#### Quaternary Science Reviews 183 (2018) 88-109

Contents lists available at ScienceDirect

### **Quaternary Science Reviews**

journal homepage: www.elsevier.com/locate/quascirev

# Chemical weathering of palaeosols from the Lower Palaeolithic site of Valle Giumentina, central Italy

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#### ARTICLE INFO

Article history: Received 24 August 2017 Received in revised form 15 January 2018 Accepted 24 January 2018

Keywords: Paleoclimatology Major and trace element composition <sup>40</sup>Ar/<sup>39</sup>Ar dating Soil weathering Middle Pleistocene Lower Palaeolithic Europe Central Italy

#### ABSTRACT

The major archaeological site of Valle Giumentina (Abruzzo) contains a well-dated Lower Palaeolithic pedosedimentary sequence that provides an excellent opportunity to study the relationships among soil weathering, volcanism and climate change at the glacial/interglacial and submillennial timescales in central Italy and the Mediterranean area during the Middle Pleistocene, as well as the humanenvironment interactions of some of the earliest settlements in central southern Europe. Highresolution analyses of geochemistry and magnetic susceptibility revealed the presence of eleven palaeosols, ten of which (S2-S11) were formed between 560 and 450 ka based on  ${}^{40}$ Ar/ ${}^{39}$ Ar dating of sanidine in tephras, i.e. spanning marine isotope stages (MIS) 14-12. The evolution of the major and trace element composition suggests that the palaeosols were mainly formed by in situ weathering of the parent material. The major phases of soil weathering occurred during the MIS 13 interglacial period (S8 and S6) as well as during episodes of rapid environmental change associated with millennial climatic oscillations during the MIS 14 and 12 glaciations (S11 and S2, respectively). Although global forcing such as orbital variations, solar radiation, and greenhouse gas concentrations may have influenced the pedogenic processes, the volcanism in central Italy, climate change in the central Mediterranean, and tectono-sedimentary evolution of the Valle Giumentina basin also impacted and triggered the formation of most palaeosols, which provided subsistence resources for the Lower Palaeolithic human communities. This study highlights the importance of having high-resolution palaeoenvironmental records with accurate chronology as close as possible to archaeological sites to study human-environment interactions.

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#### 1. Introduction

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The characterization of the environmental context of Palaeolithic sites is important to understand the impact of climate change and landscape evolution on human settlements in prehistoric times. The Lower Pleistocene hominid populations outside East Africa in temperate environments north of latitude 40° were supposed to be often spatially and temporally discontinuous with an occupation largely confined to interglacial periods (Dennell, 2003). In southern Europe, the early human occupation was strongly constrained by climatic conditions and availabilities of resources rather than physiography or cultural factors, and then populations gradually increased their degree of adaptation to diversified environments (Messager et al., 2011; Orain et al., 2013;

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https://doi.org/10.1016/j.quascirev.2018.01.014 0277-3791/© 2018 Elsevier Ltd. All rights reserved.





#### Agusti et al., 2015).

During the Middle Pleistocene, the European hominins were present in a wide range of environments, while an increase in the number of archaeological sites around 500 ka is probably indicative of a more substantial occupation than in the previous period (Roebroeks, 2001, 2006). A population model for Middle Pleistocene Europe proposed that hominins survived in glacial refugia in southern Europe and expanded northwards in interstadial and interglacial periods (Dennell et al., 2011). In this model, territorial occupation outside glacial refugia should have been restricted to warm or temperate periods.

However, these large-scale assumptions rely on a poor European Middle Pleistocene chronological framework and therefore more high-resolution continental climate records together with more accurate and precise chronology of archaeological sites are clearly needed in order to assess the impact of climatic change on the early human populations in Europe. Soil weathering proxies are potentially more efficient than global scale ice or marine records for palaeoenvironmental reconstructions at the Earth's surface, given that soils are in direct contact with the atmosphere at the time of their formation and mark the local to regional response to climate conditions (Sheldon and Tabor, 2009). In Europe, the weathering intensity of palaeosols was quantified using magnetic parameters and chemical indices based on elemental ratios in order to reconstruct the Pleistocene climatic oscillations (Bokhorst et al., 2009; Babek et al., 2011; Buggle et al., 2011; Hosek et al., 2015; Obreht et al., 2015; Krauss et al., 2016; Profe et al., 2016; Regattieri et al., 2016).

The Plio-Pleistocene sedimentary basins in southern and central Italy contain a succession of fluvio-lacustrine sediments and palaeosols, which recorded the variations of the environmental conditions of Lower Palaeolithic sites in southern Europe (e.g. Raynal et al., 1998; Lefèvre et al., 2010; Orain et al., 2013; Aureli et al., 2015; Nicoud et al., 2016; Villa et al., 2016a). Furthermore, the <sup>40</sup>Ar/<sup>39</sup>Ar dating of tephras interstratified in these basin sequences provided reliable and robust chronological frameworks independent of the sedimentary setting (e.g. Giaccio et al., 2013a, 2014; Pereira et al., 2015; Aureli et al., 2015; Villa et al., 2016a).

This paper focuses on a Middle Pleistocene soil sequence with Lower Palaeolithic industries in central Italy and examines the relationships among palaeoweathering intensity, climate change at different time scales, volcanism, tectono-sedimentary dynamics, and topographic context of several periods of human settlements. The impact of regional atmospheric and oceanic circulation patterns as well as solar, orbital, and greenhouse gas forcing on the variability of the Middle Pleistocene soil weathering from 560 to 450 ka is also discussed.

#### 2. Geological setting

The archaeological site of Valle Giumentina (VG) is located on the Adriatic side of the Apennine Chain (Maiella mountain range) at 735 m above present sea level (Fig. 1). This major Palaeolithic site contains more than ten archaeological layers with in situ lithic artefacts first attributed to the Clactonian, Acheulian and Levalloisian traditions (Demangeot and Radmilli, 1966) that are now under revision using lithic technology and techno-functional analysis (Nicoud et al., 2015, 2016). These archaeological layers were found in palaeosols developed on fluvial, glacial, or lacustrine sediments filling an intramontane basin formed by faulting and karstic dissolution during the Quaternary uplift of the Apennines (Demangeot and Radmilli, 1966; Nicoud et al., 2016; Villa et al., 2016a, 2016b; Villa, 2017). The VG basin is surrounded by anticlines developed on the Mesozoic-Cenozoic carbonate rocks in the outer zone of the central Apennine fold-and-thrust belt (Pizzi, 2003). The maximum uplift rate in the surroundings of VG can be estimated at ca. 1.1 mm yr<sup>-1</sup> over the last 0.7 Ma (Pizzi, 2003).

Villa et al. (2016a) proposed a tephrochonological record of the VG basin sequence based on  ${}^{40}$ Ar/ ${}^{39}$ Ar dating of sanidine crystals. The excavated archaeological levels are comprised between ca. 560 and < 450 ka. Hence, the VG pedosedimentary sequence could have recorded the environmental changes associated with the glacial periods of the marine isotope stages (MIS) 14 and 12, and with the MIS 13 interglacial period. These glacial-interglacial changes are in agreement with a palaeoecological study of the molluscan fauna of the VG sequence, which shows mollusc assemblages indicative of closed forest and open vegetation developed under temperate and cold conditions, respectively (Limondin-Lozouet et al., 2017). Besides, the molluscan succession of the VG sequence was allocated to the Middle Pleistocene based on biostratigraphic correlations with other well-dated molluscan records in Italy (Limondin-Lozouet et al., 2017).

#### 3. Materials and methods

#### 3.1. Sampling

A vertical cross-section with a total height of 16.3 m was excavated on the edge of a small valley incised into a sequence composed of a succession of palaeosol horizons and sediment layers filling the VG basin (Fig. 2). A total of 588 samples were taken for geochemical analyses, each 1-2 cm thick in palaeosols and each 3-10 cm thick in sediments (Table S1).

#### 3.2. Geochemical analyses

The chemical composition of samples of palaeosol and sediment was studied using energy-dispersive X-ray fluorescence (ED-XRF) spectrometry (Delta Innov-X spectrometer equipped with an Autube). The samples were previously covered with an ultrafine polyethylene film. The element contents of Si, K, Ca, and Fe were measured by using the 2-beam mining analytical mode, while the contents of Ti and trace elements (Cr, Co, Cu, Zn, Rb, Sr, Zr, Ba, Pb) were measured from the 3-beam soil analytical mode. The soil and mining analytical modes use the following parameters of voltage, amperage, and counting times: 40 kV, 0.07 mA, 15 s for the first beam of soil mode; 40 kV, 0.04 mA, 15 s for the second beam of soil mode; 10 kV, 0.045 mA, 20 s for the third beam of soil mode; 10 kV, 0.03 mA, 5 s for the first beam of mining mode; 40 kV, 0.01 mA, 10 s for the second beam of mining mode. The instrumental errors are lower than  $\pm 5\%$  for Si, K, Ca, Fe, Rb, Sr, Zr, and between  $\pm 5$  and  $\pm 15\%$ for Ti, Cr, Co, Cu, Zn, Ba, Pb. Elements lighter than Si were not measured with the used spectrometer.

#### 3.3. Magnetic susceptibility

The volume magnetic susceptibility (VMS) was measured in situ along the cross-section at a 1-cm vertical resolution with a Bartington MS2 susceptibilimeter and a MS2F probe. The measurements were achieved using the range 1 with a period of 1.5 s, an operating frequency of 0.58 kHz, and a precision of  $1 \times 10^{-5}$  (SI).

#### 3.4. <sup>40</sup>Ar/<sup>39</sup>Ar dating

 $^{40}$ Ar/ $^{39}$ Ar analyses were performed on single-crystals of potassium feldspars (sanidines) extracted from tephra deposits in four palaeosols of the sequence of Valle Giumentina (Fig. 2). Three  $^{40}$ Ar/ $^{39}$ Ar ages were published in Villa et al. (2016a) for the tephras T103b (456 ± 2 ka), T109b (531 ± 5 ka), and T115 (556 ± 6 ka). The sample preparation and analytical protocol are detailed in Villa

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