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Unveiling the culinary tradition of 'focaccia' in Late Neolithic Mesopotamia by way of the integration of use-wear, phytolith & organic-residue analyses

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Recent studies suggest that in Upper Mesopotamia during the Late Neolithic period, specifically between 6400 and 5900 BCE, simple cereal flour doughs were baked in domed ovens using ceramic pans, commonly known as *husking trays*. Adopting an integrated approach that investigates various types of evidence, such as use-wear, phytoliths, and organic residues, we further refined and explored this hypothesis. Analysis of a sample of 13 sherds belonging to these trays from Mezraa Teleilat, Akarçay Tepe, and Tell Sabi Abyad provides evidence that a limited number of them could have been used to bake 'focaccia'-like products with ingredients such as lard or oil. This research project not only further strengthens the theory that *husking trays* could have been used for baking, but also provides insights into the variety and elaboration of food practices that existed amongst early agricultural communities, demonstrating the existence of a number of different 'recipes' for a particular dish. Furthermore, from a methodological perspective, this study highlights how only an integrated approach can contribute to the knowledge of the various culinary traits and traditions of ancient communities.

Keywords Food, Late Neolithic, Pottery function, Use-wear, Phytoliths, Organic-residues

The adoption of practices aimed at promoting the growth of cereals has marked one of the most significant turning points in human history. This invaluable foodstuff, easy to store for long periods, practical to use and distribute, as well as being nutritious, quickly became a dietary staple in Neolithic Mesopotamia and has continued to be so to this day. Remarkable advances, stemming not only from new archaeological discoveries, but especially from recent methodological improvements in the analysis of ancient evidence, are now providing fascinating details on how ancient societies made practical use of this critical food resource.

Even before the widespread adoption of fully developed agricultural practices, the production of flour is evidenced by the presence of ground stone tools^{1–3} and flatbread-like products made from mixed cereal and tuber flour as early as 32,600 BCE^{4,5} and 14,400 BCE in the Near Eastern region⁶. During the Pre-Pottery Neolithic period, this practice is exceptionally exemplified by the finding at Jerf el-Ahmar in Syria of two small cakes composed of ground and charred mustard seeds or rapeseeds⁷.

Throughout the Neolithic period, the dietary practices of communities in the Fertile Crescent probably underwent gradual, but substantial, changes, chiefly due to the increasingly abundant supply of cereals resulting from the widespread adoption of agricultural practices. Archeobotanical remains testify as to how bread-like products became part of the diet of the communities in the area^{8,9}. A recent discovery at Çatalhöyük has unveiled

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a piece of leavened dough, dating to 6600 BCE, containing wheat, barley, and pea seeds; it was found near an oven structure¹⁰.

This transformation in eating behavior was to be then further boosted by the adoption and growing utilization of ceramic vessels during the Late Neolithic period (7th millennium BCE). Pottery technology greatly facilitated the preparation and preservation of food, allowing more practical applications of heat treatment.

With regard to bread-like products, one of the possible baking practices related to the adoption of ceramic containers could have been the baking of plain doughs, made from cereal flour, in large trays with their internal surfaces scored; such impressions/incisions are so characteristic—crudely made, repetitive, and uniformly distributed over the entire internal surface of the vessel—that fragments exhibiting them are generally directly associated with this ceramic form, that is, the so-called *husking trays* (hereinafter HTs)^{11,12}.

This hypothesis is corroborated by multiple sources of data. Experimental studies demonstrate that HT vessels are ideal for baking solid flour-based doughs for 2 h in a domed oven preheated to an initial temperature of 420°C; in this scenario, the scores on the internal surfaces of the pottery shape act as an anti-adhesive mechanism, which allowed the bread to be extracted whole after baking^{13,14}. The use-alteration analysis of a sample of HTs from the Syrian Jazira have revealed that the majority of the wear on the inner surfaces of the vessels was probably caused by their use in baking plain bread-like products; however, some wear appears to have resulted from other, as yet unidentified, activities. The wear pattern suggested the baking of thick loaves, experimentally estimated to weigh 3.5 kg. In addition, the phytolith analysis of that same sample revealed the presence of cereal remains, probably ground into flour¹⁵.

In later periods, our understanding of cereal-based food preparation in Mesopotamia begins to consolidate from the 4th millennium BCE onwards, particularly due to the proto-cuneiform administrative tablets that illuminate the extensive consumption and transformation of baked goods¹⁶.

Only with the extensive written documentation of the 3rd millennium BCE does the information become more precise. The Ur III texts reveal the existence of numerous bread-like products classified by size, type of flour, baking technique and the inclusion of additional ingredients such as fat, fruit, nuts, honey, spices, apples or dates^{17–20}.

However, it is unclear whether such a variety of baked goods was already being produced in earlier times.

As HTs are resilient to degradation and commonly found in many archaeological sites in Mesopotamia, they may offer a unique opportunity to explore bread-based culinary traditions as early as the second half of the 7th millennium (6400–5900 cal. BCE).

Considering that previous studies have already revealed the presence of use-wear evidence on these vessels, produced by unidentified activities, and that later Mesopotamian texts mention bread-like products incorporating various ingredients, in this study, the hypothesis that HTs were also used for baking elaborate doughs is tested.

This allows us to:

- Validate whether HTs were commonly associated with bread-based baking activities;
- Suggest the existence of a possible elaborate culinary tradition;
- Verify direct connections between use-wear patterns and specific residues.

In particular, we examine whether these vessels could have been used for baking cereal doughs seasoned with animal fat or plant oil, since similar ingredients were available in those contexts, and are more readily detectable in archaeological samples.

The research is conducted by using a focused and integrated approach²¹ aimed at investigating the articulated recipes, including use-wear analysis to detect the activities to which fragments were subjected, phytolith analysis to recover botanical remains, and organic-residue analysis to verify the presence of and characteristics of surviving lipid residues.

Materials and results

The sampled HT fragments investigated in this research belong to stratigraphic levels covering much of the Late Neolithic period from various archaeological sites in north-western Mesopotamia (Fig. 1) (SI Appendix, Table S1). Thirteen HT shards were studied, including four fragments from Mezraa Teleilat (MT), two from Akarçay (AKA) and seven from Tell Sabi Abyad (SAB) (Fig. 2).

Archaeological excavations attest that, in that period, those settlements were inhabited by communities dedicated to agriculture (e.g. cereals, legumes and flax) and animal husbandry (e.g. ovicaprids, cattle and pigs), whose products could be cooked on hearths or in domed ovens (e.g. ^{22–27}).

Use-wear analysis was carried out to evaluate the observable signs of use on the archaeological samples, with the first step involving the creation of a collection of macro and micro reference traces through experimental activity. This process was based on the results obtained from previous experiments.

Experimental replicas were used to bake plain doughs (flour, sourdough, and water) or doughs seasoned with animal fat or plant oil.

The parameters on which the trials were based were drawn from the results of a long-standing experimental activity previously conducted^{13,14}. Solid doughs made from stone-ground organic flour mixed with other ingredients were baked in replicas of HTs. These were placed in dome-shaped ovens, similar to those found in the settlements where these types of vessels were discovered, at an initial temperature of 420 °C for about two hours. In the former case, the resulting products resembled large bread loaves, while in the latter case, the presence of lipid ingredients made the products softer and more flavorful, akin to a sort of ‘focaccia’ type bread (Fig. 3a) (SI Appendix, Fig. S1).

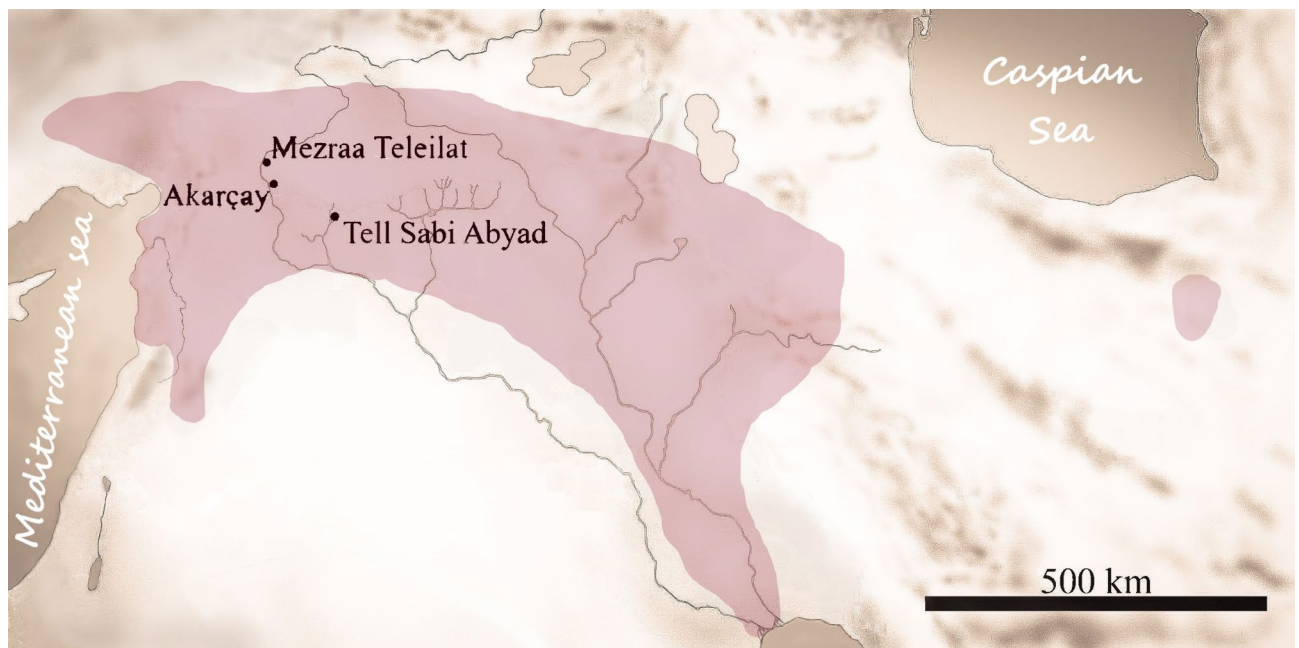


Fig. 1. The Mesopotamian region with the sites marked out where the material analyzed in this research project was found. In red is the area affected by the diffusion of HTs, one of the most widespread artifacts to be found, dating to between 6400 and 5900 cal. BCE Map created using Adobe Photoshop Express (version 3.12.430, Adobe, <https://www.adobe.com/products/photoshop-express.html>) and Paint 3D (version 6.2408.2027.0, Microsoft Corporation, <https://www.microsoft.com/en-us/p/paint-3d>) by ST.

Over the course of the activity, while in both cases soot and abrasive wear formed on the external surfaces of the used pans, the evidence on the internal surfaces varied significantly.

During the baking of plain doughs, macro-wear in the form of ‘rips’ and large bread crusts formed on the ceramic surface, while micro-wear such as depressions developed on the surface mineral inclusions present in the ceramic paste¹⁵.

In contrast, the baking of both doughs seasoned with animal fat or plant oil showed macro-wear in the form of rounded ceramic surfaces, waved profiles, dark spots, scattered crumbs, and micro-wear including rounded mineral inclusions (Fig. 3b) (SI Appendix, Fig. S2, Table S2).

Although the use-wear process in both cases is based on the adhesive mechanism, the presence of lipid substances in the second case may have acted as a lubricant by reducing the friction between the ceramic surface and the ‘focaccia’, thus generating a different wear pattern.

Similarly, wear patterns on archaeological fragments were examined (SI Appendix, Fig. S3, Table S3) and classified into four distinct groups. Through comparison with known experimental patterns, it was possible to hypothesize the activity that might have generated them:

- Group I: ripped ceramic surface and depressions on mineral inclusions; produced by adhesive activity (SAB88 S12 120-5-3, SAB Q14 34-4, SAB88P15 35-105, and MT136);
- Group II: rounded ceramic surface; waved profiles, dark and light spots; rounded mineral inclusions on the surface (Fig. 3c), produced by a lubricant-mediated adhesive activity (AKA 16, MT 167, MT 110, and MT 40);
- Group III: fragments where use-wear from both Group I and Group II co-exist; produced by adhesive and lubricant-mediated adhesive activities (SAB88-549, SAB Q14 39);
- Group IV: overlapped traces (AKA 17, SAB88 371, SAB Q14 50-24).

Phytoliths were recorded in different amounts in all the samples ranging from between 0.2 and 14 million per 1 g of sediment (SI Appendix, Table S4). By far, the largest concentrations were noted among samples from the internal surfaces of the trays coded as MT 40 and MT 136 (over 10 million/1 g of sediment respectively). Both the phytolith concentrations and the weathering index varied considerably among the samples (2–14%, SI Appendix, Table S4). The assemblages do not seem to have been highly affected by weathering alteration or dissolution, pointing to generally good preservation conditions. Furthermore, there is no clear pattern of microfossil abundance and weathered morphotypes between the internal surfaces of the samples and the reference samples from the external surfaces of the trays. All samples yielded multicellular phytoliths with the only exception being the external surface of the HT sample SAB Q14 34-4, where they were completely absent. There does not seem to be a clear pattern between sample types (internal *versus* external) regarding the concentrations of multicelled morphologies either. The associations between weathered and multicellular phytoliths, readily used as indicators of the general state of conservation and integrity of the microfossil records,



Fig. 2. Archaeological fragments analyzed in this research project.

further point to good preservation conditions, although these may have been dependent on a wide range of depositional and post-depositional factors^{28–32}.

Grasses dominated the phytolith assemblages in all samples, being around 80–95% of all the noted morphotypes (SI Appendix, Table S4). In addition, diagnostic morphotypes derived from the floral parts of these plants were abundantly noted in most of the assemblages. The richest proportions of floral phytoliths by far were recorded in samples AK 17, MT 40, SAB88, Q14 39 and SAB126 P15 35-105 (over 40% of all the counted grass morphotypes, Table S4). Grass inflorescences were represented mainly by decorated elongate dentate edges (echinate) and dendritics, as well as epidermal cells such as hairs and *papillate* (Fig. 4a). Further, epidermal appendages produced by grass leaves and culms, including stomata, acute bulbous (trichomes), and bulliforms, were also common in all the samples in varied amounts (Fig. 4b). Grasses mostly belonged to the Pooideae sub-family and were characterized by short-cell rondels and polylobates (up to 50% of the total of the grasses, Fig. 4c). As well as rondels and polylobates (Pooideae sub-family), bilobates and crosses from the Panicoideae sub-family and saddles (from chloridoids) were also recorded in most of the samples, along with elongated (tower) short-cell rondels, which are commonly produced by *Hordeum* sp. taxa³³.

Moreover, multicellular or anatomically connected phytoliths from the husks and culms of grasses, primarily including barley (*Hordeum* sp.) and wheat (*Triticum* sp.) were also recorded in most of the samples, along with multicelled epidermal tissues from the leaves and stems of pooids (Table 1; Fig. 4d). Also of significance is the average number of individual phytolith morphotypes, contained within multicellular forms produced by grasses, displaying differences among the samples (Fig. 4a, d). The average number of single-celled phytoliths recorded in a pilot study on the SAB samples was ca. 9.2 (No. min. cells = 3; No. max. cells = 31), whereas in the current work the number of MT assemblages was ca. 7 (No. min. cells = 3; No. max. cells = 14)^{3,15,28,29,34}. These averages were consistent with experimentally-produced hulled cereal dehusking, multiple rounds of sieving and processing for grinding into flour^{3,34}. Experimental reference datasets obtained from the dehusking of hulled cereals (*Triticum monococcum* and *Hordeum vulgare*) indicated that the average number of individual phytolith morphotypes contained within multicelled forms derived from dehusking by-products, including light chaff, glume fragments or bracts, as well as fragmented grains from cereals and weeds was ca. 24 to 30, whereas, as regards sieved flour, it reached ca. 5 to 6.5^{3,34}. These previous experimental studies quantitatively demonstrated the impact of cereal processing and, in particular, the mechanical degradation of the phytoliths suffered through the different processing stages (e.g. dehusking, sieving and grinding). In summary, the size of multicelled phytoliths from the grasses recorded in the current study further supports the hypothesis that these are remains more likely

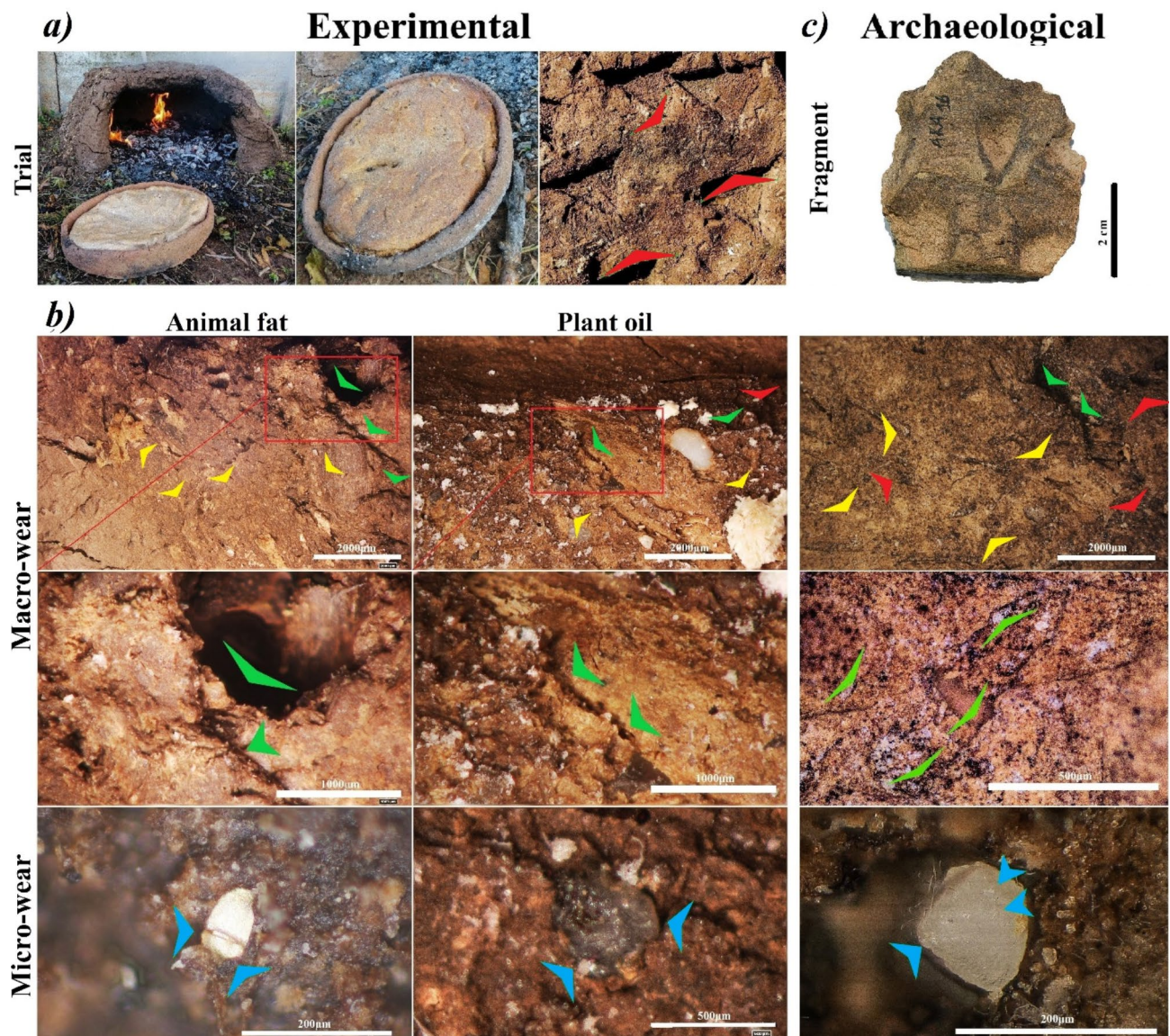


Fig. 3. Use-wear analysis: (a) Experimental baking test of dough seasoned with animal fat in an HT replica. (b) Surfaces of two HT replicas, each used to bake dough 22 times—one for baking dough seasoned with animal fat and the other with plant oil. (c) Archaeological fragment of husking tray AKA 16. The red arrows indicate dark spots; the green ones indicate waved profiles; the yellow ones scattered crumbs/light spots; and the blue ones the rounding of mineral inclusions (micro-traces).

to be associated with ground flour-like products, possibly linked to bread preparation, rather than dehusking assemblages derived from grain cleaning.

Lipid organic residues were utilized in previous studies to explore the HT function, focusing on samples from the sites of Tell Kashkashok and Tell Sabi Abyad. However, these efforts, which relied on solvent extractions, unfortunately yielded no positive results^{35,36}. New analyses conducted on all previously presented samples, excluding fragments SAB88-549 and SAB88 S12 120-5, combined gas chromatographic analysis with acidified methanol extractions (SI Appendix, Table S5, Fig. S4).

Overall, five out of the eleven (45%) fragments analyzed presented a lipid signal above 5 µg/g (AKA16, MT167, MT136, SAB Q14 39, SAB 88–371), the accepted threshold for the positive identification of a residue^{37,38}; while two of them (MT110 and MT40) were at the limit. Lipid concentrations were always low (mean 12 µg/g, median 9 µg/g) despite the use of acidified methanol techniques, best suited for contexts with limited preservation of organic matter^{38–40}. Thus, sample contact with fat may have been sporadic and / or resulting from certain cases. Different amounts of phthalate plasticizers and other modern contaminants were detected in all the studied samples, but their presence did not affect the interpretation of the recovered residues. Sample MT136, which yielded by far the highest fat concentration in its wall (28 µg/g), was further studied by performing additional extractions near the rim and at the base (SI Appendix, Table S6). The quantities of fat detected, similar to those

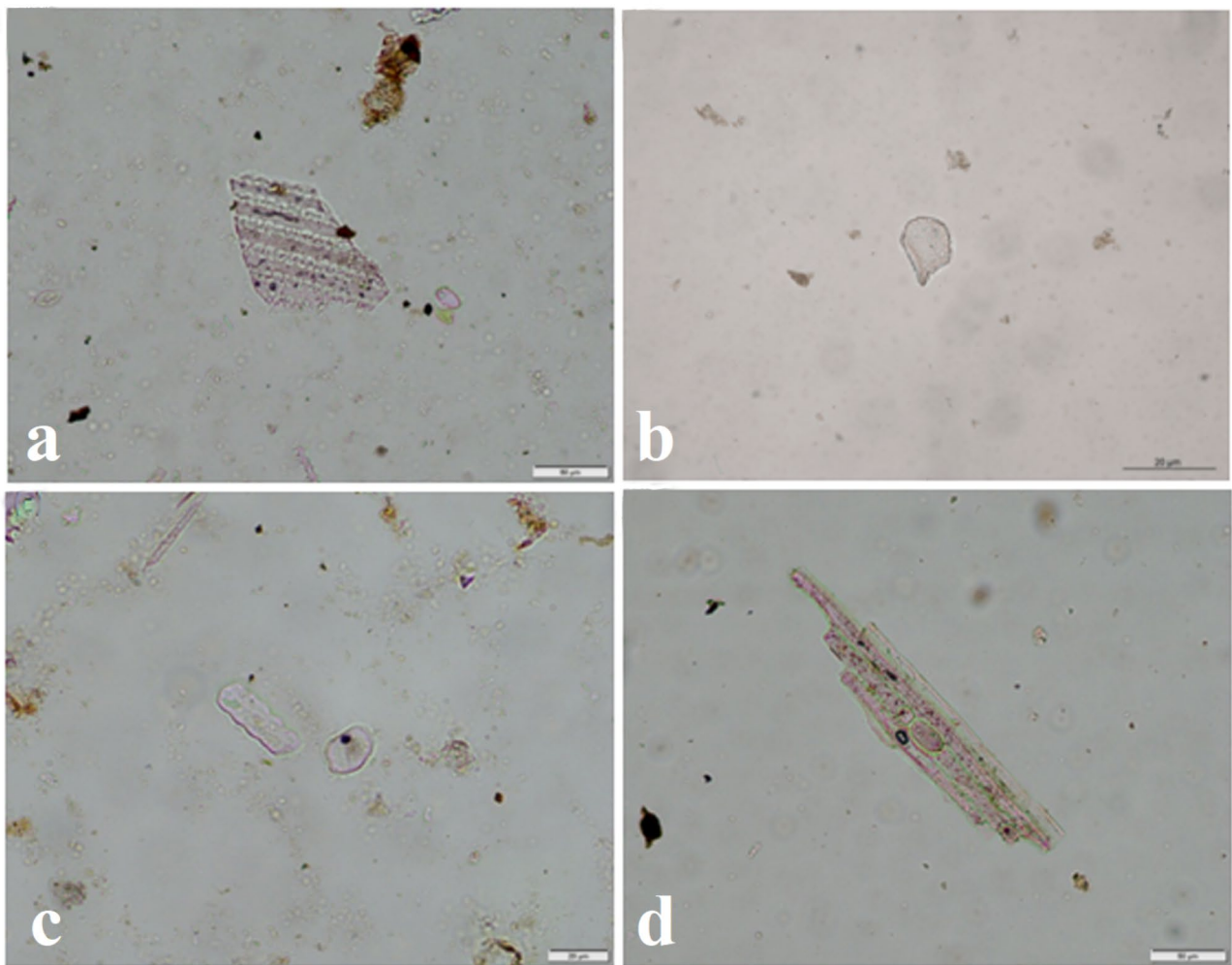


Fig. 4. Photomicrographs of phytoliths identified in HT samples (200× or 400×): (a) multicelled elongate dendritics (SAB88 549); (b) bulliform flabellate (AKA 17); (c) grass silica short cell polylobate and rondels (SAB88 549); (d) multicelled elongates with epidermal appendages (*acute bulbosus*) and rondels from grass leaves (SAB88 S12 120-5).

Item no.	Use-wear	Phytoliths	Organic residues	Interpretation
AKA 16	Lubricant-mediated adhesive activity		Degraded animal fats and plant oil	Baking seasoned doughs
AKA 17	Adhesive activity?	<i>Triticum</i> sp.	No significant lipids preserved	Baking wheat doughs
MT 40	Lubricant-mediated adhesive activity		Minimum quantity lipids preserved	Baking seasoned doughs
MT 167	Lubricant-mediated adhesive activity		Degraded animal fats	Baking seasoned doughs
MT 110	Lubricant-mediated adhesive activity		Minimum quantity lipids preserved	Baking seasoned doughs
MT 136	Adhesive activity	<i>Triticum</i> sp., <i>Hordeum</i> sp.	High quantity degraded animal fats	Baking plain wheat /barley doughs? Multifunctional?
SAB Q14 50-24	Undetermined	<i>Hordeum</i> sp.	No significant lipids preserved	Undetermined
SAB88 549	Adhesive and mediated adhesive activities	<i>Triticum</i> sp.	No significant lipids preserved	Baking plain and seasoned wheat doughs
SAB Q14 39	Adhesive and mediated-adhesive activities, Carbonization	<i>Triticum</i> sp.	Degraded animal fats	Baking plain and seasoned wheat doughs
SAB Q14 34-4	Adhesive activity, Carbonization		No significant lipids preserved	Baking plain doughs
SAB88 371	Adhesive and abrasive activities?	<i>Hordeum</i> sp.	Degraded animal fats	Baking plain and seasoned barley doughs
SAB88 S12 120-5	Adhesive activity	<i>Triticum</i> sp.	n/a	Baking plain wheat doughs
SAB88 P15 35-105	Adhesive activity	<i>Triticum</i> sp., <i>Hordeum</i> sp.	n/a	Baking plain wheat /barley doughs

Table 1. Description of samples, analyzed in this study, obtained from HTs found in Akarçay (AKA), Mezraa Teleilat (MT), and Tell Sabi Abyad (SAB), including details of main use-alteration observations, organic residues and diagnostic multicellular phytoliths, together with their inferred function.

in the wall, did not suggest the presence of a clear filling line⁴¹. Furthermore, the similarity of the P/S, L/M, VLCFA% indices suggest that the origin of the detected residue is homogenous throughout the vessel profile.

Regarding the type of fats recovered, all positive samples were characterized by a series of long-chain saturated free fatty acids (FA), 12–22 carbons long. These were complemented by trace amounts of monounsaturated octadecenoic and hexadecenoic acids, and their associated oxidation products, such as nonanedioic acid and decanedioic acid (AKA16, SAB Q14 39 and SAB88 371). The similar amounts of the two dominant FA, palmitic and stearic acids, suggest that the residue is most consistent with the hydrolysis and oxidation of animal fat triacylglycerols. This is further supported by the low relative amount of very long-chained fatty acids (VLCFA). In one case, sample AKA16, the degraded animal fat signal is combined with evidence of a plant residue, demonstrated by a higher amount of lauric acid compared to myristic acid and the detection of a series of even-chained alcohols, 12–28 carbons long^{42–44}.

Finally, the presence of a series of three ketones, Hentriacontan-16-one (31 K), Tritriacontan-16-one (33 K) and Pentatriacontan-18-one (35 K) (Fig. 5), explained by the thermal decarboxylation of palmitic and stearic acids^{45–47}, was detected in samples AKA16 and SAB371, thereby proving that the original fatty contents of the vessels were, at least in the case of these two samples, heated to increased temperatures of at least 300 °C.

The results obtained from the various analyses outlined seamlessly integrate with each other, allowing for a thorough and solid interpretation of the archaeological samples (Table 1).

The integration of results from these three analytical techniques converges in the interpretation that the fragments in question belonged to pans used for baking different types of:

- cereal-based doughs with no lipid ingredients added (SAB88 S12 120-5-3, SAB Q14 34-4 and SAB88P15 35-105). The fragment MT136 also falls into this category as, in fact, it exhibits superficial rips and anomalies in the quantities and distribution of lipids when compared to the rest of the sample. The pan to which the fragment belonged could have been used for different activities or came into contact with lipids only after ceasing its baking function;
- cereal-based doughs seasoned with lipid-containing ingredients such as animal fats (MT167, SAB88 371, MT40, MT110) or a combination of animal and plant lipids (AKA16); the surface of the SAB 88 371 fragment shows adhesive traces, albeit altered by subsequent abrasive traces. Furthermore, the fragment presents residue patterns (phytoliths and lipids) similar to those of other fragments. It is likely that, after use, this vessel underwent post-depositional alterations. For this reason, the fragment was included in this category.
- both plain and seasoned doughs (SAB88 549, SAB Q14 39).

Nevertheless, insufficient evidence was found to definitely interpret fragments AKA17 and SAB88 Q14-50-24.

A late neolithic bakery practice

While recent finds suggest that baked goods, crucial to today's global population, were already being produced before the widespread adoption of agricultural practices, our research explores the immediately subsequent developments as these types of behavior became more established. In particular, this study has delved into a possible baking practice using specialized containers employed by Late Neolithic Mesopotamian communities (Fig. 6).

The use-wear analysis detected macro- and micro-traces on the majority of the analyzed fragments from Mezraa Teleilat, Akarçay, and Tell Sabi Abyad; they may have formed as a result of their use as baking pans.

The phytolith analysis results point to the processing of cereals and, in particular, to the preparation of bread-like products that are consistent with the available macro-botanical records that are dominated by the presence of hulled barley and emmer wheat. Of particular note is the size of multicellular grass phytoliths (multi-celled or anatomically interconnected) from a selection of HTs from Mezraa Teleilat and Tell Sabi Abyad, indicative of products based on ground flour, possibly linked to bread-type products.

Experimental work has revealed that the doughs had to be rather firm in order to be easily removed from their containers once baked. In addition, they had to be thick, as indicated by the wear patterns on the walls of the HTs, which indicate probable partial leavening for digestibility.

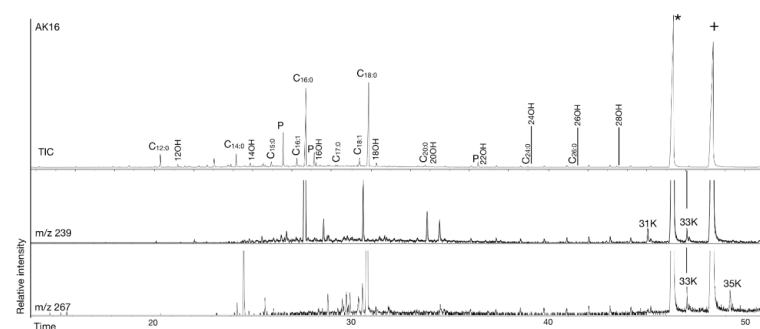


Fig. 5. Total Ion Count (TIC) Chromatogram of sample AKA16A and partial chromatograms for the diagnostic m/z 239 and 267 ions indicative of ketones arising from the thermal decarboxylation of fatty acids.



Fig. 6. ‘Focaccia’ bread seasoned with lard baked in a replica of HT.

Other ingredients such as animal meat/fat or plant seeds/oil, as evidenced by the use-wear and organic residues analyses from each site under examination, could have also been added to the doughs.

The presence of ketones 31, 33 and 35 carbons long suppose that, when detected, the residue and the container likely reached a temperature of probably around 300 °C when in use. This is in agreement with experimental data, which indicate that placing a HT in a domed oven, preheated with glowing embers to an initial temperature of 420°C for two hours, can yield a uniformly baked bread loaf or ‘focaccia’ weighing about 3.5 kg.

The co-existence of two distinct use-wear patterns, each associated with its respective residues, not only confirms that this vessel shape could have been used for baking, but also could have been used to prepare various dishes akin to bread or even ‘focaccia’ products.

Each of these two different baked goods was likely baked in its specific HT. However, in some cases, the overlap of use-wear evidence might suggest that a vessel could have occasionally been used for both types of recipes. The limited number of samples analyzed does not allow for detecting any clear association between the type of scores on the internal ceramic surface of the vessels and the type of dishes.

Although the range of samples analyzed is limited to the area of western Upper Mesopotamia, the probable anti-adhesive mechanism of HTs suggests a close association of this specialized form with the practice of baking.

If HTs are to be considered as being material evidence of this specific baking technique, the entire phenomenon has to be viewed as a significant centuries-long culinary tradition developed within the early agricultural communities of Upper Mesopotamia, and to a much lesser extent of Lower Mesopotamia. In this case, a certain degree of variability within the culinary tradition regarding baking methods and specific recipes

may be expected, influenced by ingredient availability, seasonal resources, and the cultural preferences of each community. It depicts a culinary landscape rich in nuances, not simply tied to resource availability, but reflecting the vibrancy of the cultural context.

Methodologically, this study demonstrates that only integrated analysis can comprehensively reconstruct the history of the ceramic fragments under examination; only consistent data can lead to more robust interpretations, particularly concerning multi-ingredient products. Integrating various analyses prevents the provision of partial data, which, when viewed alone, could lead to erroneous interpretations.

Methods

Use-wear analysis was performed on the entire sample of thirteen fragments employing the methodologies outlined in^{15,48–50}.

The macro-traces were analyzed and documented using a Nikon SMZ-U Stereomicroscope using reflected light with 1 × objective, 10 × eyepiece and magnification from 0.75 × to 7.5 × with a TouPView camera and a portable Dino-lite AM7915MZTL digital microscope. The micro-traces were observed and investigated with a Nikon Eclipse ME600 metallographic microscope together with an Amscope camera and a Hirox Digital Microscope RH-2000.

The use-wear analysis of the archaeological material being sampled was brushed and washed with distilled water before the optical analysis took place.

Phytolith analysis was conducted on twenty-four sediment samples selected from *husking tray* fragments found in Akarçay, Mezraa Teleilat, and Tell Sabi Abyad. The internal surface samples (coded as I Samples) were extracted by way of washing and brushing with distilled water, while samples from their external surfaces were obtained by dry brushing and served as comparison references (E samples, SI Appendix, Table S4). The methodology used for phytolith extraction followed⁵¹.

An aliquot of ca. 20 mg of dried sediment was treated with 50 µl of a hydrochloric volume solution (6 N HCl) and 450 µl of Sodium Polytungstate at a 2.4 g/ml density [$\text{Na}_6(\text{H}_2\text{W}_{12}\text{O}_{40}) \cdot \text{H}_2\text{O}$]. To examine the samples under the microscope, slides were mounted with 50 µl of the sample. A minimum of 200 phytoliths with diagnostic morphologies were counted at 200× and 400×. Slides were examined using an Olympus BX43 optical microscope. Phytolith morphological identification was based on modern plant reference collections^{36,52–54} and specialized literature^{1,55–57}, as well as on comparative experimental records³.

The terms used follow the International Code for Phytolith Nomenclature 2.0, ICPN v. 2.0⁵⁸.

Organic residue analysis was carried out on all the *husking tray* fragments of the sample except for SAB88-549 and SAB88 S12 120-5.

To extract lipids surviving from potential use, acidified methanol extractions were performed on 1 g of ground pottery spiked with internal standard 10 µg of *n*-tetratriacontane. Four ml of methanol were added to the sample, and the mixture was ultrasonicated for 15 min and acidified with 0.8 mL of concentrated sulphuric acid. The mixture was heated at 70 °C for 4 h, then left to cool at room temperature, extracted with 2 ml of *n*-hexane three times and neutralized with potassium carbonate. Samples were dried under a gentle stream of nitrogen and dissolved in 100 µl of *n*-isooctane prior to injection. Selected samples were further derivatized with BSTFA + 1%TMCS to yield trimethylsilyl ester derivatives prior to GC-MS analysis.

Samples were analyzed first with a 7820 A Agilent Gas Chromatograph (GC) fitted with a Flame Ionization Detector (FID). The injection was done in splitless mode at a temperature of 300 °C and eluted through an HP-1 capillary column (60 m long, 250 µm internal diameter, 0.25 µm film thickness) using hydrogen as the carrier gas. The oven temperature was initially set at 50 °C for 1 min, and then increased at 6 °C/min to 320 °C, where it remained for 20 min.

Positive samples were subsequently analyzed by Gas Chromatography-Mass Spectrometry. Briefly, 1 µl was injected into an Agilent 6890 N coupled to an Agilent 5973 N Mass Spectrometer. The GC was fitted with a DB-1 column measuring 30 m × 250 µm × 0.25 µm. The GC injector was operated in splitless mode and helium was used as the carrier gas. The oven temperature was set at 50 °C for 2 min and then increased at 10 °C/min to 300 °C and held at that temperature for 15 min. The Mass Spectrometer was run in electron impact mode and masses were acquired in full scan mode between 50 and *m/z* 600. Results were compared to the NIST2.0 database.

In the case of sample AKA16a, compound-specific isotopic analyses were performed on a Delta V Thermo Fisher isotope ratio mass spectrometer linked to a Trace GC Thermo Fischer Scientific. Helium was used as the carrier gas, and the combustion reactor was set at 940 °C. Samples were diluted in isooctane and between 0.5 and 2 µl of solution were injected into a DB-5 MS (60 m × 0.25 mm × 0.25 µm) column depending on the sample's fatty acid concentration. The injector temperature was set at 310 °C, and the oven was initially set at 80 °C for 1 min, then ramped up at 30 °C/min to 120 °C, and then the temperature was increased to 320 °C at 6 °C/min and held at this setting for an additional 21 min. Results have been presented in the standard notation relative to the Vienna Pee Dee Belemnite (V-PDB) standard. $\delta^{13}\text{C}\text{‰} = (\text{R}_{\text{sample}} - \text{R}_{\text{standard}}) / \text{R}_{\text{standard}}$ has been applied for the correction. Values were further corrected to account for the methylation of the carboxyl group using a mass balance correction.

The analyses were performed at:

- LTFAPA Laboratory, Sapienza University of Rome (use-wear);
- ICTA and Archaeobotany Laboratories, Autonomous University of Barcelona (UAB) (organic residues and phytoliths);
- The Prehistory Laboratory, Istanbul University (use-wear);
- The ARHA Laboratory, Koç University (use-wear and organic residues);
- The Institució Milà i Fontanals (IMF), Spanish National Research Council (IMF-CSIC) (phytoliths).

Data availability

All data generated or analyzed during this study are included in this published article (and its Supplementary Information files).

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Author contributions

S.T. and C.L. designed the research; S.T. performed the experimental and use-wear analysis, A.B.B. the organic residue analysis, and M.P. the phytolith analysis; M.L.M., A.G.B., and M.M. provided the archaeological material; A.B.B. wrote the organic residue section, M.P. wrote the phytolith section, and S.T. wrote the remaining parts of the paper. S.T., A.B.B., M.P. and C.L. reviewed the text.

Competing interests

The authors declare no competing interests.

Additional information

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