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Fermented beverage and food storage in 13,000 y-old stone mortars at Raqefet Cave, Israel: Investigating Natufian ritual feasting

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ABSTRACT

Fermented and alcoholic beverages played a pivotal role in feastings and social events in past agricultural and urban societies across the globe, but the origins of the sophisticated relevant technologies remain elusive. It has long been speculated that the thirst for beer may have been the stimulus behind cereal domestication, which led to a major social-technological change in human history; but this hypothesis has been highly controversial. We report here of the earliest archaeological evidence for cereal-based beer brewing by a semi-sedentary, foraging people. The current project incorporates experimental study, contextual examination, and use-wear and residue analyses of three stone mortars from a Natufian burial site at Raqefet Cave, Israel (13,700–11,700 cal. BP). The results of the analyses indicate that the Natufians exploited at least seven plant taxa, including wheat or barley, oat, legumes and bast fibers (including flax). They packed plant-foods, including malted wheat/barley, in fiber-made containers and stored them in boulder mortars. They used bedrock mortars for pounding and cooking plant-foods, including brewing wheat/barley-based beer likely served in ritual feasts ca. 13,000 years ago. These innovations predated the appearance of domesticated cereals by several millennia in the Near East.

1. Introduction

The consumption of fermented and alcoholic beverages is one of the most prevalent human behaviors, but the time and cultural context of its origins remain unclear. Archaeological evidence for alcohol production and use is usually associated with fermenting domesticated species in agricultural societies, such as ancient Egypt, Mesopotamia, China, and South America (Goldstein, 2001; Jennings, et al., 2005; Katz and Voigt, 1986; McGovern, et al., 2004; Samuel, 1996; Wang, et al., 2016). It has long been speculated that humans' thirst for beer may have been the stimulus behind cereal domestication (Braidwood, et al., 1953), and some scholars have attributed this invention to the Natufians in the Near East (ca. 15,000–11,500 Cal BP) (see Hayden, et al., 2013). The Natufians were innovative in many material and social realms, and paved the way to the establishment of the first sedentary Neolithic villages at about 11,500 cal BP (Bar-Yosef, 1998). To test the so far unsubstantiated "Natufian beer hypothesis" we examined three stone mortars from the first chamber at Raqefet Cave (13,700–11,700 cal. BP), a site with a long archaeological sequence (Lengyel, 2007), that also served as a Natufian burial place in Mt. Carmel, Israel (Fig. 1A; Fig. S1A) (Nadel, et al., 2013).

Excavations at Raqefet Cave between 2004 and 2011 revealed a radio-metrically dated Natufian graveyard with ca. 30 burials (Barzilai, et al., 2017; Lengyel, et al., 2013). Clear indications for burial-associated rituals include repetitive interments, floral grave lining in some of the burials (Nadel, et al., 2013), and animal bones in the graves representing punctuated funerary feasts (Yeshurun, et al., 2013). About 100 bedrock features (e.g. mortars and cupmarks hewn in the cave floor and the terrace) were found (Nadel and Lengyel, 2009), some directly associated with burials. Two deep narrow boulder mortars (BM1 and BM2) were found in situ and juxtaposed to human remains. Thus, they were the focus of our contextual, use-wear and residue analyses. BM1 has a cylindrical shaft ca. 33 cm deep and a hole at the base; it is stored in the Laboratory for Ground Stone Tools Research, Zinman Institute of

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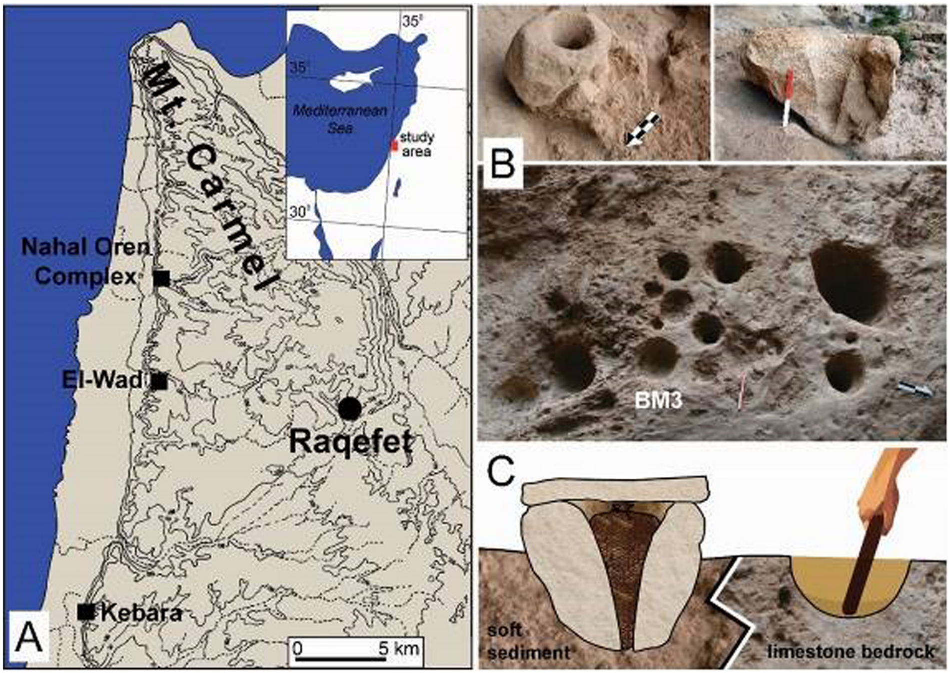

Fig. 1. Site location and artifacts analyzed. (A) The location of Raqefet Cave and three additional Natufian sites in Mt. Carmel; (B) Field photos of the studied boulder mortars (BM1,2) and the location of BM3 on the cave floor (scale bar and arrow: 20 cm); (C) a functional reconstruction of the mortars: a boulder mortar used to store plants in a basket with a stone slab on top, and a bedrock mortar used for pounding and cooking plants and brewing beer.

Archaeology, University of Haifa after the excavation in 2006. BM2, a funnel-shaped shaft ca. 35 cm deep, has remained inside Raqefet cave. A well-preserved bowl-like bedrock mortar (BM3), 18 cm deep and 27 cm in rim diameter, located in a cluster of bedrock features in the middle of the cave floor was included in the current study (Fig. 1B; Fig. S1B,C).

2. Methods

We collected and examined microbotanical residues and use traces on the mortars in 2015. To test possible post-depositional contaminations in the residue samples, we obtained control sediment samples from various archaeological and natural contexts (see below).

2.1. Residue sample collections and process

The residue samples were collected by employing spot extraction method described in previous publications (Loy and Fullagar, 2006) with modification, as follows:

(1) Surface sediments cleaning: Each mortar was first cleaned with a clean toothbrush to remove the dusts on the surface. BM1 and BM3 were covered with sediments; we cleaned the sediments by applying distilled water on the tool surface, waited for a minute, extracted the water with a pipette, and transferred it into test tubes which were marked as Wash 1 (W1) (Fig. S2).

(2) Ultrasonic cleaning: A small amount of distilled water was dropped on the same location of W1, an ultrasonic tooth brush was used to clean the vessel's surface for 6 min (Fig. S2); the water was extracted and transferred to test tubes, marked as Wash 2 (W2). BM2 had a rather clean surface, and was subjected only to the ultrasonic cleaning. It was hypothesized that W1 samples may include residues from the soil matrixes, while W2 samples are more directly related to the tool function. However, when the results from the two washes are compared, both contain considerable amounts of residues, although those from W1 are outnumbered by W2. No starch was found in the control samples on the outside wall and near the tools (see below), indicating that the starches from W1 and W2 residue samples are mostly related to the function of the tools; thus we analyzed all starch granules recovered as one assemblage.

(3) Microfossil Extraction: Residue samples were processed with standard protocols established in the Stanford Archaeology Center, which involve two procedures. (1) EDTA dispersion; after centrifuge and decanted supernatant, 4 ml of 0.1% EDTA ($Na_2EDTA{\cdot}2H_2O$) solution was added to each tube (15 ml). The tubes were placed in an automatic shaker for 2 h to disperse the sediments, then filled with distilled water and centrifuged for 5 min at 1500 rpm, and the supernatant was decanted. (2) Heavy liquid separation; 4 ml of SPT (sodium polytungstate) at a specific gravity of 2.35 was added to each tube. The tubes were then centrifuged for 15 min at 1000 rpm. The top 1–2 mm layer of organics was carefully removed from each tube by a new pipette and then transferred into a new 15 ml tube. The samples were topped off with distilled water and centrifuged for 5 min at 1500 rpm to concentrate the starch and phytoliths at the bottom of the tube, and the supernatant was decanted. The rinse was repeated two more times to remove any remaining SPT.

(4) Microscopic analysis: Extractions obtained from residue samples were mounted in 50% (vol/vol) glycerol and 50% (vol/vol) distilled water on glass slides and scanned under a Zeiss Axio Scope A1 fitted with polarizing filters and differential interference contrast (DIC) optics, at 200× and 400× for both starch and phytoliths. Photographs were taken using a Zeiss Axiocam HRc3 digital camera and Zeiss Axiovision software version 4.8.

2.2. Microfossil identifications

Starch identifications were based on our modern reference data, including more than 130 species in 23 families collected from many parts of the World, including Israel. We particularly recorded starch morphologies from relevant species under native, ground, cooked, malted, and fermented conditions (Wang, et al., 2017). We also consulted published information for phytolith and starch identification (Madella, et al., 2005; Nadel, et al., 2012; Piperno, 2006; Piperno, et al., 2004). Congo red staining was used to identify the presence of damaged and gelatinized starches (Lamb and Loy, 2005). Fiber identifications were based on published information (e.g., Goodway, 1987; Luniak, 1953; Shimony, 1995; Wang and Wang, 2005), as well as our reference collection, which includes flax (*Linum* sp.), hemp (*Cannabis* sp.), jute (*Corchorus* sp.), cotton (*Gossypium* sp.), and sheep wool.

2.3. Use-wear sample collection

After residue samples were obtained, use-wear traces were collected from stone mortars by applying dental impressions (Polyvinyl siloxane, or peels). Multiple samples were taken from various locations on each tool (Fig. S2). Use-wear traces were interpreted based on our experimental data reference (Fullagar, et al., 2012; Liu, et al., 2013) and experimental study carried out in this project. The peels from Natufian artifacts and experimental tools were examined under a compound Zeiss microscope (Imager.A2m) at magnifications of 50×, 100×, 200×, and 500×. Photographs were taken using a Zeiss Axiocam HRc3 digital camera and Zeiss Axiovision software version 4.8.

2.4. Control samples

Researchers around the world have demonstrated that starch granules on stone artifacts have survived for many millennia (Barton and Torrence, 2015; Torrence and Barton, 2006); however, contamination of modern starch in ancient samples, either from lab equipment or from post-depositional contexts, has been a major concern in residue analyses (Crowther, et al., 2014; Ma, et al., 2017; Mercader, et al., 2017; Torrence and Barton, 2006). We are fully aware of this problem, and have regularly cleaned and tested our lab facilities for contamination control. We also separate our lab facilities for processing ancient samples from those for modern samples to avoid cross contamination. The results of testing show that modern fibers or starch granules on furniture and equipment in our lab are minimum.

In order to test for possible contamination in post-depositional contexts, we analyzed six control samples from unused surface of BM1 and various contexts inside and outside Raqefet Cave for the presence of microfossils. These include two sediment samples from the exterior surface on the wall and base of BM1 (BM1-S1 and S2); two soil samples (C4, C5), 40 m and 100 m northwest of the cave, respectively; one sediment sample from the cave floor (C2); and one sediment sample from underneath a human skeleton (C1). The control samples were processed with the same method as for the residue samples.

2.5. Comparison between residues and control samples

The results show that residue samples extracted from three mortars contain together considerable amounts of starch granules (n = 121), phytoliths (n = 232), and fibers (n = 107). In contrast, the control samples revealed extremely low numbers of starches and fibers with very different compositions. Starch is present in two soil samples (C4 and C5) collected outside the cave (one granule from each sample, severely damaged, possibly Triticeae), but absent in the samples from the BM1 exterior surface and the cave floor. A total of 12 fibers were found in six control samples, including bast (n = 1), animal hair (n = 3), cotton (n = 5), synthetic (n = 1), and UNID (n = 2). Phytoliths from grass husk and inflorescence were found in the mortar residues but were absent in all control samples (Tables 1–3). The results suggest that the microfossils in the residue samples are mostly related to the tool use rather than post-depositional contamination.

3. Beer-brewing materiality and experimental studies

Ancient brewing processes had great regional variations (Hayden, et al., 2013; Jennings, et al., 2005; Samuel, 1996), but would have involved three basic stages. (1) Malting: grains are germinated in water, drained, dried, and then stored for use. (2) Mashing: the malt is coarsely ground or crushed in a container, mixed with water, and heated until the temperature reached to and maintained in the range of 65 °C -70 °C over a period of time around 30 min to 4 h. (3) Fermenting: yeast is added to, or naturally settled on the wort, the brewing container is covered with a lid, and the wort is allowed to sit for one or more days until the fermentation is completed (Hayden, et al., 2013). To successfully complete a brewing process, the use of certain types of materials and tools is necessary, including malted cereals, storage facilities, plant processing/grinding tools, and cooking/fermentation utensils. To test the hypothesis of Natufian beer brewing, it is crucial to identify the traces of cereal fermentation and associated material remains in the archaeological record.

We conducted experimental studies to provide comparative references for use-wear and residue analyses in order to investigate the functions of the mortars in relation to food processing and beer brewing. These include (1) processing different materials on stone objects to analyze use-wear patterns, and (2) brewing beer with various grain species to observe morphological changes on starch granules.

3.1. Experimental use-wear study

We have conducted a series of experiments in recent years, using various stone tools to work on different objects, including soil, stone, and plants, and created a database of comparative samples (Fullagar, et al., 2012; Liu, et al., 2017). The Reqefet Cave mortars are made of limestone with hardness 4 on the Mohs scale. Therefore, we used limestone slabs (tested hardness 4 on the Mohs scale) to process several materials.

It has long been suggested that Natufian mortars were used for processing (e.g., pounding) plant foods (Dubreuil, 2004; Dubreuil, 2008; Garrod, 1957; Wright, 1994). Our residue analyses revealed not only starches, but also abundant bast fibers, suggesting that some mortars may have been in contact with fibers or textiles. Accordingly, we processed braid fabric made of flax, bag made of jute, basket made of sweetgrass (*Muhlenbergia filipes*), cattail leaves (*Typha* sp.), wood (oak), and limestone. We particularly paid attention to the forms of striations and polish. Each material was abraded on the limestone slab for 10–15 min, and the dental impression peels were applied on the worked area of the slab to obtain the use traces.

The use traces from abrading limestone-on-flax fabric are characterized by long and very fine striations, while those traces from working on jute, sweetgrass, and cattail leaves show long, but notably

Table 1
Raqefet Cave mortars starch counts and size range.

Taxa	Triticeae	Panicoideae	*Avena*	Fabaceae	*Cyperus*	*Lilium*	USOs	UNID	Total	Malting damage	Fermentation damage	Other damage	Damaged starch total
BM1	5	4	15				3	9	36	3			**3**
BM2	2	7			8	1		5	23	2			**2**
BM3	6	3		4	25		8	16	62		19	23	**42**
Total N.	13	14	15	4	33	1	11	30	121	5	19	23	**47**
Total %	10.0	11.7	12.5	3.3	27.5	0.8	9.2	25.0	100.0	4.1	15.7	19.0	**38.8**
Min. size (μm)	15.31	12.32	6.31	15.53	7.18	22.93	12						
Max. size (μm)	41.08	20.46	15.23	33.48	16.46	22.93	50.71						
Mean (μm)	23.28	16.57	11.21	27.24	11.79	22.93	24.79						

Table 2
Raqefet Cave phytolith record.

Phytolith morphotypes	Taxonomic attribution	BM1	BM2	BM3	C1	C2	C4	C5	BM1-S1	BM1-S2
		Residue samples			Control samples					
Silica skeletons										
Opaque perforated platelets										
Undetermined multi-cell						1				
Single-cell phytolith										
Phragmites bulliform	*Phragmites* leaf	2								
Bilobate	Poaceae	1				3				
Polylobate	Poaceae			1		1				
Cross/quadra-lobate	Poaceae			1						
Saddle	Poaceae			3						
Rondel	Poaceae	7								
Common bulliform	Poaceae	26						1	1	2
Elongate dendriform/echinate/crenate/columellate	Grass seed husk/inflorescence	12	8							
Elongate psilate/sinuate	Grass leaf/culm	77	4		5	60	4	2	8	35
Arcicular hair	Monocots	2	1						1	2
Rectangle	Poaceae	85		2		5				3
Total		212	13	7	5	70	4	3	10	42

Table 3
Fibers found in the Raqefet Cave mortar residue and control samples.

	Bast	Animal hair	Cotton	Synthetic	UNID	Total
Residue samples						
BM1	16	4	0	0	20	40
BM2	23	2	0	0	13	38
BM3	15	0	1	1	12	29
Total	54	6	1	1	45	107
%	50.5	5.6	0.9	0.9	42.1	100.0
Control samples						
RAQ-C1					1	1
RAQ-C2		3			1	4
RAQ-C4	1					1
RAQ-C5			2	1		3
BM1-S1			1			1
BM1-S2			2			2
Total	1	3	5	1	2	12
%	8.3	25.0	41.7	8.3	16.7	100.0

wider and deeper striations. These differences are apparently caused by variation in softness and size of the plant fibers, as flax is softer and thinner than others. Abrading wood produced large areas of polish with long, wide striations, and the polished areas show rounded edges. Limestone-on-limestone abrading produced flat polished areas with long, wide, and deep furrow striations (Fig. 2).

3.2. Morphological characteristics of damaged starch granules from beer making

During the processes of cereal-based beer brewing (malting, mashing, and fermentation), enzymatic digestion and gelatinization cause starch modification in certain patterns. Previous studies have published detailed descriptions on the morphological changes of starches affected by these processes (Babot, 2003; Bowler, et al., 1980; Claver, et al., 2010; Dronzek, et al., 1972; Evers, et al., 1971; Galliard and Bowler, 1987; Henry, et al., 2009; Lineback and Ponpipom, 1977), which are summarized below (for a detailed discussion see Wang, et al., 2017).

In principle, the transformation from starch to beer is a two-phase conversion. During the first phase, starches are broken down into fermentable sugars, a process known as saccharification. This process can be initiated by the enzymes naturally present in cereal grains during germination, or by exogenous enzymes in human saliva and other natural sources such as honey and various plants. In our experiments, starch granules from sprouted barley show four types of damage, representing several continuous phases of enzymatic action. The damage types include random pits on the granule surface, missing areas in the shape of concentric rings, a hollowed center, and a completely digested interior (Fig. S3b–e) (Wang, et al., 2017).

The second phase of beer making involves mashing and fermentation, during which yeasts convert sugars into alcohol and carbon dioxide. Mashing and fermentation create conditions with two important effects for starch modification. The first is low-temperature gelatinization, which involves uncoiling and melting of chains in crystalline regions, making those relatively resistant regions more susceptible to enzymatic attacks (Colonna, et al., 1987). The second is enzymatic hydrolysis, during which enzymes produced from germinated grains take a further step in starch degradation. The cumulative effects of gelatinization and enzymatic hydrolysis produce a distinctive damage type on cereal grains. For wheat and barley, the typical fermented starch is a "collapsed" granule with a depressed or hollowed center and a much less attacked peripheral region, giving an appearance similar to a pizza crust. Under polarized light, the granule exhibits a darkened center without extinction cross, and a ring of light on the outer edge (Fig. S3f). Due to the low temperature gelatinization, some granules also show swelling and distortion (Wang, et al., 2017).

Heating ungerminated seeds in warm water produces a moderate level of starch gelatinization, characterized by slight swelling and more pronounced lamellae (Fig. S3g,h). Different from fermentation, starch granules resulting from this process appear to expand evenly (2D for wheat/barley), without visible central depression or holes as those present in the fermented starches. Therefore, the brewing process causes visible diagnostic alternations to some starch granules. They do not resemble the morphological changes resulting from non-fermentation processes, according to our experiments (Wang, et al., 2017) and other published works (Henry, et al., 2009). These morphological characteristics are used to identify starch changes caused by malting and fermentation in the Raqefet assemblage.

4. Results

4.1. Starch types

Among the 121 starch granules recovered from the Raqefet Cave residues, six types, corresponding to certain taxa, were identified (Table 1; Fig. 3).

Type I, Triticeae starches (n = 13; 10% of the total), are lenticular in shape, with faint lamellae and occasionally scattered craters. The size range is 15.31–41.08 µm. Triticeae starch granules possess a bimodal size distribution, involving the presence of both large granules oval to

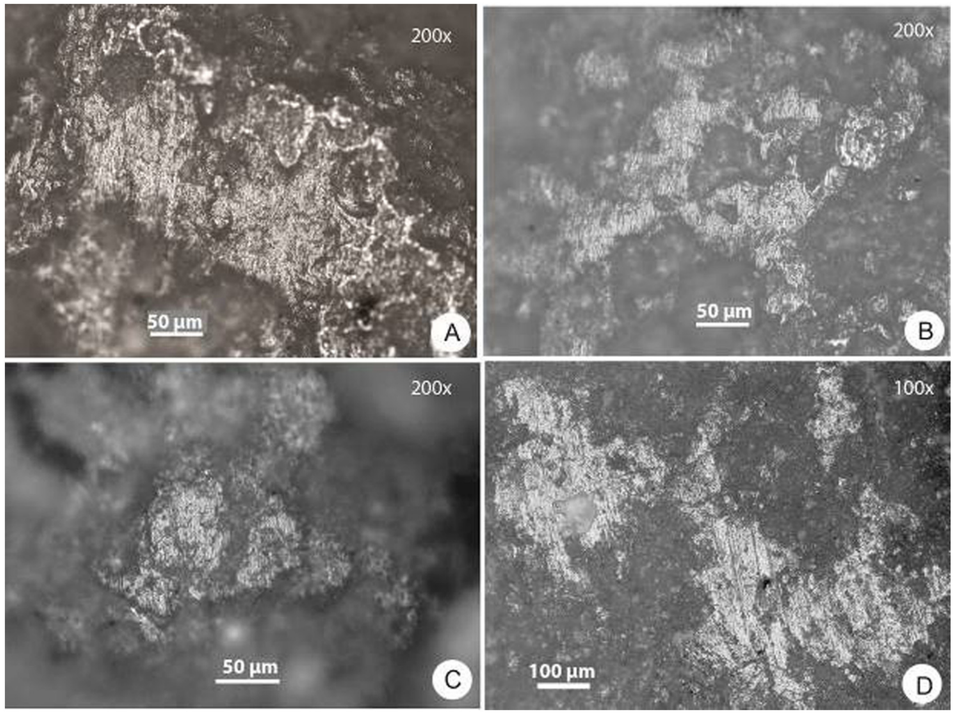

Fig. 2. Use-wear traces on experimental tools.
(A) Flax fabric abraded on limestone for 15 min; (B) jute fabric abraded on limestone for 15 min; (C) oak wood abraded on limestone for 15 min; (D) limestone-on-limestone abrading for 10 min.

sub-round in 2D shape, lenticular in 3D (A-type) and small almost spherical granules with a central hilum (B-type). Type I starch granules in our samples are of the A-type. They occur on all mortars and are likely from wild wheat and/or barley.

Type II, Panicoideae starches (n = 14; 11.7% of the total), are polygonal in shape, with centric or nearly centric hila, linear or Y-shaped radiating fissures, and nearly "+" shaped extinction crosses. The size range is 12.32–20.46 µm. It is unclear, however, what taxa under Panicoideae these starch granules may belong to.

Type III, *Avena* spp. starches (n = 15; 12.5% of the total), are sub-round, oval, and polygonal in shape, with centric hila, "+" shaped extinction crosses, and no visible lamellae. The size range is 6.31–15.23 µm. They were found only in BM1, including individual granules and a cluster of granules with various forms. Such a combination of diverse starch forms in one species resembles oats (*Avena* spp.), based on our reference data and finds from the 23,000-years-old site of Ohalo II (Nadel, et al., 2012).

Type IV, Fabaceae starches (n = 4; 3.3% of the total), are only present in MB3. They are oval in shape, with clear lamellae and extinction crosses showing a dark area in the centers, and the size range is 15.53–33.48 µm. These are typical characteristics of legume. At least two species of wild pulses (wild lentil *Lens orientalis* and wild races of pea *Pisum sativum*) are native to the Levant (Weiss and Zohary, 2011), but we are unable to determine what species these starches may belong to.

Type V, *Cyperus* sp. starches (n = 33; 27.5% of the total), are sub-round and oval in form, with centric hila, blurry extinction crosses (possibly due to damage), and no visible lamellae. The size range is 7.18–15.21 µm. They were found in BM2 and BM3, and particularly abundant in BM3. The granules are similar to *Cyperus rotundus* in form and size (1.90–19.15 µm) based on our reference collection. A previous study has identified multi-cell phytoliths from sedge leaves and husks at Raqefet (Power, et al., 2014), and macro-botanical remains of *Cyperus rotundus* tubers have been found at the Natufian site of Shubayqu1 in Jordan (Pedersen, et al., 2016).

Type VI, *Lilium* sp. starch (n = 1; 0.8% of the total), is 22.93 µm in size and elongate ovoid in shape, with extremely eccentric extinction cross and visible lamellae. This starch was found in BM2. One lily species (*Lilium candidum*) is native to Israel. The plant has assumed great symbolic, decorative and medicinal value in the region since antiquity due to its large white flower (Lev and Amar, 2000; Zaccai, et al., 2009).

Eleven granules (9.2% of the total) found in MB1 and MB3 are classified as underground storage organs (USOs), which cannot be identified to specific taxa based on our current reference data. They are oval, elongate, or nearly triangular in shape with extremely eccentric hila. The size range is 12.00–50.71 µm. Thirty starch granules (25.0% of the total) are unidentifiable (UNID), as they lack diagnostic features based on our reference data.

Many starch granules appear damaged (n = 47; 38.8% of the total), and can be classified into three categories based on our food processing experiments. (1) Malting damage (n = 5; from BM1 and BM2), which are characterized by the hollowed-out structure and/or pitted surface on starch granules (Fig. 4A–D); (2) fermentation damage (n = 19; 15.7%; from BM3), indicated by the hollowed-out and swollen appearance, with their peripheral areas relatively intact (Fig. 4E–L); and (3) other damage (n = 23; 19.0%; from BM3), characterized by deep fissures, faint or lack of extinction cross, or missing a part of the granule (Fig. 3E,G), which may have been caused by a number of actions, such as grinding, pounding, and enzyme attacks (Table 1) (see below for more details).

4.2. Phytolith types

The phytolith assemblage contains morphotypes mostly derived from grass inflorescence and culm/leaf, including twenty dendritic forms most probably from wild wheat/barley husks. Two phragmites bulliforms were found in BM1, possibly related to the use of reed-made objects. Raphides, which exist in many species including tubers (Crowther, 2009), are present in all samples (Table 2; Fig. 5). It is

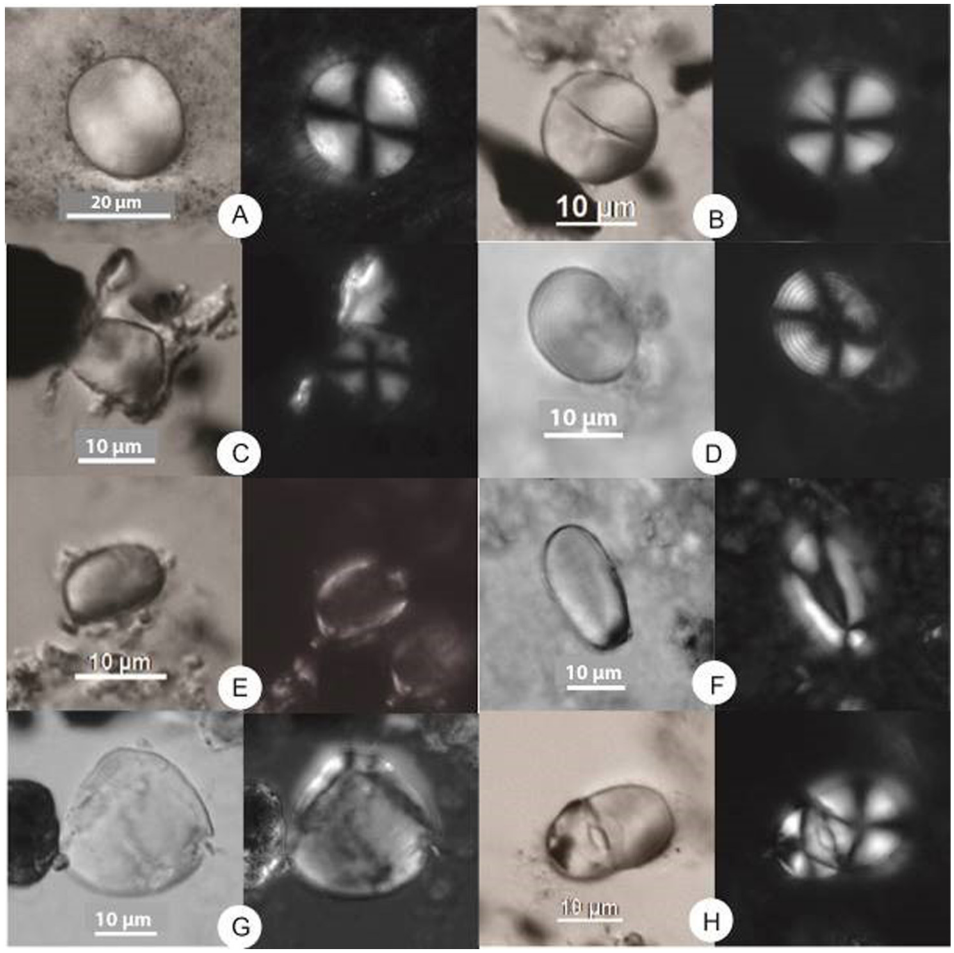

Fig. 3. Starch types from Raqefet Cave mortars.
(A) Type I, Triticeae; (B) Type II, Panicoideae; (C) Type III, *Avena* sp.; (D) Type IV, Fabaceae; (E) Type V, *Cyperus* sp.; (F) Type VI, *lilium* sp.; (G) unidentified USO; (H) unidentified starch (Each starch granule shown in DIC and polarized views).

possible that some of these raphides belong to tubers, but their exact taxonomic affiliations are unclear in this assemblage.

4.3. Fiber types

Large numbers of fibers (n = 107) were found in the residue samples, and can be classified into four groups: bast fiber, animal hair, cotton, and synthetic fiber, of which bast fibers are predominant (n = 54; 50.5% of the total) with very low frequencies of the other three groups (n = 1–6; 0.9–5.6% of the total). Many fibers (n = 45; 42.1%) do not show diagnostic features, and are classified as UNID (unidentifiable) (Table 3).

Bast fiber is plant fiber collected from the phloem or bast surrounding the stem of certain dicotyledonous plants. They are identified by their segmented structure with transverse dislocation (nodes) (Bergfjord and Holst, 2010; Goodway, 1987). The most common bast fibers in the ancient Near East include flax, nettle (*Urtico dioica*), hemp, and Jute, of which flax was the earliest one documented in the archaeological record, dating to the Late Natufian period at Tell Abu Hureyra (Hillman, 2000). Flax is considered as a native plant and the first fiber and/oil crop among the Neolithic "founder crop assemblage" in the Near East (Zohary, et al., 2012), while other bast fiber plants occurred much later in the archaeological record, and it is unclear if they were native to the Near East in antiquity (Strand, 2012). It is not easy to separate different types of bast fibers on the basis of their morphological features, and the characteristic traits used for identification have been controversial (Bergfjord and Holst, 2010; Bergfjord, et al., 2010; Haugan and Holst, 2014; Kvavadze, et al., 2009; Kvavadze, et al., 2010a). Nevertheless, many of the bast fibers in our samples are consistent with typical flax, characterized by small cross-section diameter, evenly distributed x-nodes, and straight extremities (Haugan and Holst, 2014; Kvavadze, et al., 2010b). It is likely, therefore, that some of these bast fibers from the Raqefet Cave stone mortars are flax (Fig. 6A,B).

Animal hairs are characterized by a round cross-section and scaled surface patterns (Goodway, 1987; Teerink, 1991). Six animal hairs were found in BM1 and BM2, and three from a control sample (RAQ−C2, soil from the cave floor) (Fig. 6C). People may have unitized animal hair since the Paleolithic, exemplified by the presence of colored and twisted tur hairs at Dzudzuana Cave in Georgia (ca. 30,000 cal BP) (Kvavadze, et al., 2009). Therefore, the BM1 and BM2 hair remains may have been components of Natufian paraphernalia. This hypothesis, however, needs to be tested in future studies.

Cotton fibers are characterized by the ribbon-like form and flattened medulla, some are twisted spirally or randomly (either clockwise or counter-clockwise), edges are thickened, and surface is often smooth with some showing oblique network (Goodway, 1987; Kvavadze, et al., 2010b; Matthews, 1904). The world-oldest cotton fiber remains have been found on a copper bead from Mehrgarh, Indus Valley, dating to the 6th millennium BC (Moulherat, et al., 2002). The earliest examples of cotton in the Levant were found at Dhuweila in eastern Jordan, dated to the Chalcolithic or Early Bronze Age (4450–3000 BCE) (Betts, et al., 1994). Therefore, the presence of cotton at Raqefet would be of modern origins. Only one cotton fiber (0.9% of the total) was found in a Raqefet

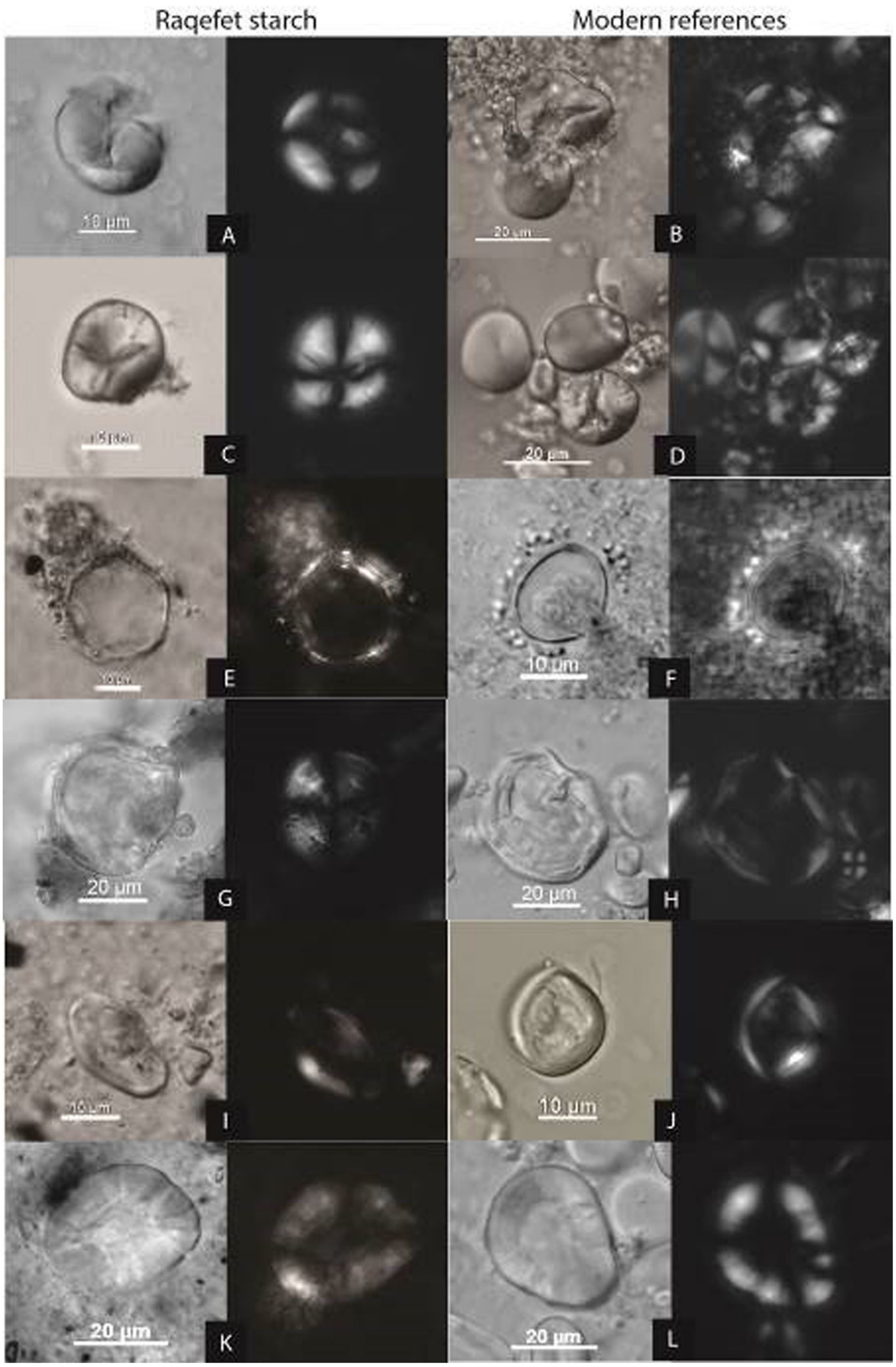

Fig. 4. Damaged and gelatinized starches from Raqefet Cave compared with modern reference. (A) Type I starch with deep fissures, hollowed center, and broken body (BM1); (B) modern malt barley starch comparable with (A); (C) Type I starch with deep fissures, pitting, and central depression (BM2); (D) modern malt barley starch comparable with (C); (E,G,I) Type I starch with signs of fermentation/gelatinization, hollowed center, swollen and distortion, disappearance of extinction cross (BM3); (F,H,J) modern fermented barley starches comparable with (E,G,I); (K) fermented/gelatinized legume starch (BM3); (L) modern fermented legume starch comparable with (K).

Cave residue sample (BM3), while five (35.7% of the total) were found in the control samples (three from BM1 exterior surface and two from soil samples outside the cave), suggesting a very low level of post-depositional contamination in our ancient residues.

Synthetic fibers, such as polyester and nylon, are structureless, with uniform diameter and rod-like appearance, which are of modern origin. One synthetic fiber was found in BM3 (0.9% of the total), similar to cotton, suggesting a very low level of post-depositional contamination.

Given the sharp contrast in the numbers of bast fibers between the ancient and control samples, and the very low frequencies of modern fibers in ancient samples, we are confident that the bast fibers, including flax, are associated with the function of Natufian mortars.

4.4. Use-wear traces

Fifteen use-wear samples from two boulder mortars (ten from BM1 and five from BM2) and three from the bedrock mortar were examined (Fig. S2). The polish and striation patterns on the boulder mortars are alike, but differ noticeably from those on the bedrock mortar, suggesting functional difference between the mortar types (Fig. 7). The details of use-wear patterns on each tool are discussed together with residue analyses in the next section.

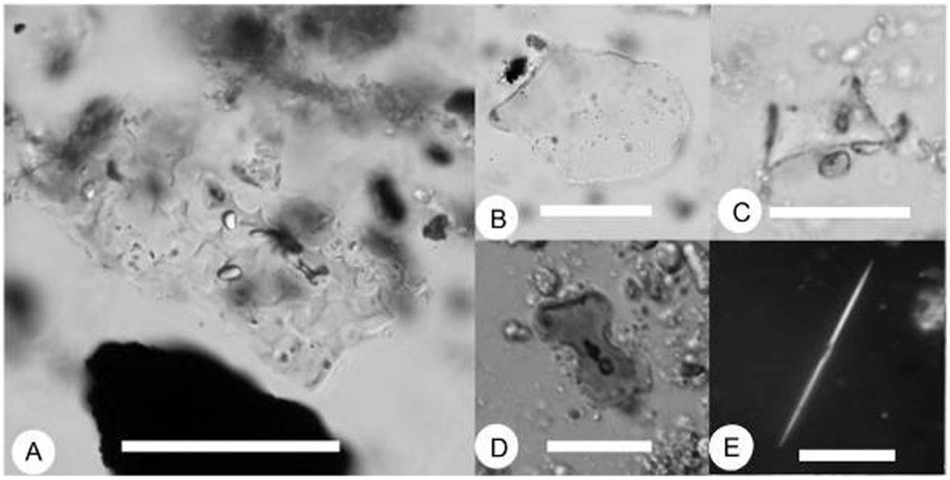

Fig. 5. Phytoliths recovered from Raqefet Cave mortars.
(A) Dendritic epidermal phytoliths; (B) *Phragmites* bulliform; (C) Rondel; (D) Bilobate; (E) Raphide (Scale bars: 50 μm in a; 20 μm in others).

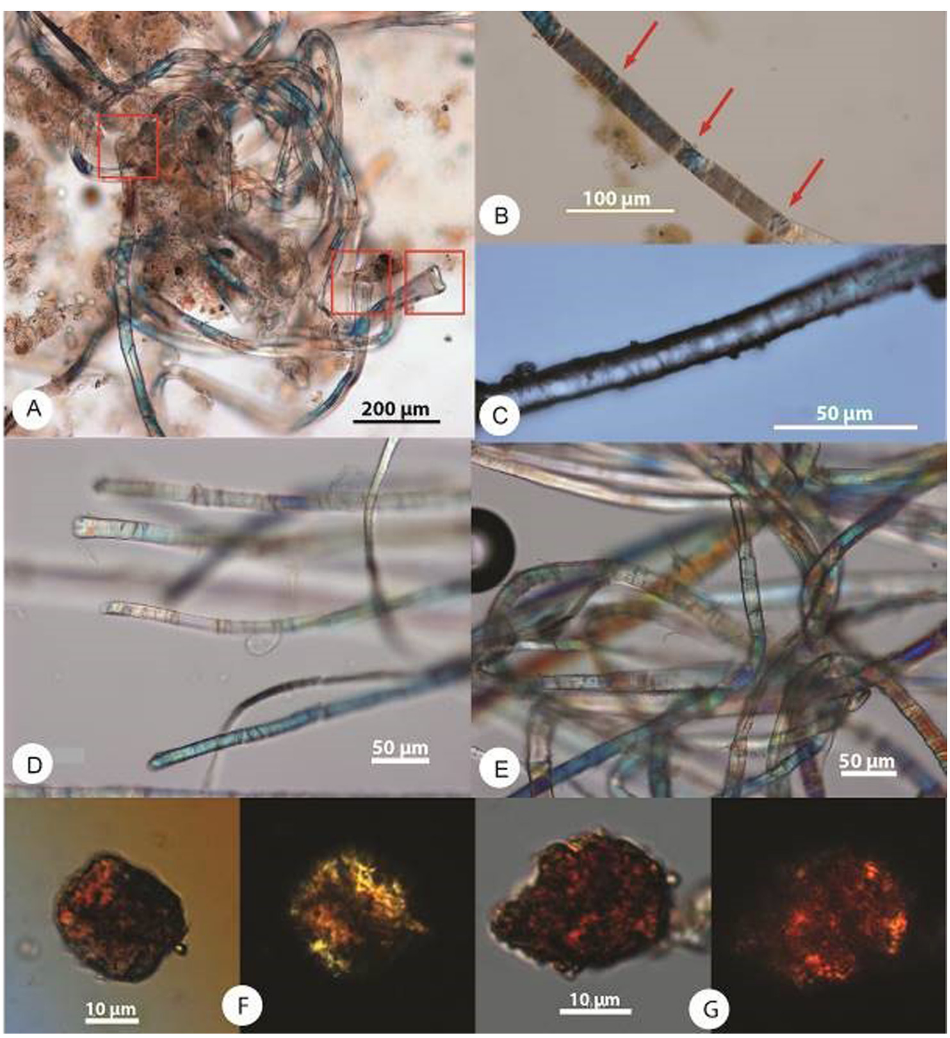

Fig. 6. Flax fibers, animal hair, and gelatinized starch granules stained with Congo red.
(A) Long and twisted flax fibers (BM2) showing straight ends, highlighted by red boxes; (B) flax fiber (BM1) showing segmented structure, arrows pointing to x-node feature; (C) animal hair; (D) modern flax fibers showing straight extremities and segmented structure; (E) modern braid flax fabric after abrading, showing twisting and entanglements; (F,G) gelatinized starches stained with Congo red (BM3). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

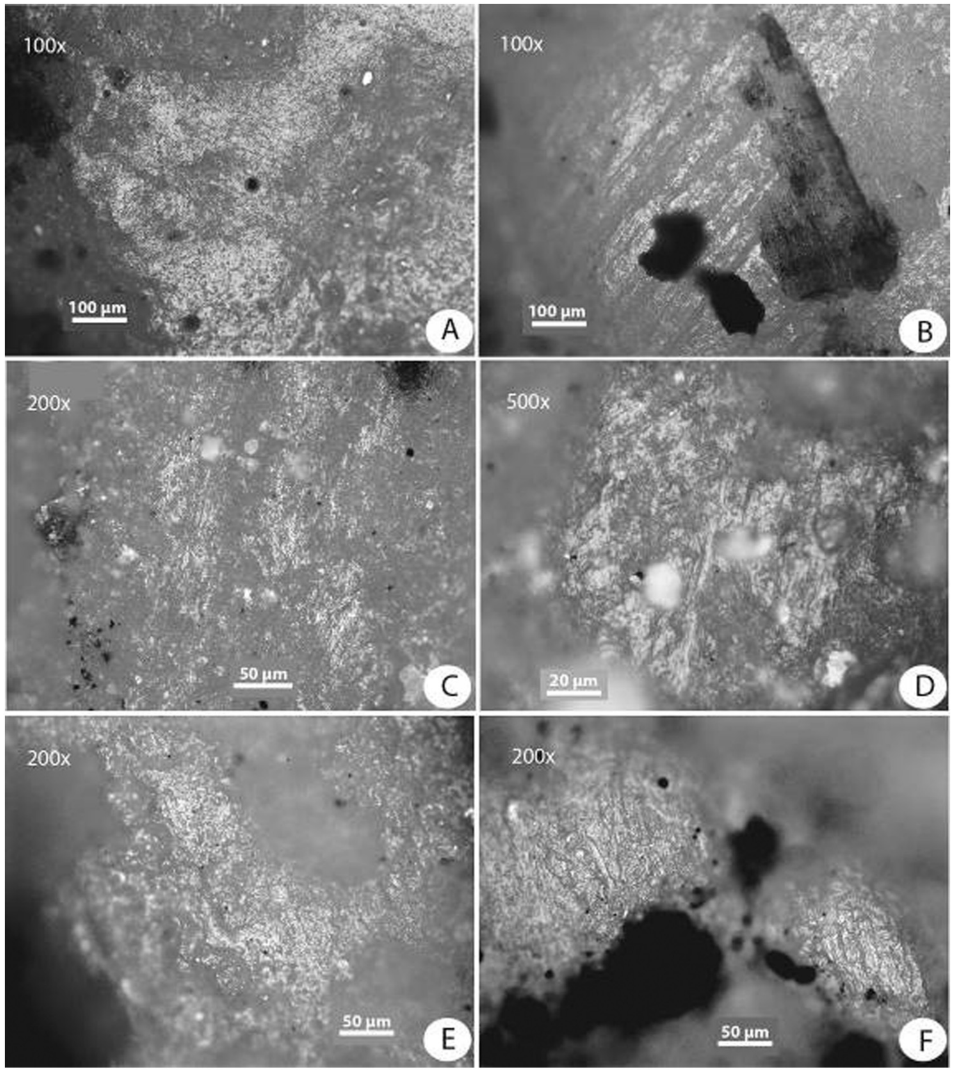

Fig. 7. Use-wear traces on the Raqefet Cave mortars.
(A) BM1 rim, high polish with fine, multidirectional striations; (B) BM1 rim, long furrows; (C) BM1 interior wall, polish with very fine, vertical striations; (D) BM1 interior wall, showing vertical striations under high magnification (500×); (E) BM2 interior wall, polish with very fine, vertical striations; (F) BM3, extensive high polish with vertical wide striations.

5. Functional analyses of the Raqefet Cave mortars

Boulder mortars: On BM1, the rim area shows high-level polish with fine or furrow striations, while its interior surface exhibits medium-to-high level polish with long and very fine striations mostly running vertically. Similar use traces (very fine, vertical striations) were observed on the interior surface of BM2 (Fig. 7A–E). Based on our experimental study (Fig. 2), the rim area of BM1 is likely to have been in contact with various materials with different hardness, including stones, and their use traces are overlapping. Notably, BM1 was found in situ with a large slab by the rim, probably used as a lid (Fig. S1B), which was likely the source of the stone-on-stone use-wear traces on the rim area. The shafts of both mortars were mostly in contact with vegetal-fiber materials, and the movements were mainly vertical in direction. The very fine striations best match those from abrading flax in our experiment (Fig. 2A).

Residue analysis from BM1 and BM2 revealed abundant plant micro-remains, including 59 starch granules, 225 phytoliths, and 79 fibers (including 39 bast fibers). The starch assemblage contains Triticeae (n = 7), Panicoideae (n = 11), oats (n = 15), *Cyperus* sp. (n = 8), lily bulb (n = 1), USOs (n = 3), and undetermined types (n = 14) (Tables 1 & 2). Five Triticeae starch granules exhibit distinctive damage features typical of malting, which are characterized by their hollowed-out structure and/or pitted surface (Fig. 4A,C; Table 1). The phytolith assemblage contains morphotypes mostly derived from grass inflorescence and culm/leaf, including nine dendritic forms most probably from wild wheat/barley husks. Raphides may derive from USOs (Fig. 5; Table 2). Some long bast fibers, likely flax, appeared twisted and entangled (Fig. 6A), suggesting an origin of spun or weaved materials, such as baskets. The patterns of breakage and entanglements appear consistent with those from flax fabric after abrading on limestone in our experiment (Fig. 6D,E). Although we cannot be sure that all fibers are remains of baskets, the strong correlation between the vegetal-fiber related use-wear traces and abundant bast fibers in the residues on the boulder mortars is probably not a coincidence.

The use-wear patterns and microbotanical assemblage suggest that the two boulder mortars were used at least during part of their life-history as storage containers for plant foods, including wheat/barley malts. They were covered with lids, probably made of stone slabs and other materials. The foods are likely to have been placed in baskets made of bast fibers for easy handing, and the movements of the baskets in the mortars were mainly vertical. The deep narrow shafts may have provided cool conditions suitable for storing food, especially for keeping cereal malts (Fig. 1C).

Bedrock mortar: Three use-wear samples taken from BM3 show high level of polish with clear vertical striations. The striations are wider and deeper than those on BM1 and BM2, and the polished areas show rounded edges (Fig. 7F). These features closely resemble those from wood-on-limestone abrasion in our experiment (Fig. 2C). The use traces on the lower part of the mortar are more extensive than those on the upper part. The use-wear pattern suggests that this mortar was used for pounding materials with a wooden pestle.

Sixty-two starch granules and seven phytoliths were recovered from BM3. The identified starches include Triticeae (n = 6), Panicoideae (n = 3), legume (n = 4), *Cyperus* sp. (n = 25), and USOs (n = 8). Forty-two granules show various forms of damage, among which 19 are characteristic of gelatinization due to mashing (Table 1). For example, four Triticeae and two legume starches appear hollowed-out and swollen, with their peripheral areas relatively intact (Fig. 4E,G,I,K). This type of damage/alteration is indicative of a beer-making process in our experiment (Fig. 4F,H,J,L), as described above. In addition, the presence of gelatinized starches was confirmed by the application of Congo red (Lamb and Loy, 2005) on a BM3 subsample (Fig. 6F,G). Other micro-remains include seven grass phytoliths and 29 fibers (Tables 2, 3).

Combining use-wear and residue data, we interpret BM3 as a multi-functional vessel for food preparation, which included pounding plant foods and brewing wheat/barley-based beer, probably with legumes and other plants as additive ingredients. Crushing or pounding cereal malts into grist before mashing is a necessary step during beer brewing. Being watertight and harder than organic containers, such as baskets, this bedrock mortar is advantageous for crushing grains and particularly for retaining liquids and heat over long durations during brewing. An analogy can be found in Mexico where bedrock mortars were used as fermentation pits for making alcohol (Bruman, 2000). The stone-boiling method may have been used for cooking and mashing in Natufian sites (Hayden, et al., 2013), although this suggestion needs to be tested in future studies.

6. Discussion and conclusions

This multi-disciplinary research on a sample of the Raqefet Cave stone mortars sheds new light on Natufian ritual behavior. The Natufians at Raqefet Cave collected locally available plants, stored malted seeds, and made beer as a part of their mortuary rituals to venerate the dead and/or to enhance group cohesion among the living (Hayden, et al., 2013; Munro and Grosman, 2010; Nadel and Lengyel, 2009; Power, et al., 2014; Rosenberg and Nadel, 2014; Yeshurun, et al., 2013). They used stone mortars as brewing facilities, involving three basic stages: malting, mashing, and fermenting. The deep boulder mortars (BM1 and BM2) were used at least in part as storage containers for keeping cereals and USOs, including malted wheat/barley; the bedrock mortar (BM3) was used for pounding, cooking, and brewing beer (Fig. 1C).

The Raqefet Cave mortars reveal the earliest evidence for making basketry with bast fibers, including flax, as malting equipment. Using boulder mortars for food storage was a significant innovation, which may have facilitated the shift towards a sedentary life style (Kuijt and Finlayson, 2009). The time and effort invested in the manufacture of deep stone mortars in mortuary contexts and in acquisition of knowledge apparently required for beer brewing indicate an important ritual function played by alcoholic beverages in the Natufian culture. The Raqefet Cave beer was likely very low in alcoholic content, but it accounts for the earliest known experiment in making fermented beverages in the world. It certainly fits the elaborate Natufian mortuary practices in general, and the direct evidence for using flowers in the graves and conducting wakes by them, as was established for Raqefet Cave (Nadel, et al., 2013; Yeshurun, et al., 2013).

The evidence of beer brewing at Raqefet Cave 13,000 years ago provides yet another example of the complex Natufian social and ritual realms. Beer brewing may have been, at least in part, an underlying motivation to cultivate cereals in the southern Levant, supporting the beer hypothesis proposed by archaeologists more than 60 years ago (Braidwood et al., 1953). The earliest cereal-based beer reported here, and the earliest bread remains recently recovered from the Natufian site of Shubayqa 1 in east Jordan (14,600-11,600 cal BP) (Arranz-Otaegui et al., 2018), demonstrate the wide range of Natufian technological innovations and their complex social organization.

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jasrep.2018.08.008.

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