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Using plant microfossils from dental calculus to recover human diet: a case study from Tell al-Raqā'i, Syria

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Abstract

Dietary reconstructions based on plant microfossils, such as starch grains and phytoliths, have been useful in increasing our understanding of past human populations. Microfossils have been recovered from sediments, stone tools, and, more recently, dental calculus. Methods for recovering microfossils from dental calculus have yet to be firmly established and there is some question about potential damage to the teeth. Using a sample of teeth from the middle Holocene site of Tell al-Raqā'i, Syria, we tested using a dental pick to sample the calculus. ESEM images taken before and after sampling show no damage to the enamel surface, and examination of the recovered microfossils show that this method provides ample material for study, even when not all of the calculus is removed from the tooth. Preliminary identification of the plant microfossils suggests that these individuals were consuming a variety of plant foods, but that domesticated cereals such as wheat and barley made up a surprisingly small portion of their diet.

Keywords: Starch grains; Phytoliths; Dental calculus; Diet; ESEM; Plant microfossils

The recovery and identification of plant microfossils has provided a means for archaeologists to reconstruct a more comprehensive view of the diet of past human populations. Plant microfossils, such as phytoliths and starch grains, recovered from ancient sediments, stone tools, and pottery have been used to answer a variety of questions about tool use, origins of agriculture and other aspects of human behavior such as site organization (e.g., Albert et al., 2003; Balme and Beck, 2002; Fullagar et al., 2006; Kaplan et al., 1992; Loy et al., 1992; Madella et al., 2002; Parr and Carter, 2003; Pearsall et al., 2004; Perry, 2004; Piperno, 2006; Piperno et al., 2000, 2004; Piperno and Holst, 1998; Rosen, 1997; Van Peer et al., 2003). Dietary information from sediments and tools may be incomplete or contain biases, however, because these sources may record the use of plants that were not eaten, and may miss others that were not processed prior to consumption, like fruits or roasted tubers. To address these problems, several researchers have begun to investigate how to recover microfossils preserved in dental calculus.

The formation of dental calculus traps food particles, including plant microfossils, into the matrix of calcium phosphate deposits on teeth. These deposits are heavily mineralized and survive well in archaeological contexts (Lieverse, 1999). Plant microfossils are well-protected in the calculus, and, once recovered, they can provide a direct record of the plants placed in the mouth. Both starches and phytoliths have been recovered from human calculus dating to Neolithic and more recent time periods (Lalueza Fox and Pérez-Pérez, 1994; Lalueza Fox et al., 1996; Juan-Tresserras et al., 1997; Scott Cummings and Magennis, 1997), whereas only phytoliths have been documented on non-human animal teeth dating to one million years ago (Ciochon et al., 1990; Middleton and Rovner, 1994; Gobetz

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and Bozarth, 2001). Despite this considerable potential, some questions exist about the best methods for sampling calculus, while at the same time preventing damage to the teeth (Boyadjian et al., 2007).

In this paper, we present data from a site of middle Holocene age in Syria that show how a careful removal of calculus with a dental pick can provide large numbers of microfossils while causing no damage to the teeth and leaving enough calculus for future study. SEM studies confirm that this method produces no new scratches on the enamel surface around and under the calculus deposits. This method works equally well on teeth with thick or thin deposits of calculus. The dental calculus examined in this study provides the oldest combined sample of starches and phytoliths from human teeth yet reported. The identification of the microfossils reveals that these individuals were consuming several different species of plants, and that barley and wheat, the expected major sources of starches, probably made up a surprisingly small portion of their diet.

1. Materials and methods

We sampled five human teeth from burial and other contexts from the site of Tell al-Raqā'i, Syria. The site was excavated by Curvers and Schwartz between 1986 and 1993 as part of a salvage operation along the Habur River Valley prior to the construction of a dam (Curvers and Schwartz, 1990; Schwartz and Curvers, 1992). The site is small, only about 0.5 ha in areal extent and 7 m high, with seven discrete occupation levels. The youngest of these dates to the Hellenistic period, and the oldest dates to the early third millennium BC. Several features of the site, including its small size, the scarcity of pottery, numerous signs of specialized production (including the large 'rounded building', thought to be a grain storage area), and evidence of administrative technology, led the excavators to hypothesize that Tell al-Raqā'i was a grain collecting and processing site for a larger, separate, urban area.

Table 1
Sample names, dates, provenience and burial contexts of human teeth recovered from the site of Tell al-Raqā'i, Syria

Tooth sample	Age	Original provenience number	Burial context
RH1	Early–mid third millennium BC	36/102-48	Brown and grey soil with pebbles, ash and clay oven fragments in level 3, area 72
RH2	Third millennium BC to Islamic	30/114-5	Yellow-brown soft soil above level 3 architecture, below present-day surface of the tell
RH3	Third millennium BC to Islamic	48/102-12	Black ash lens, above level 3 architecture, which could be later level 3 or post-third millennium
RH4	Early third millennium BC	48/108-62	Brown soil with mudbrick debris, above level 4 architecture and below level 3 architecture, in a deposit that could be assigned to either level
RH5	Third millennium BC to Islamic	48/96-11	Fill in a stone drain above level 3 architecture, under present-day surface of the tell

Many charred seeds were preserved at the site, and the study of these macrobotanical remains was performed by van Zeist (2003). He found evidence of the use of several different kinds of grains, including emmer wheat (*Triticum dicoccon* Schrank), einkorn wheat (*Triticum monococcum* L.), free-threshing hard wheat or bread wheat (*Triticum durum* Desf.), and hulled, two-rowed barley (*Hordeum distichum* L.), and several other varieties of wild grains (*Aegilops* spp., *Hordeum* spp., *Avena* spp.). Seeds from other edible plants were also preserved, but in much smaller quantities, including peas (*Lathyrus sativus* L. or *Lathyrus cicera* L., and possibly *Pisum sativum* L.), vetch (*Vicia* spp.), orache (*Atriplex* spp.), mallow (*Malva sylvestris* L.), safflower (*Carthamus tinctorius* L.) and thistle (*Centaurea* spp.), among many others. The plants found in the macrobotanical study provided a list of foods that could be expected to occur as microfossils in the dental calculus.

The five samples we examined were all isolated adult human teeth. Two of the teeth were from securely dated contexts from the third millennium layers of the site (Table 1), while the three others were from layers of uncertain age.[1] The teeth came from a variety of soil contexts within the site, ranging from ashy lenses to fill within a drain. The five teeth were chosen to show the range of calculus expression, from thick deposits on RH1 to very little on RH2 (Fig. 1). While the chemical and mechanical processes that lead to calculus formation are fairly well-understood (Nancollas and Johnsson, 1994; Lieverse, 1999; Jin and Yip, 2002), these processes may vary considerably among individuals, due to differences in dental hygene, age, diet preferences, and even genetic make up. Few empirical data are available with regard to the rate of calculus formation on teeth and how much of an individual's lifespan evident calculus represents. Nevertheless, because calculus does accumulate over an individual's life if not removed, we may surmise that at least several years diet is represented in every individual studied. Additional research is needed on this point.

The area of thickest calculus on each tooth was chosen for sampling, and these areas were examined under an ESEM at 100× magnification, following established dental microwear analysis methods (Gordon, 1988). Several images were taken of the sampling area on each tooth, and these were then aligned into composite images using 'Analysis Docu' software provided with the ESEM. These images provided the baseline against which we assessed any damage to the teeth.

The calculus was then sampled, using a dental curette to pick off small flakes of calculus into a 14 ml glass collection tube. Glass test tubes are preferred over plastic, as the small flakes of calculus can easily become statically charged and

[1] Because none of these teeth were found in recognized burials, we cannot completely rule out the possibility that some of them may have been displaced downward from more recent contexts. The dietary signal recovered from the teeth strongly suggests this is not the case, as described below.

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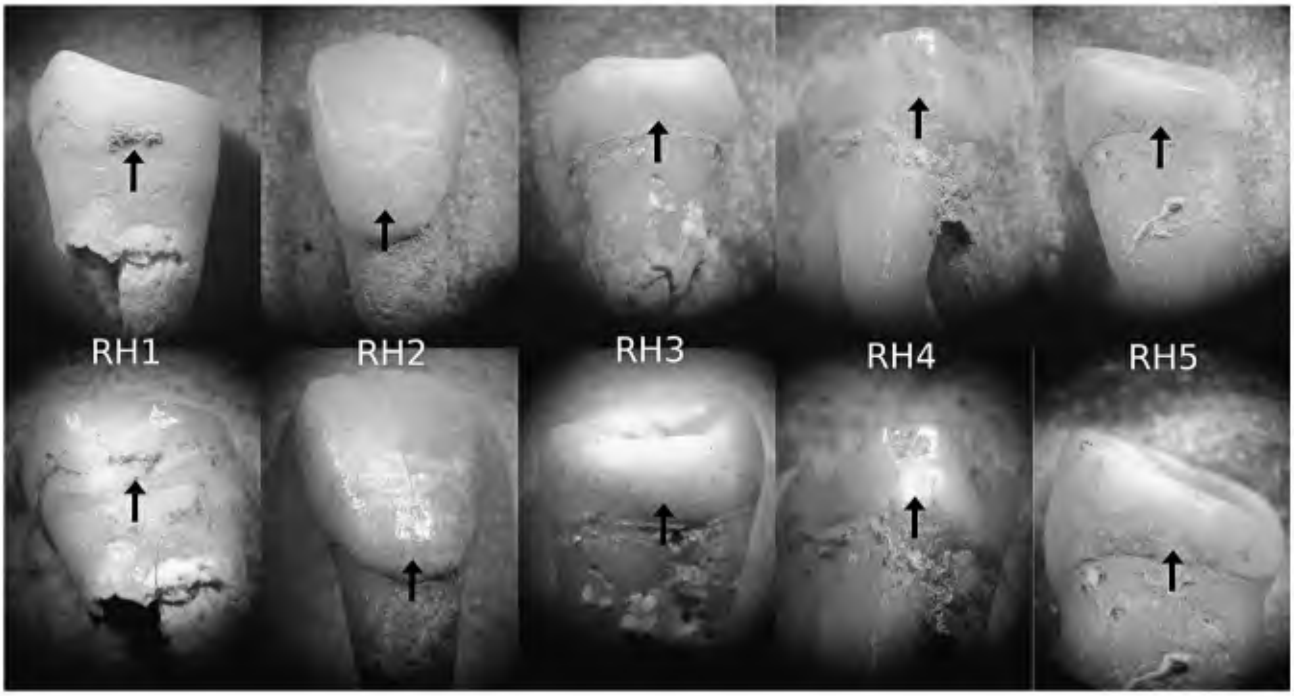

Fig. 1. Top row: teeth RH1 through RH5 prior to sampling. Arrows highlight areas of thickest calculus deposit. Bottom row: RH1 through RH5 after sampling.

stick to plastic tubes. The curette was washed and boiled for 60 s between samples to remove microfossils and prevent cross-contamination. A 10% solution of sodium hexametaphosphate (Calgon) was added to each sample to deflocculate the calculus and ease dispersal. After 24 h, the sample was sonicated for 5 min, then centrifuged (2000 rpm for 2 min), and the supernatant pipetted off. It was then rinsed two times with distilled H_2O. The calculus was dissolved in a 10% solution of HCl for roughly 12 h, and then rinsed twice in distilled H_2O. Experiments on modern starch samples from *Zea mays* have shown that weak solutions of HCl and room temperature reactions over a 24 h period do not damage the starch grains (Henry, unpublished data), though this acid processing step may not be necessary when the calculus is already in a fine powder. The remaining sample was mounted on a microscope slide in a 1:4 glycerin/water solution, and examined under a light microscope at 40×. Each microfossil was counted and photographed, and compared to a reference collection of microfossils from modern plants in order to identify it to family, genus or species where possible.

Finally, the teeth were re-examined under the ESEM using the methods described above. The before and after composite images were then compared using MIPAV, a software package designed by the NIH for processing medical images (McAuliffe et al., 2001). We looked for differences between the before and after images, such as the appearance of new scratches or pits, in order to assess possible damage. Comparing the images also allowed us to measure exactly the area of the calculus that was removed, and to calculate the concentration of microfossils per area sampled. Weighing the calculus before processing it would provide another measure of concentration of microfossils, but was not done in this case.

To identify starch grains and phytoliths that we isolated from the teeth, we utilized a reference collection of economic and other plants native to the study region, as well as those from other areas. This collection includes several hundred reference starches and over 2000 reference phytoliths, including wild wheat, barley, oats, rye, *Aegilops*, other grasses, acorns, pistachios, wild peas, and others. Many of the Near Eastern plants in the collection were utilized previously by Piperno and colleagues for their study of starch grains at the site of Ohalo II, Israel (Piperno et al., 2004). Other plant samples, including some grasses and legumes, were sampled from folders housed at the United States National Herbarium, National Museum of Natural History, Washington, DC, and from a collection of Near Eastern plants previously sampled for a study of goat diet (Wiedemann et al., 1999). We supplemented these wild reference specimens with modern domesticated Near Eastern crops, such as peas, wheats, barley, oats, and chickpeas. The majority of the microfossils isolated from the teeth were starch grains; therefore, our interpretations will focus on the starch data.

To prepare modern starch grain mounts, the parts of the plant most likely to produce starch grains (e.g., the caryopsis or the pulse) were scraped with a scalpel over a microscope slide, hydrated, and examined using a Zeiss Axioskop 2 polarizing light microscope with attached AxioCam camera, linked to a PC running AxioVision software, or a Leitz Laborlux 12 Pol S light microscope with attached Polaroid Camera, linked to a PC running Polaroid DMC le software. Phytoliths were isolated from the leaves, stems and bracts of plants by standard techniques involving dissolution in concentrated nitric acid and potassium chlorate, followed by rinsing in water and mounting in Permount (Piperno, 2006). All reference microfosssils were photographed and their specific features were described.

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As has been described and illustrated in detail elsewhere (Piperno et al., 2004), the Near Eastern cereals *Hordeum* and *Triticum*, as well as related genera such as *Aegilops*, produce starch grains that can be distinguished in archaeological samples. The criteria for identification are based on features such as overall shape, surface ornamentations (e.g., presence of lamellae or crater-like depressions on the grains), and position of the hilum (the botanical center of the grain). Studies have not yet been carried out exploring whether starch grains from domesticated wheat and barley can be distinguished from those of their wild ancestors, but genus-level identification of these taxa appears to be secure (Fig. 2a–c). In addition, our analysis reveals that *Avena* spp. may produce identifiable starches (Fig. 2d). Likewise, studies of legume starches have shown that this family of plants produces diagnostic starch grains (Piperno, unpublished data). Legume starch grains are often large and oval with clear lamellae and a particularly distinctive longitudinal cleft fissure (Fig. 2e–h).

2. Results

Despite striking differences in the amount of calculus deposits, all of the teeth produced starch grains, some in large numbers (Table 2). As with other studies of human dental calculus (Lalueza Fox and Pérez-Pérez, 1994; Lalueza Fox et al., 1996; Juan-Tresserras et al., 1997; Scott Cummings and Magennis, 1997), we found very few phytoliths in our sample, with one tooth preserving none at all. The concentration of microfossils averaged about 30 starches/mm^2 and 1.5 phytoliths/mm^2 among the three teeth that had thick enough calculus to allow measurement of the removed area.

The soil contexts from which the teeth were recovered varied from tooth to tooth, but this did not appear to directly influence the numbers or types of microfossils recovered. For example, both RH1 and RH3 were found in ashy layers, but RH1 preserved the most microfossils and RH3 the fewest.

Some of the starch grains were identifiable to the family and genus-level. Most identified starches belong to the Poaceae or grass family, based on several diagnostic features. These include lenticular shape, central hilum, presence of evident lamellae, usually central fissures, and size (Fig. 3a). Most identified grass starch grains belong to the genus *Hordeum*, while several others appear to be from *Avena* (Fig. 3b, c, Table 2). Overall, however, grass seed starches make up a surprisingly small, if consistent, proportion of the total number of starch grains recovered, averaging about 12% with a range from 7% to 16%. Also interestingly, none of the recovered starch grains had traits diagnostic of legumes.

Most of the recovered starch grains are not yet identified; however, many possess features that likely will make them diagnostic as our modern reference collections are expanded. Many of the starch grains appear to derive from two different and distinctive types (Fig. 3d, e). Type one is a large grain (~15 μm), with a facetted, often five- to six-sided overall shape, no lamellae, a central hilum, and occasional fissures. Type two is smaller (8–10 μm), oval or reniform, with faint

Fig. 2. Starch grains from modern reference samples. The top row includes starch grains from cereals that show the stereotypical, lenticular forms with variably visible lamellae and surface craters. The bottom row includes only legumes, with the key characteristics of these starch grains highlighted, namely the distinctive longitudinal cleft fissure, lamellae and oval shape. (a) *Hordeum bulbosum* (bulbous barley), with faint lamellae and distinct oval outline; (b) *Hordeum vulgare* (domesticated barley), with lamellae and few but clear craters; (c) *Triticum dicoccoides* (wild emmer wheat) with no lamellae but several clear craters, particularly at the edge of the grain; (d) *Triticum aestivum* (hard red winter wheat) with many, deep craters, covering one half of starch grain; (e) *Lens culinaris* (red lentil), showing the distinctive lamellae and longitudinal cleft fissure of the legumes; (f) *Cicer arietinum* (chickpea), with clear lamellae and longitudinal cleft fissure; (g) *Pisum sativum* (green pea), the typical form; (h) *Pisum sativum* (green pea), with two alternate forms. Top starch grain is rotated so that longitudinal cleft fissure is perpendicular to the line of sight, and therefore hidden. The bottom starch grain shows a variant form of the fissure with extra side cracks. Scale bar is 25 μm.

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Table 2
Recovered microfossils extracted from dental calculus on teeth from the site of Tell al-Raqā'i, Syria

Tooth sample	Number of starches	Starch density (per mm^2)	Number of phytoliths	Phytolith density (per mm^2)	Identified microfossils
RH1	76	41	5	3	Three *Hordeum* starches, four possible *Avena* starches, five starches consistent with other grass seeds, two probable grass bract phytoliths
RH2	35	[a]	0	[a]	Two *Hordeum* starches, two possible *Avena* starches, two starches consistent with grass seeds
RH3	10	[a]	1	[a]	One *Hordeum* starch
RH4	27	12	1	0.5	Two starches consistent with grass seeds
RH5	53	31	2	1	Four *Hordeum* starches, one possible *Avena* starch, two starches consistent with other grass seeds, one grass bract phytolith

[a] Area of removed calculus could not be measured, so the density was not calculated.

lamellae, and a central and often-darkened hilum. These two forms are found on almost every tooth, and likely record the presence of two common food sources.

Several of the starches show signs of damage that are consistent with the processing of foods and exposure to heat, such as cracking, distortion, shattered surfaces, widened extinction crosses, and general swelling of the starch grains (Henry and Hudson, unpublished data; del Pilar Babot, 2003; Perry et al., 2006). Some have reduced or damaged extinction crosses when viewed under polarized light, which often occurs when starches are exposed to heat and water (Henry and Hudson, unpublished data) (Fig. 3f). One starch grain is clearly gelatinized, as would be expected from cooking (Lineback and Wongsrikasem, 1980; Samuel, 1996) (Fig. 3g). Other starch grains show cracking and other signs of mechanical damage, as results from milling or grinding of the seeds (Fig. 3h).

The ESEM images reveal that the teeth suffered no damage, even those teeth with moderate to small amounts of calculus. No new scratches or pits were created on the surface of these teeth by our sampling procedure (Fig. 4). This was even the case on tooth RH2, which had deposits of calculus invisible to the naked eye (Fig. 5). On the other teeth, which had relatively larger deposits, SEM images show that the sampling did not remove all of the calculus, leaving deposits for future research.

3. Discussion

The removal of dental calculus from archaeological teeth with a dental pick and subsequent processing has been shown to be an effective means to recover plant microfossils for study, while also preventing damage to the teeth. As with other studies of dental calculus that have attempted to recover both

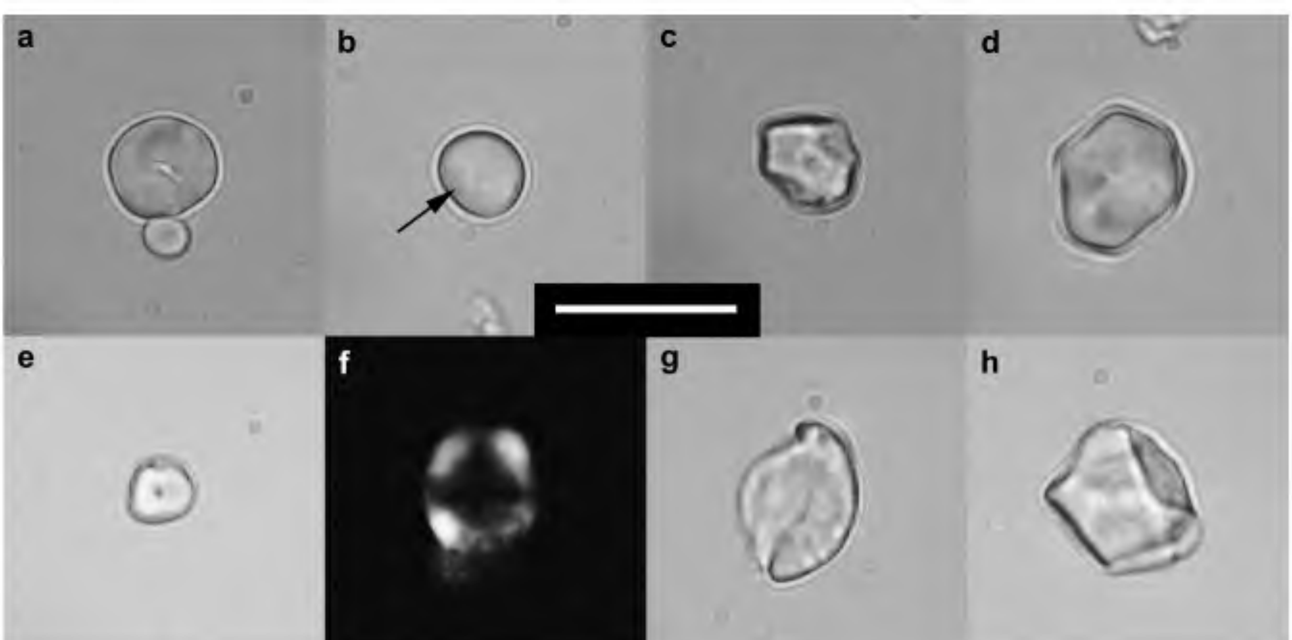

Fig. 3. Examples of starches recovered from the teeth. (a) A starch grain most likely from the caryopsis of a member of the *Poaceae*; (b) A starch grain identified as *Hordeum*, very similar to *H. bulbosum*. Arrows point to lamellae; (c) A starch grain that is consistent with some members of the genus *Avena*; (d) Unidentified but likely diagnostic starch type I, angular/facetted; (e) Unidentified but likely diagnostic starch type II; (f) Starch showing damaged extinction cross (wide center, fuzzy borders) consistent with damage from cooking; (g) fully gelatinized starch; (h) starch showing damage consistent with grinding or milling. Scale bar is 25 μm.

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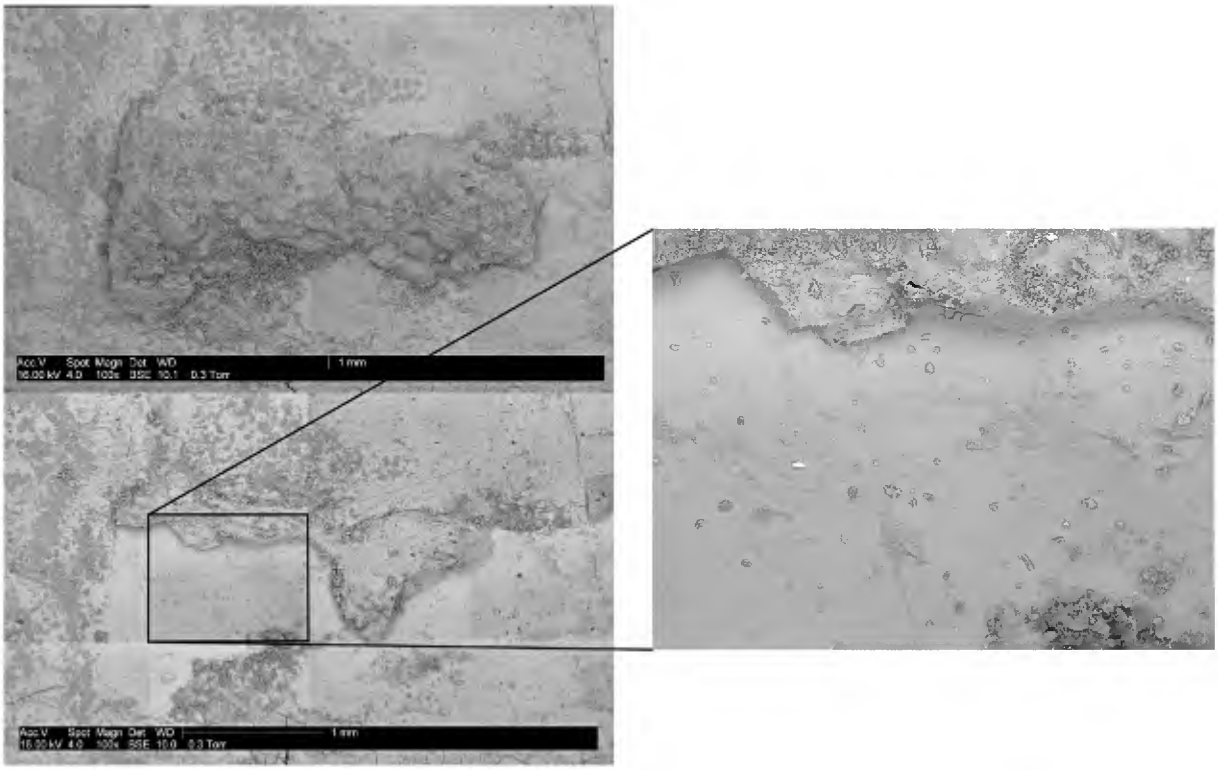

Fig. 4. Composite ESEM image of tooth RH1 before and after sampling. Inset shows closeup of one area that had calculus removed during sampling, which evidences no scratches or pits.

starch grains and phytoliths (e.g., Boyadjian et al., 2007; Lalueza Fox and Pérez-Pérez, 1994; Lalueza Fox et al., 1996; Juan-Tresserras et al., 1997; Scott Cummings and Magennis, 1997), we have found that human calculus contains more starches than phytoliths.

Some of the identified starches belong to grass seeds, including barley and, probably, wild relatives of barley, which is consistent with the archaeobotanical analysis that recovered many samples of these plants. However, not a single starch grain identifiable as wheat was recovered, and the low percentage of starches from grass seeds in all of the samples is similarly surprising, since it suggests that cereals made up only a small portion of the diet of these individuals. Both wheat and barley produce high concentrations of starch grains, so if they had been a major part of the diet of these individuals, the starches should have been easily visible in the teeth samples. The scarcity of starch grains from cereals in general, and wheat in particular, further emphasizes the antiquity of the teeth, as these foods are staples of modern diets. The overall lack of barley and wheat also seems to support the archaeological evidence that the site was a cereal storage and processing area, but not necessarily a place where the grains were consumed. The excavators have argued that the early inhabitants of Tell al-Raqā'i were engaged in a specialized economic activity of storing cereals for external, not local, consumption. We may be seeing evidence of a rural group relying on a variety of wild and cultivated foods for their own nourishment, while selling or otherwise parting with the high-quality foods (i.e. cereals) they grew. It is also possible that the cereals stored at Tell al-Raqā'i were intended as fodder for large flocks of sheep and goats raised for large-scale wool production (Zeder, 1998; McCorriston, 1998), or that the storage facilities at the site contained other materials besides cereals.

It is currently unclear what other plants made up the majority of diet for the individuals studied from Tell al-Raqā'i. It is possible that some of the unidentified starches are from plants such as *Carthamus*, *Malva* or *Phragmites*, remains of which were found at the site, but this is currently speculative because we do not yet have modern reference starch collections of these taxa. Safflower (*Carthamus*) was used historically as a dye, but the seeds are also a potent source of oil, and they may have been eaten. The common reed (*Phragmites*) has edible root, culm and seeds, and while no seeds were found in the archaeobotanical remains, some of the culm remains found at the site may have come from this plant (van Zeist, 2003). Mallow (*Malva*) has edible leaves and seeds as well as several medicinal uses, including as an astringent, diuretic, and salve. It is also possible that the starches came from other members of the Poaceae family that were not included in our reference collection, such as *Trachynia* or *Bromus*.

Several of the recovered starches provided evidence of damage consistent with cooking and processing. It is intriguing that so many cooked starches survived, since heat caused starch to gelatinize and there is evidence that cooking makes starches more susceptible to organic decay (Samuel, 2006), though others have shown that grains damaged from heat and other factors can survive for a long time on archaeological artifacts (Barton, 2007). It is likely that cooking would also

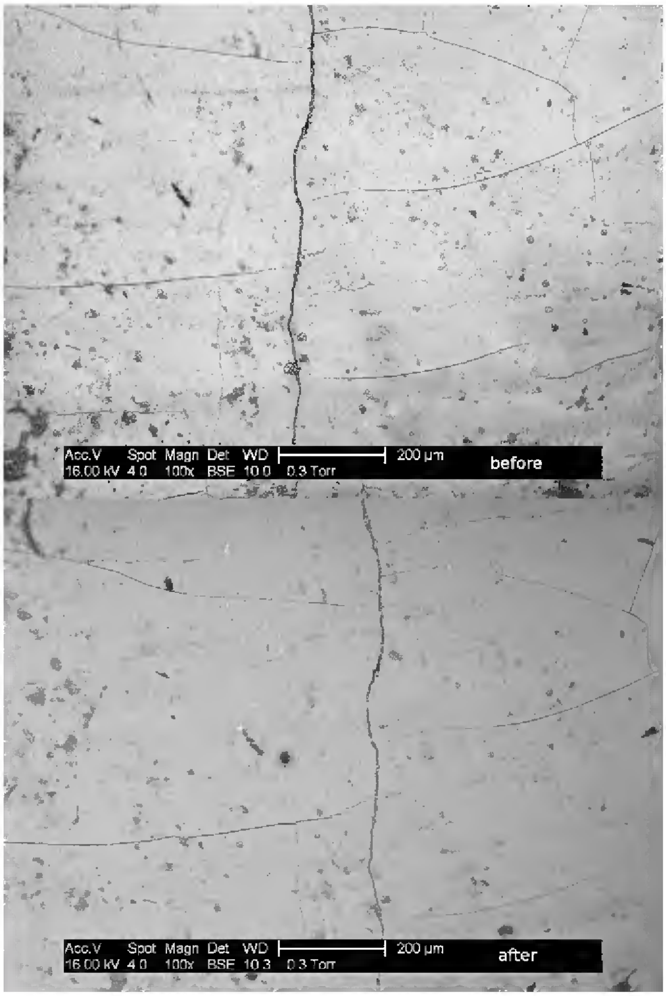

Fig. 5. Single ESEM images of the same area of tooth RH2, which had no visible calculus but produced several starches, while acquiring no damage from the processing.

make the starches more susceptible to enzymatic digestion in the mouth, but more research into the relationship between cooking and starch digestibility needs to be done. The speed with which dental calculus forms, along with its protective covering may have helped to preserve these starches.

In summary, we have shown that the sampling of dental calculus with a dental pick is a robust method for recovering plant microfossils, even from teeth with no visible calculus concretions. Furthermore, this method does not damage the teeth, making it useful for samples for which destructive sampling is problematic. The starches recovered in this study dating to roughly 4700 bp (third millennium BC), are the oldest yet reported from dental calculus from the Old World. We expect that it will be possible to use these methods on much older teeth.

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