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Late Pleistocene coprolites from Qurta (Egypt) and the potential of interdisciplinary research involving micromorphology, plant macrofossil and biomarker analyses

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ABSTRACT

As part of a rock art dating project at Qurta (Upper Egypt), samples were collected from an organic deposit and from an accumulation of individual faecal pellets. Radiocarbon dating of these relatively well-preserved materials indicates an unexpectedly old age of ca. 45,000 BP or older. In order to identify the biogenic nature of these deposits and to reconstruct the palaeo-environment at the time of their formation, micromorphological, palaeobotanical, and biomarker analyses were carried out. All data indicate that the organic deposit and the pellets were produced by different species. The presence of a novel biomarker, which only occurs in animal urine (hippuric acid), contributed to the conclusion that the organic deposit most likely represents the remains of a rock hyrax (*Procavia capensis*) latrine, whereas the pellets stem from small bovids. Plant macroremains from the pellets indicate that the animals browsed in the more vegetated areas, presumably near the Nile, although the general environment was probably mainly arid and open. Combined with the dates, this suggests that the pellets date to MIS 3 or 4. Our results demonstrate the great potential of an interdisciplinary approach to the study of Quaternary coprolite deposits, allowing for more adequate and more complete interpretation.

1. Introduction

1.1. Coprolite research: methods and applications

Coprolites, i.e., fossil or subfossil excrements, can provide a wealth of information to archaeologists and palaeontologists. They constitute palaeodietary records of humans and animals (Reinhard and Bryant 1992; Ghosh et al. 2008; van Geel et al. 2008; Riley 2012) and contain valuable palaeoenvironmental and palaeoclimate proxies, especially in arid areas where few other archives are available (Scott and Woodborne 2007; Ghosh et al. 2008; Mercuri 2008; Linseele et al. 2010; Marinova et al. 2013; Cremaschi et al. 2014; Carr et al. 2016). In addition, they are informative on the digestive processes (Gill et al. 2010; van Geel et al. 2011), health status and sanitary conditions (Reinhard 1992; Schelvis 1992) of extant and extinct species.

Coprolite research traditionally entails macroscopic and microscopic analysis of plant macrofossils, pollen, phytoliths, spherulites and intestinal parasites (Brochier et al. 1992; Reinhard and Bryant 1992; Bryant and Dean 2006; Shahack-Gross 2011), although micromorphological studies of undisturbed dung materials in thin section are scarce (see Macphail and Goldberg 2018). Dung deposits and related zoogenic accumulations in rock shelters constitute a unique source of information for palaeoecological reconstructions due to the long-term accumulation of various organic materials (Savinetsky et al., 2012). Botanical remains in these deposits not only represent dietary components, they also represent a considerable part of the vegetation that was accessed by the animals and may thus be exploited for reconstructing palaeoenvironment and palaeoclimate, especially in arid areas where few other archives are available.

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Recently, these approaches have been increasingly supplemented with biomolecular and isotopic analyses. Lipid biomarkers have proven particularly valuable in identifying suspected faecal remains in contexts where macroscopic evidence is lacking, including manured soils, latrines or putative animal pens (e.g., Simpson et al. 1999; Bull et al. 2001; Baeten et al. 2012). Among lipid biomarkers, 5β-stanols and bile acids received much attention as they are not only diagnostic for faeces, they also provide information about the biological source of the excrements (Bull et al. 2002; Gill et al. 2010; Hofmann et al. 2010). In that respect, recent analysis of modern reference materials contributed to a greater discriminative power of faecal steroids (Prost et al. 2017; Zocatelli et al. 2017). Faecal 5β-stanols ratios also indicate the degree of plant consumption (van Geel et al. 2008, 2011), and recently contributed to a better understanding of the evolution of human diets (Sistiaga et al., 2014a, 2015). Aside from these faecal steroids, coprolites often contain other lipid biomarkers such as alkanes, alkanols or triterpenoids, which generally represent dietary components of the animal's last meal (van Geel et al. 2008, 2011; Gill et al. 2009; Carr et al. 2010). Ancient DNA analysis provides precise taxonomic identification of the source animal and the coprolite's organic content (e.g., Poinar et al. 1998, 2001), while stable isotope analysis provides insights into the source animal's dietary components and trophic level (Ghosh et al. 2003; Carr et al. 2010, 2016), as well as palaeoclimatic issues (e.g., Chase et al. 2009; 2010, 2012; Meadows et al. 2010).

1.2. The Qurta rock art and the find of the coprolite deposits

The circumstances of the discovery of the Qurta rock art and its broader environmental and cultural Late Pleistocene context have already been detailed in previous publications (e.g., Huyge 2008; 2009; Huyge and Claes 2008; Huyge and Ikram 2009) and will not be reiterated here. Since 2005, three rock art sites have been identified at Qurta in Upper Egypt: Qurta I, II and III (henceforth QI, QII and QIII; Fig. 1). A major breakthrough in obtaining absolute dates for the Qurta rock art, was the discovery in 2008 of some partly buried rock art panels at QII (Fig. 2). The hillslope deposits covering the rock art have been dated directly using optically stimulated luminescence (OSL) and indirectly using radiocarbon dating of bioapatite from microvertebrate faunal remains within these sediments (Huyge et al. 2011). The OSL dates give a minimum age for the covered petroglyphs of between 10 and 17 ka. The two radiocarbon dates on animal bones are congruent, although not entirely internally consistent, with the OSL dates: 12,130 ± 45 ^{14}C yr BP (KIA-41532) and 10,585 ± 50 ^{14}C yr BP (KIA-40546) (Huyge et al. 2011). The discrepancy may result from different events having been dated and/or some exchange of carbonate between the bone material and its environment.

Further fieldwork at QII in March 2011 (Huyge and Vandenberghe, 2011) led to the discovery of several more buried petroglyphs at different levels, and, at QII location 4 (QII.4), of the massive organic deposit (QII.4.3) and the faecal pellet accumulation (QII.4.2) that are the subject of this contribution (Fig. 2). Both are unrelated to the rock art, but their study was initiated because of the broad interest of our group in interdisciplinary coprolite research (Linseele et al. 2013). The massive organic deposit covers a different part of the rock face, and the faecal pellet accumulation was found in sediments covering the rock face with the art (QII.4), but well below the level on which the rock art was executed. Micromammal bones were also found, in sediment below the level of the faecal pellets. The dating of the organic deposits and of the bones (see Section 3.2.) therefore bears no relevance for the age of the rock art.

1.3. Aims of the coprolite research at Qurta

In this paper, we describe and evaluate the dates we obtained on the massive organic deposit, the faecal pellet accumulation and the micromammal bones in relation to the previously published dating information from Qurta. We report on the nature and the content of the massive organic deposit and the faecal pellet accumulation, as we have determined through the analysis of lipid biomarkers, micromorphology, and plant fossils. DNA analysis has not been attempted because of previous experiences with subfossil dung pellets from a Holocene cave site in Egypt, where insufficient DNA was preserved for amplification (Linseele et al. 2013). Our aims were as follows: (1) verify the biogenic source of the massive organic deposit and the faecal pellet accumulation; (2) examine the dietary components of both materials and relate this information to the palaeoenvironment through the palaeovegetation record preserved in the deposits; and (3) evaluate the efficiency of the adopted multidisciplinary approach with respect to objectives 1 and 2.

2. Material and methods

2.1. Description of the samples and their find context

The rock art sites of Qurta are located on the east bank of the Nile, along the northern edge of the Kom Ombo Plain, ca. 40 km south of Edfu and ca. 15 km north of Kom Ombo, on the higher parts of the Nubian sandstone (Said 1962, p. 318) scarp bordering the Nile floodplain, at an altitude of ca. 120–130 m above sea level (i.e., ca. 35–45 m above the current floodplain).

The massive organic deposit (QII.4.3) was found in a near-vertical position, stuck to the upper part of a rock face, within a deep cleft between the sandstone rocks bearing rock art panels QII.4.2 and QII.4.3 (Fig. 2). This cleft was completely filled up with loose, fine, sandy sediment. The massive organic deposit is ca. 5 cm thick and covers a surface of ca. 0.5 m^2. Its total weight is estimated at ca. 1.5–2 kg. It contained some localised remains of faecal pellets that, though much degraded, were macroscopically recognisable as such (see Fig. 3c). The deposit showed some traces of animal burrows and lacked any distinctive layering. The label “massive” refers to this lack of structure of the deposit. About half of the total volume was sampled (Fig. 3a).

The faecal pellet accumulation (QII.4.2) was found at a much lower level, in loose, fine, sandy, wind-blown sediment, ca. 1.8–2 m below the massive organic deposit and ca. 0.5–0.7 m below rock art panel QII.4.2 (Figs. 2 and 3b). All pellets were collected. They are ca. 200 in number and are ovoid in shape. Some well-preserved pellets were present (Fig. 3d), but the surface of most of them seems to have been affected by degradation processes that may also have altered their appearance (Fig. 3e).

The collected organic deposit and the faecal pellets were stored at the Royal Belgian Institute of Natural Sciences after analysis.

2.2. Radiocarbon dating

In preparation for radiocarbon dating of macrobotanical inclusions, one sample of the massive organic deposit and one sample of the faecal pellet accumulation were pre-treated through a series of washes, each lasting ca. 30 min, with hot HCl, NaOH and HCl (1%) alternating with, and ending with, water. With the exception of plant fibres, representing ca. 5% of the initial sample, the pre-treatment removed everything, including humus-like materials (Bonneaul et al., 2011). The micromammal bones from below the pellet accumulation, which were identified as belonging to at least three individuals of mouse (cf. *Mus musculus*), did not contain any collagen. Therefore, the bone apatite was dated. The pre-treatment of the bones consisted of soaking in 1% acetic acid for 24 h. After pre-treatment samples were graphitized using H_2 over a Fe catalyst. Targets were prepared at the Royal Institute for Cultural Heritage in Brussels (Belgium) (Van Strydonck and van der Borg 1990–1991) and ^{14}C concentrations were measured with accelerator mass spectrometry (AMS) at the Leibniz Labor für Altersbestimmung und Isotopenforschung in Kiel (Germany) (Nadeau et al. 1998). The Leibniz-Labor AMS system is a 3 Million Volt Tandetron 4130 AMS system from High Voltage Engineering designed for the analysis of ^{14}C.