Quaternary International 398 (2016) 129-135

Contents lists available at ScienceDirect

## Quaternary International

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

### Dental calculus reveals potential respiratory irritants and ingestion of essential plant-based nutrients at Lower Palaeolithic Qesem Cave Israel

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#### A R T I C L E I N F O

*Article history:* Available online 18 June 2015

Keywords: Lower Palaeolithic Diet Plants Respiratory irritants Dental calculus

#### ABSTRACT

Reconstructing detailed aspects of the lives of Lower Palaeolithic hominins, who lived during the Middle Pleistocene, is challenging due to the restricted nature of the surviving evidence, predominantly animal bones and stone tools. Oesem Cave, Israel (420–200 ka) is a site that has produced evidence for a wealth of innovative features including controlled use of fire, represented by a repeatedly used hearth. Numerous charred bone and stone tools as well as wood ash have been found throughout the ten metres of archaeological deposits. Here, we describe the presence of a range of potentially inhaled, and ingested, materials extracted from samples of dental calculus from the Qesem Cave hominins. These finds offer an insight into the environment in and around the cave, while micro-charcoal highlights the need for smoke management in enclosed environments. Plant fibres and a phytolith may be evidence of oral hygiene activities or of using the teeth to work raw materials. Starch granules and chemical compounds provide a direct link to ingested plant food items. This extends the evidence for consumption of plant foods containing essential nutrients including polyunsaturated fatty acids and carbohydrates, into the Lower Palaeolithic. Together, these results represent a significant breakthrough towards a better understanding of Middle Pleistocene dietary breadth and highlight some of the challenges facing the adoption of the habitual use of fire for cooking by the Qesem Cave hominins, as well as offering an insight into their ecological knowledge and technological adaptability.

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#### 1. Introduction

The Middle Pleistocene was a period of major biological and behavioural change in human evolution (Nowell and White, 2010). Qesem Cave is a karst chamber cave in Israel (420,000–200,000 BP) (Gopher et al., 2010; Mercier, 2013) that has been in excavation since 2001 (Fig. 1). Its finds add weight to the notion that the Late Lower Palaeolithic period was crucial in terms of biological,

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http://dx.doi.org/10.1016/j.quaint.2015.04.033 1040-6182/© 2015 Elsevier Ltd and INQUA. All rights reserved. economic and cultural development of the human species, eventually leading to the appearance of anatomically modern humans as well as Neanderthals. At Qesem Cave, evidence for innovative behaviour includes development of a new mode of adaptation, possibly triggered by the disappearance of elephants, which led to an increasing need to hunt the abundant medium-sized ungulates (Stiner et al., 2009; Ben-Dor et al., 2011; Hershkovitz et al., 2011; Barkai and Gopher, 2013; Blasco et al., 2014; Shahack-Gross et al., 2014). This in turn led to a requirement for higher efficiency in processing food (Ben-Dor et al., 2011; Barkai and Gopher, 2013), to maximize dietary yield of both animal and plant based calories (Groopman et al., 2015). The use of fire inside Qesem Cave, recorded







from 400,000 years ago (Karkanas et al., 2007; Mercier, 2013), and the repeated use of a central hearth from 300,000 years ago (Gopher et al., 2010; Mercier, 2013; Blasco et al., 2014; Shahack-Gross et al., 2014), may be linked to this. The Amudian industry that dominates the cave's lithic assemblages is a distinct bladedominated late Lower Palaeolithic Acheulo-Yabrudian Cultural Complex (AYCC) industry (Gopher et al., 2005; Barkai and Gopher, 2013). It post dates the Acheulian industry and predates the Middle Palaeolithic Mousterian traditions.

Eight hominin teeth from Qesem Cave have been studied thus far (Hershkovitz et al., 2011) and additional teeth are presented in Hershkovitz et al., 2016). Here, we present evidence for potentially inhaled and ingested material in dental calculus extracted from three of these teeth. Dental calculus is formed by bacteria and calcium phosphate salts which combine to create calculus. Recent studies have shown that dental calculus can act as a store for inhaled and ingested material, with evidence for chemical compounds surviving at least into the Middle Palaeolithic (Hardy et al., 2012). Though the Qesem Cave samples were extremely small, we have been able to provide the earliest evidence of exposure to potential respiratory irritants, including micro-charcoal, pollen and evidence for fungal spores, in human history. Our results also extend the survival of chemical compounds into the Lower Palaeolithic and offer the first direct evidence that the diet of these hominins included plants, incorporating both carbohydrates and the essential polyunsaturated fatty linoleic and linolenic acids.

#### 2. Materials and methods

Most archaeological human remains at Tel Aviv University, Israel, are stored in individual closed boxes; in the case of the individual teeth, these were stored individually in closed plastic bags, within the closed boxes. Three dental calculus samples from three teeth were identified and extracted. Tooth G/9b 520-525, was found in the mid-area of the cave, while the teeth G/22 685–690 and G/22 705–710 were found in the same location in the cave, but at slightly different elevations. It is possible they may be from the same individual, as their age of death estimates are similar and the interproximal facets on the teeth articulate well together. However, this cannot be unequivocally confirmed (Hershkovitz et al., 2011). Extraction took place inside a specially constructed box, covered in aluminium paper. As soon as the samples were removed, the paper was folded and inserted individually into Eppendorf tubes; they remained in these tubes until their extraction for analysis. Ideally, each sample of dental calculus should be split into two for different analyses to be conducted on each piece; however, here, the largest sample weighed only 0.36 mg and was too small to split. The largest piece was therefore selected for chemical analysis using the dual methods of sequential thermal desorption-gas chromatography-mass spectrometry (TD-GC-MS) and pyrolysis-gas chromatography mass spectrometry (Py-GC-MS). Optical microscopy, for morphological examination, was conducted on the two smaller pieces [S1].

#### 2.1. Chemical analysis

TD/Py-GC-MS was performed on a CDS Pyroprobe 2000 via a CDS1500 valved interface (320 °C), to a Hewlett–Packard 5890 Series II GC fitted with a split injector (280 °C) interfaced to a Trio 1000 mass spectrometer (electron voltage 70 eV, filament current 220  $\mu$ A, source temperature 200 °C, multiplier voltage 450 V, interface temperature 300 °C). The acquisition was controlled by Windows based MasSpecII32 Data System, in full scan mode (35–650 amu). The sample was weighed into a quartz tube with glass wool plugs. The tube was placed into a pyroprobe platinum heating coil and sealed into the valved interface. The sample was

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