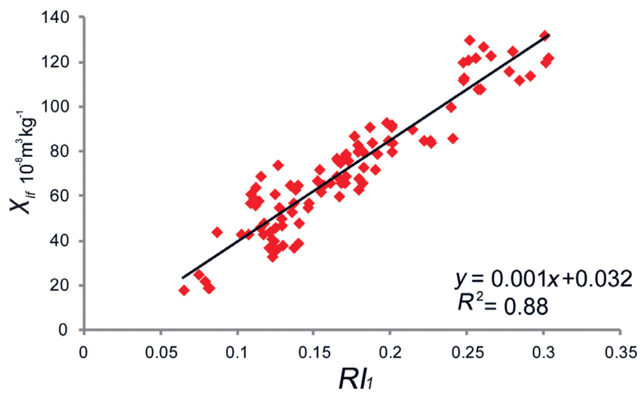

Figure 8. The linear function fitting the Redness Index (*RI1*) and low-frequency magnetic susceptibility measurements (χ_{lf}) of Dukatar LPS.

properties. Significant correlation ($p<0.01$) is observed between *RI* and *MI*, wherein higher *RI* values indicate more intensive red colour, while higher *MI* values indicate its darkening ($R^2=0.76$, not shown). The Dukatar pedocomplex is characterized by the significant presence of carbonates which are excreted from higher horizons and deposited in the central and basal part of the palaeosol S5. The light, whitish colour of the carbonates can be registered through the *L* values and melanization index (*MI*) values. This can be particularly well noted in values observed at a depth between 180 and 195 cm from the upper part of the examined section. As mentioned in the previous chapter, combined utilization of parameters for determining rock magnetism and colorimetric studies has a rather good perspective (LUKIĆ et al., 2014).

4.2.3. Interpretation of the Dukatar pedocomplex colorimetric data

Figure 8. illustrates the significance ($p<0.01$) of the positive relationship between χ_{lf} and *RI1* ($R^2=0.88$); indicating that during the Middle Pleistocene, throughout MIS 13-15 (when pedocomplex S5 was formed) conditions were, most likely, favourable for haematite formation. The regression function for the investigated section is y=0.001x+0.032. BUGGLE et al. (2014) noted that the haematite/goethite value ratio (Hm/(Hm + Gt)) increased between the S4 and S5 palaeosols. These results are somewhat in accordance with the presented initial palaeopedological and geochemical indicators which possibly suggest more intensive development and the effect of eluvial processes in older pedocomplexes (BUGGLE et al., 2013). The soil redness index (*RI1*) of the Dukatar S5 pedocomplex corresponds to a large extent with the detected variations recorded in the χ_{lf}. Therefore, *RI1* can be used as a convenient alternative to magnetic susceptibility for generating paleoclimatic data in the loess province of Vojvodina (north Serbia).

The haematite content appears to be higher in the upper part of the examined section, with the highest values occurring between 146 cm and 180 cm. These findings are confirmed by the values of the soil redness rating index (*RR*). This is in direct contrast to *RI1*, which, after the calcified part of the profile, displays a continuous decrease in values towards the bottom part of the section. With regard to haematite prevalence, the *RR* index between 171 cm to 288 cm in the upper part of the profile records values equal to 0.

The observed differences between these proxies can be explained by their nature, which uses distinct variables upon which differences in soil redness and haematite content are expressed. It should be noted that the *RR* index represents a value which, in a semi-quantitative manner, indicates possible haematite content in the soil. The *RR* index therefore represents a somewhat more conservative method, as the threshold hue used is 7.5YR or greater. In contrast, the *RI1* is not limited in terms of colour hue, and therefore can indicate in a more sensitive manner lesser concentrations of ferromagnetic minerals that cannot produce colour values of at least 7.5YR. Based upon the soil colour index values recorded at the Dukatar section, the use of the *RI1* index is deemed to be a more appropriate metric, since this index is more sensitive to lithological changes and soil development intensity (e.g. MARKOVIĆ et al., 2009). During future studies of this pedocomplex, diffuse reflectance spectroscopy (DRS) should be used in order to determine haematite content in the most precise way possible, as well as haematite and goethite ratios in this pedocomplex.

Based on findings from BUGGLE et al. (2014), which refer to other S5 pedocomplexes in north Serbia, possible palaeoenvironmental conditions could be noted: in common with the Batajnica and Stari Slankamen LPSs, the investigated pedocomplex at Dukatar was formed during the MIS 13-15. It is understood that these stages were characterized by higher summer temperatures and/or longer drought periods. Slightly higher temperatures during the winter months (>0 ºC) could serve as an initial explanation for the increased production of haematite in the S5 pedocomplex, since the presence of organic matter hinders its formation. In addition, a marked increase in magnetic particle concentrations in the Dukatar S5 pedocomplex can be seen on the χ_{lf} plot. Likewise, it is worth mentioning that the S5 pedocomplex in a chronostratigraphic context corresponds well with the S5 lithological unit on the CLP (Fig. 9). The results of χ_{lf} for the Dukatar pedocomplex and Luochuan S5 on the CLP (LU et al., 1999) are presented and highlighted on the Figure 9. Namely, this highly developed pedocomplex (corresponding to the MIS 13-15) is used as a stratigraphic marker in central China because of its dark brown-reddish colour (CHEN et al., 2002; GUO et al., 2013; HAN et al., 1998, LU et al., 1999). Based on the studied pedogenetic characteristics, HAN et al. (1998) assert that the S5 pedocomplex was formed during a period characterized by relatively stable and drier climatic conditions in relation to the climatic conditions during the Holocene. According to YIN and GUO (2008), the existing climatic conditions were spurred by the strengthening of summer monsoon circulation in the northern hemisphere. In addition, the measured values of χ_{lf} presented in this study correlate well with research results of other LPSs both in the region and on the Eurasian continent (BUGGLE et al., 2009; MARKOVIĆ et al., 2009, 2012b, 2015).

In general, higher χ_{lf} values in the S5 pedocomplex on the Dukatar profile also indicate a possible reduction in the excess moisture in the soil, which is associated with slightly higher temperatures during the winter months; this results in a reduction in the precipitation/evaporation ratio. The aforementioned interpretations suggest climatic conditions similar to those in the Mediterranean, prevailed in the loess region of both the Lower and Middle Danube Basin during MIS 13-15 (BUGGLE et al., 2014). The prevailing influence of the Mediterranean climate on soil formation persists for around 180 km southwards of the investigated Dukatar site, as described at the Stalać LPS. Here, a well rubified cambisol was able to form during both MIS 9 and MIS 7, when the Middle Danube basin was already experiencing colder, continental climate conditions (OBREHT et al., 2016).

4.3. Comparative views on interglacial palaeoclimatic and palaeoecological conditions during the formation of S1 and S5 pedocomplexes in northern Serbia

The same methodological approach undertaken for this study was also adopted by LUKIĆ et al. (2014) at the Orlovat LPS (Figure 1). As the Orlovat and Dukatar are the only LPSs investigated from a joint rock magnetic and colorimetric perspective in the north part of Serbia, the comparison between the sections is presented here.

Well defined differences in interglacial palaeoclimatic and palaeoecological conditions become apparent when studying palaeosol S1 at the Orlovat-brickyard, and pedocomplex S5 Dukatar presented in this study (Fig. 10). The S1 palaeosol is a strongly developed soil of approximately 400 cm thickness formed during the MIS 5 period. The aforementioned thickness indicates higher accumulation rates during the final part of MIS 5 and MIS 4 (MARKOVIĆ et al., 2014). The S1 palaeosol has significantly higher χ_{lf} values ranging from 35 to 70 × $10^{-8}m^3kg^{-1}$ in contrast

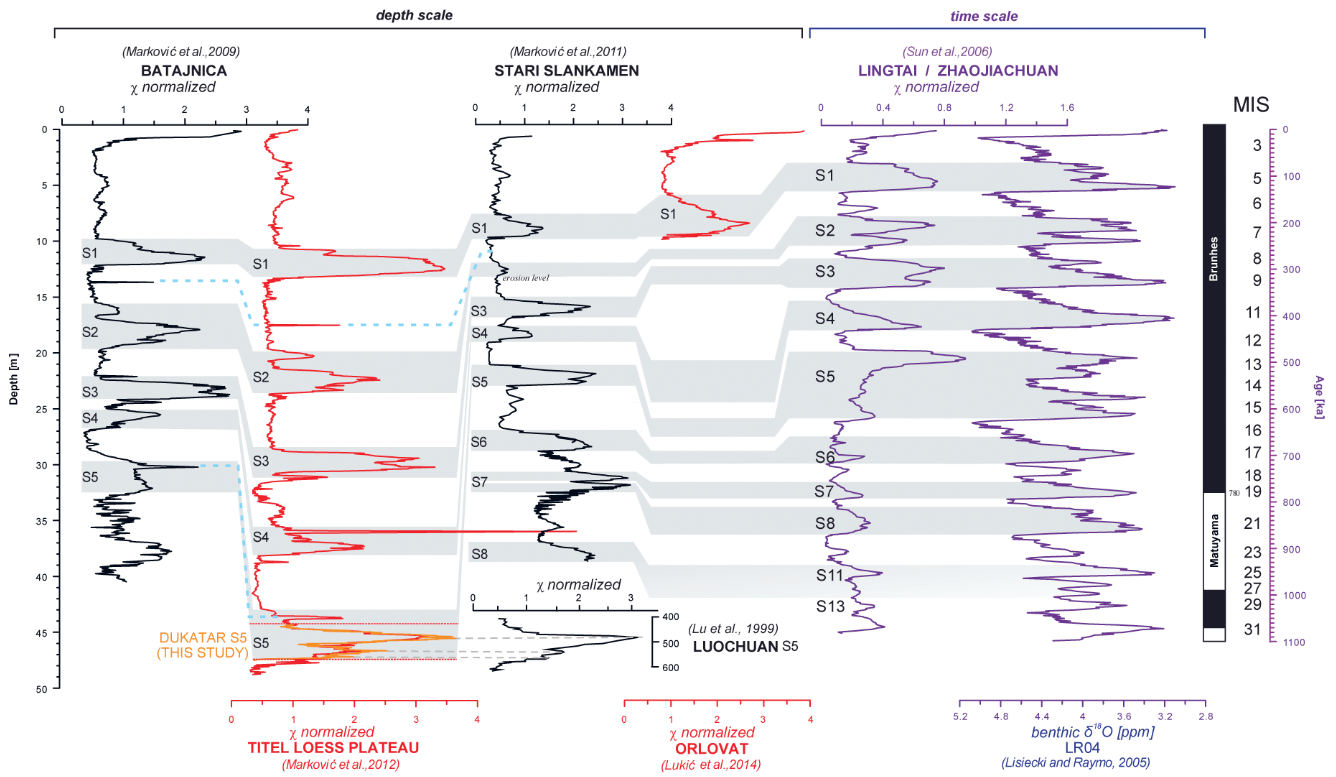

Figure 9. The normalized magnetic susceptibility of some of the oldest LPS in Serbia: Batajnica (MARKOVIĆ et al., 2009), TLP (MARKOVIĆ et al., 2012), Stari Slankamen (MARKOVIĆ et al., 2011), and Orlovat (LUKIĆ et al., 2014) presented on a depth scale and compared to Lingtai/Zhaojiachuan (SUN et al., 2006) and LR04 (LISIECKI & RAYMO, 2005) benthic oxygen isotope record presenting the time scale. MIS stages as well as magnetic reversals are presented. Note the data from Dukatar S5 next to the TLP LPS.

to the L1 loess unit which shows small variations in χ_{lf} values (cca. 20 × $10^{-8}m^3kg^{-1}$). These values gradually increase towards the base of S1, possibly indicating pedogenetic formation of superparamagnetic and single-domain ferromagnetics (LUKIĆ et al., 2014). The χ_{lf} plot shows high similarity with the corresponding palaeosols at other sites in Vojvodina, the Carpathian region and in the CLP (MARKOVIĆ et al., 2009, 2012, 2014). The measured values of *a** chromaticity generally follow the χ_{lf} values, *RI* and *MI*. Warmer climatic conditions during the lower Eemian are presumably reflected in higher *a** chromaticity, *RI* and *MI* values at the top of the S1 lithological member. Humid episodes, which provided sufficient moisture in the soil, occurred during the late spring and early summer. The existence of a highly differentiated Ah horizon, characterized by the accumulation of or-

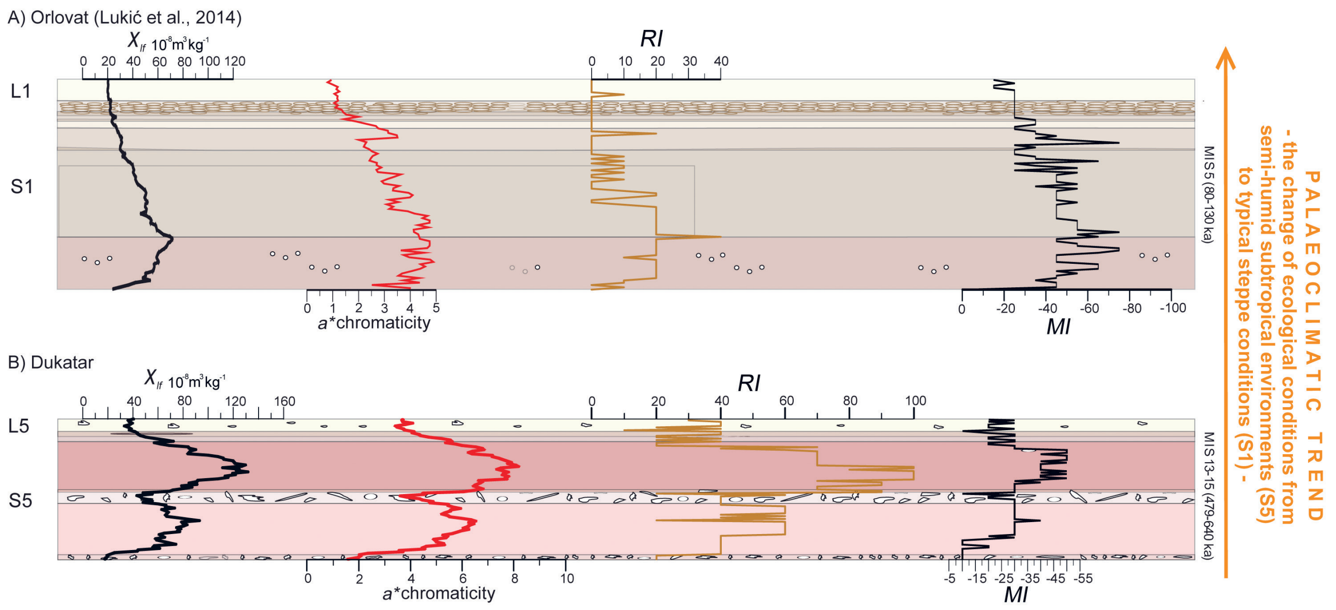

Figure 10. The low-frequency magnetic susceptibility measurements (χ_{lf}), *a** chromaticity, Rubification index (*RI*) and Melanization index (*MI*) for A) Orlovat LPS (LUKIĆ et al., 2014); and B) Dukatar LPS (covered by this study).

ganic matter supports this conclusion (LUKIĆ et al., 2014). In general, this chernozem palaeosol indicates a weakening of pedogenesis from interglacial conditions towards the early glacial period (MARKOVIĆ et al., 2012, 2014).

The χ_{lf} record of Dukatar S5 shows variations due to the presence of carbonate concretions and hydromorphic features. Based upon the data presented in Fig. 10, it can be observed that the concentration of ferromagnetic minerals is significantly higher in the Dukatar S5 pedocomplex compared to the S1 in Orlovat. Significantly higher rubification index values in the S5 pedocomplex indicate the dominance of different pedogenetic conditions to those in which the S1 steppe palaeosol was formed. This finding, to a large extent, coincides with other loess-palaeosol studies in which rubification index values were presented (e.g. VIDIĆ et al., 2004; MARKOVIĆ et al., 2009, 2011; BUGGLE et al., 2014); all indicate a strongly expressed palaeoclimatic transition from sub-Mediterranean to arid continental climatic conditions in this part of Europe during the last five glacial-interglacial cycles. The variations in the *RI* and *a** values presented in this study provide further evidence of palaeoclimatic evolution during different phases of Pleistocene. Compared to the S1 palaeosol, *MI* has significantly lower values due to the presence of different prevailing palaeoclimatic and palaeoecological conditions during the observed interglacial periods. The study of LAAG et al. (2021) asserts that that the general climatic trend was characterized by progressively reduced precipitation and air temperature over the last 430 kyr (covering the formation of palaeosols S4 to S1), gradually leading to more arid climate conditions (from sections about 50 km south of the Dukatar LPS).

5. CONCLUSIONS

The aim of this paper is to present the first reconstruction of palaeoenvironmental and climate variability for the MIS13-15 S5 Dukatar pedocomplex based on high resolution results of the SDI, colour indicators and proxies, supported by χ_{lf}. The observed difference in variations of the soil redness index *RI1* appear to be influenced by haematite content in this rubified pedocomplex, whereas it is significantly reduced in the soil redness rating index (*RR*). Based on the presented values of the soil colour index for the Dukatar section, it could be noted that the use of the *RI1* index is deemed more appropriate, since this index appears more sensitive to lithological changes and soil development intensity, while the *RR* index is considered a somewhat more conservative method. The *RI1* can therefore be considered a more useful palaeoenvironmental proxy, which should be more widely applied in similar multiproxy investigations as a possible tracer of iron-bearing minerals. Moreover, it should be emphasized the multi-proxy approach set out in this study has proved a very favourable methodology for assessing palaeoclimatic and palaeoenvironmental conditions from palaeosols. While the studied indices have confirmed the assertions of earlier studies, regarding the climatic transition from humid Mediterranean towards the more arid continental one, more detailed investigation is required to verify precise ferromagnetic contents, their ratio, and correlation to colorimetric perspectives, in order to clarify pedogenic and paleoclimatic context.

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