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SAR-OSL dating of Late Pleistocene loess in Southern Romania using fine and coarse-quartz

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Loess deposits cover significant areas in Europe, extending from NW-France and Belgium through to central Europe, the Ukraine and Western Russia. The loess palaeosol sequences of the Carpathian Basin-Lower Danube region (Romania, Serbia and Bulgaria) are thought to represent the most continuous and high resolution archives of regional climate and environmental change during the Late and Middle Pleistocene in SE Europe and a link between similar deposits in central Europe and Eurasia. However, in comparison to other loess sequences elsewhere in Western, Central and Eastern Europe, the deposits in Romania have been much less extensively studied.

Luminescence dating is, at present, the only method that allows establishing an absolute chronology for loess deposits by virtue of its ability to directly date the moment of sediment deposition. Moreover, the aeolian nature of loess ensures that the luminescence signal is completely reset prior to deposition, a prime requisite for luminescence dating. Thus, loess sediments are ideal materials for developing, testing and applying luminescence techniques. This approach is essential for securely linking loess records from Romania in a chronologically reliable regional framework and to extend this information to other sites from central and eastern European loess belt, in order to understand past paleoenvironmental dynamics at both regional and continental scales.

Systematic and high-resolution optically stimulated luminescence dating studies on Romanian loess have emerged only in the past four years. Previous single aliquot regenerative optically stimulated luminescence (SAR-OSL) dating of fine (4–11 µm) and coarse (63–90 µm) grains of quartz focused on representative loess sections in southeastern Romania (Mircea-Vodă (**Timar et al., 2010; Timar-Gabor et al., 2011, 2012**), Mostiştea (**Vasiliniuc et al., 2011**), Costineşti (**Constantin et al., 2014**), Urluia (**Fitzsimmons and Hambach, 2014**)). They reported significant differences between the ages yielded by fine and coarse quartz extracts from the Last Glacial cycle loess and the cause of such phenomena, despite thorough investigations, still eludes the scientific community.

The current study aims at extending the area of study to the more westerly loess deposits in the Lower Danube Basin and to provide the first absolute chronology of the Late Pleistocene loess in southern Romania. Magnetic susceptibility measurements and a magnetic time-depth model accompany the luminescence investigations.

The site of Lunca (43°50′55″N, 24°45′56″E) is located in the southern Wallachian Plain, on the left bank of the Olt River, approximately 15 km north to its confluence with the Danube River. This section develops over a ∼36-m-deep ravine cut, and it has been previously the subject of rock magnetic susceptibility measurements that identified at least 6 loess-palaeosol alternations.

Seven loess samples (LCA 1-7) were extracted at very high-resolution (10 cm) from the 56 to 120 cm of the upper part of the topmost loess layer using 6 cm-diameter and 20 cm–long stainless steel cylinders. They were open under subdued red-light conditions in the laboratory and the material at each end of the cylinder was removed for high-resolution gamma spectrometry measurements and to determine the water content. The core sediment from each tube was processed according to standard procedures to extract fine (4–11 µm) and coarse (63–90 µm) quartz.

Luminescence investigations confirmed the reliability of the SAR-OSL protocol (**Wintle and Murray, 2006**) previously applied to Romanian loess (preheat at 220 °C for 10 s, cutheat at 180 °C and elevated temperature OSL). The equivalent doses (<80 Gy) determined for fine quartz samples (LCA - 1, 2, 4, 6) are higher than those on coarse material and the measured De fine/De coarse ratios agree within error limits with the expected ratios. This is consistent with the expected results from annual dose rate considerations, according to which the alpha particles' contribution to the annual dose rate of finer grains determine higher equivalent doses for finer material.

In the case of the youngest sample (LCA 1), the fine and coarse quartz OSL ages perfectly agree. Almost 28 cm-below the lower boundary of the actual soil (S0), this sample yields an Early Holocene age. This suggests that the loess deposition continued well into the Early Holocene and the formation of the strongly developed modern chernozem started less than 11.3 ± 1.0 ka ago and. Similar results are reported for loess sections in Mircea-Vodă and Urluia in Dobrogea (**Timar-Gabor et al., 2011** and **Fitzsimmons and Hambach, 2014**), where fine quartz OSL ages of 8.7 ± 1.3 ka and 11.3 ± 1.6 ka (Mircea-Vodă) and 11.1 ± 1.1 ka (Urluia) have been obtained for the upper part of the L1 loess layer. Delayed start of the soil formation has been also dated in loess sections in Vojvodina region, Serbia. Quartz OSL ages of 10.1 ± 0.8 ka (63–90 µm) and of 8.6 ± 0.7 ka (4–11 µm) (Orlovat; **Marković et al., 2014** and **Timar-Gabor et al., 2014** an IRSL age of 10.2 ± 1.1 ka (Mosorin; **Bokhorst and Vandenberghe (2009)**), and a cal^{14}C age of 7.3 ± 0.4 cal ka BP (Surduk; **Hatté et al. (2013)**) have been obtained on loess collected from well below the Holocene boundary and **Schmidt et al. (2010)** dated to 7.6 ± 0.5 ka the lower part of the modern soil in Stari Slankamen. However, in Crvenka **Stevens et al. (2011)** reported a fine quartz OSL age of 7.7 ± 0.6 ka for the modern soil and 13 ± 1 ka at its boundary with last glacial loess.

These Early Holocene ages obtained on southeastern European loess indicate that in some circumstances the terrestrial archives lag in recording the regional environmental responses to Holocene global climate shifts. Also, along with previously reported data in Central and further east Europe sites (**Antoine et al., 2001** and **Rousseau et al., 2001**) such results question the synchronicity of the Holocene/Late Pleistocene boundary in terrestrial loess versus global integrated archives.

Samples LCA 2 to LCA 7 yield ages spanning from 19 ka to 23 ka on fine quartz, and from 20 ka to 31 ka on coarse quartz and, that roughly agree within 2σ, and thus can be correlated to MIS 2 (**Lisiecki and Raymo, 2005**). Our data is in agreement with the quartz OSL ages reported by **Timar-Gabor et al. (2011)** and (**Fitzsimmons and Hambach, 2014**) on Last Glacial Maximum in the sites of Urluia and Mircea-Vodă.

Based on the coarse (~20–31 ka; LCA 2-7) and fine (~19–23 ka; LCA 2–7) quartz chronologies, sedimentation rates of 0.04 ± 0.01 m ky^{-1}(R^2 = 0.83) and of 0.12 ± 0.01 m ky^{-1} (R^2 = 0.82) respectively, have been computed for the loess deposited during Last Glacial Maximum in Lunca. These are significantly lower values when compared to those reported for the Urluia site (0.6–2.4 m ky^{-1} **Fitzsimmons and Hambach (2014)**) or for the Serbian and Hungarian loess (>0.6 m ky^{-1} in e.g. Surduk **(Antoine et al., 2009)**; Crvenka (**Stevens et al., 2011**); Süttő (**Novothny et al., 2011**)). However, similar low loess accumulation rates during the LGM have been computed for the Mircea-Vodă section (0.04 m ky^{-1} **Timar-Gabor et al. (2011)**) as well as for Serbian loess (**Schmidt et al., 2010**). The strongly variable data available in the Lower Danube basin impose caution when establishing a regional loess depositional model in this part of Europe.

From a methodological standpoint, the Late Pleistocene samples from Lunca provided further evidence regarding the discrepancy previously observed on coarse and fine quartz OSL ages in SE Romanian and Serbian loess. The quartz OSL dose–response curves were mathematically described by a double saturating exponential function. As **Timar-Gabor and Wintle (2013)** in Costineşti, **Constantin et al. (in press)** reported for the Lunca section that natural dose response does not coincide with the average laboratory dose response, both in terms of the function used for fitting and the saturation characteristics. The coarse quartz natural and laboratory dose–response curves coincide up to ~50–100 Gy while for fine material the two growth curves start to deviate at doses more than 100 Gy. This was further confirmed by the fact that the Late Pleistocene samples (De<80 Gy) collected from Lunca, yielded OSL ages were in agreement between the two quartz grain sizes. Sample (LCA 1) that yielded OSL ages in perfect agreement represents the only case where the equivalent doses on the two quartz grain-sizes lie on the regions where the laboratory and the natural dose–response curves.

In conclusion, the SAR-OSL protocol was successfully applied to date fine and coarse quartz extracts from Late Pleistocene and Holocene loess samples in southern Romania. The obtained OSL chronology sustained the hypothesis that, at least at a local scale, the Holocene-Pleistocene limit is not recorded synchronously by the terrestrial archives compared to other proxies and that establishing a regional sedimentation framework in southeastern Europe is not a straightforward issue. Regarding the problem of the discrepant chronologies previously reported for Romanian loess, the concordant OSL ages obtained on young loess samples from Lunca sustain our confidence that for equivalent doses lower than 80 Gy loess OSL ages on both quartz fractions are reliable.

Thus, the optically stimulated luminescence method remains a powerful and robust tool for building absolute chronologies of loess-palaeosol archives in Romania covering the Last Termination and its limit to Holocene.

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