

# An experimental approach for tracing olive processing residues in the archaeobotanical record, with preliminary examples from Tell Tweini, Syria

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**Abstract** To help the interpretation of possible olive through more experimental replication and additional processing residues at archaeological sites, this study examines the changes which occur in pre- and post-charring breakage surfaces of olive stones at tissue level. Fractures and alterations in the olive stones can be observed and heated to different temperatures (230, 330, 430 °C) on oxidising and non-oxidising conditions. The structures obtained by the experiment were studied with scanning electron microscopy (SEM) and the characteristics for different temperatures, oxidation regimes and pre- or post-charring fractures were recorded. Furthermore, the experimental specimens were compared with recent and possibly old fractures of several archaeological olive stones from Tell Tweini, Syria. Criteria to infer their formation are discussed. These criteria could be developed further

**Keywords** Charring experiments · SEM · Olive processing · Introduction

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Numerous charring experiments have been conducted on plant materials, such as seeds and fruits, to aid archaeobotanical analyses (Hopf 1955; Helbrink 1970; Kerber-Grohne and Pienning 1980; Wilson 1984; Kislev and Rosenzweig 1989; Boardman and Jones 1990; Wright 1998). Cereals and pulses are common finds in charred plant assemblages and have accordingly featured in experimental studies: for recent advances, see Braadbaart 2004. However, more and more other taxa and types of plant remains including wood (Braadbaart and Poch 2008), food remains (Valamoti 2002; Valamoti et al. 2008), grape (Mangafa and Kotsakis 1996; Margaritis and Jones 2006), olive (Adam-Veleni and Mangafa 1996; Margaritis and Jones 2008a, b) and other kinds of cultivated and wild-growing seeds and fruits are being studied by such experiments as well (Gustafsson 2000; Markle and Reich 2008; Sievers and Wadley 2008; Wilson 1984; Wright 2003).

Charred and often broken olive stones were recovered in large quantities from Tell Tweini, ancient Gibala (Bretschneider and van Lerberghe 2008) on the Syrian coast (Fig. 1). The presence of the olive is recorded both by pollen evidence and plant macro remains from the Bronze

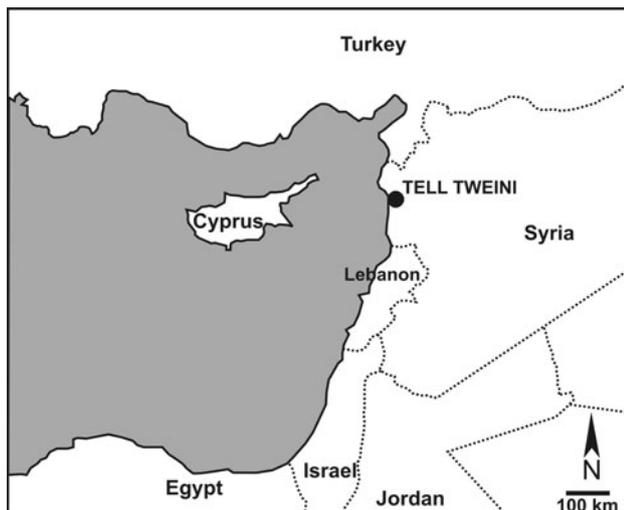


Fig. 1 Map of the study area with the location of Tell Tweini

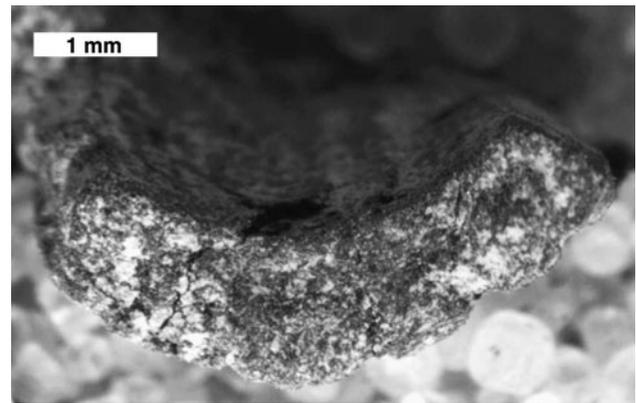


Fig. 2 Fossil olive stone from Tell Tweini, pit 942, early Bronze Age

and Iron Age layers at the site. The palynological research are explored. based on numerical approaches and estimating the correlation of the olive pollen curve with those of other cultivated plants, suggests that the olive stands around the site during the late Bronze and Iron Age were wild in character (Kaniewski et al. 2009). The abundant archaeobotanical finds of olive stones, on the other hand, raised the question whether the olives found were originally crushed in the course of processing for oil or just charred and broken afterwards. Thus in the current paper we explore the possibility of investigating olive oil production through the possible by-products of olive pressing. This investigation in conjunction with the pollen evidence, might help us obtain direct evidence of the possible pressing of olive oil and to refine the picture of olive use, growing and processing during the Bronze and Iron Age in the northern Levant.

Observations on olive pressing residues used as fuel from a Jordan led Neef (1990) to suggest that olive fragments with fractures rounded along the edges had been crushed during ancient times, while post-depositional breakage is often recognizable by the sharp edges of the broken faces of the stones. Rounded fractures, but also dull fragmentation faces (example given in Fig. 2) have been said to be indicative of breakage prior to deposition (Simchoni and Kislev 2006; Margaritis and Jones 2008a, b).

Previous studies were primarily restricted to macroscopic observations on olive remains. Adding to the observations of the authors we mentioned above, the current study aims to explore whether differences between pre- and post-charring breakage surfaces could also be detected at an ultrastructural level. A further objective was to investigate the influence of varying charring conditions (lower or higher temperature, degree of oxygenation) on

the morphology of the breakage surfaces. Finally, the potentials and limitations of the proposed approach for the study of olive processing residues from archaeological sites

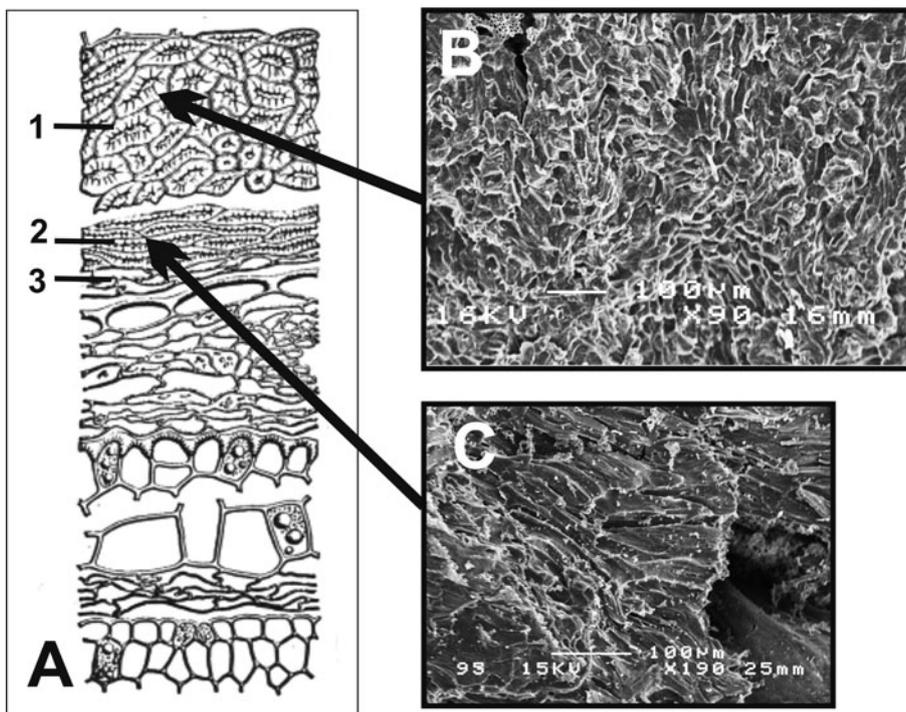
## Materials and methods

### Sample preparation

Fresh olives from cultivars for oil production were harvested by hand from Syrian and Italian groves and stored in paper bags. The fresh olive fruits were crushed and pounded manually with a stone mortar in the laboratory, in order to produce breakage surfaces. Several vertical pounding movements were needed to soften the fruit flesh and produce crushed stones. The aim of the procedure was not to imitate the historically known methods of olive processing, but just to produce enough breakage surfaces for the experimental charring and ultrastructural observations.

About 10–20 whole or broken fruits were then charred in a muffle furnace. Such units achieve high temperatures in a short time, allowing rapid processing, and their temperature is easily controlled. The conditions inside the muffle furnace are not non-oxidising, so various methods should be used to prevent the supply of air to the material. For the current study, the experimental materials were placed in ceramic crucibles and embedded in sand, covered completely under a ca. 3 cm layer. Thus in the following, when

Fig. 3 Microscopic structure of olive stone endocarp. a Cross section after Winton and Winton (1932), b SEM image of the outer layer, c SEM image of inner layer



experimental heating of wheat, pea, sunflower and wood

Results strictly non oxidising conditions (see Braadbaart 2004;

Braadbaart et al. 2007; Braadbaart and Poole 2008).

On the SEM images produced from the experimentally charred and archaeological olive stones, we first identified mainly to resemble the approximate duration of pre in the cell layers for recognizing the pre- and post charring domestic preplaces or furnaces. Of course longer durations breakage as described in Winton and Winton (1932). The such as all day might have been involved in the actual endocarp (Fig. 3a) consists of three layers: (1) outer stone cells, which are large, isodiametric or transversely elongated, often with a large lumen; (2) inner stone cells, which are narrow and transversely elongated; and (3) parenchyma more or less compressed (Winton and Winton 1932). The (temperatures, duration) were chosen because the archaeological layers of the seed coat, endosperm and cotyledons are more complex and are not subject of the current study. SEM that most of the studied samples originate from fuel used in a domestic context. This formation process has also been mentioned on experimentally charred olive stones. Another inferred in previous studies by Neel (1990) and Salavert (2008).

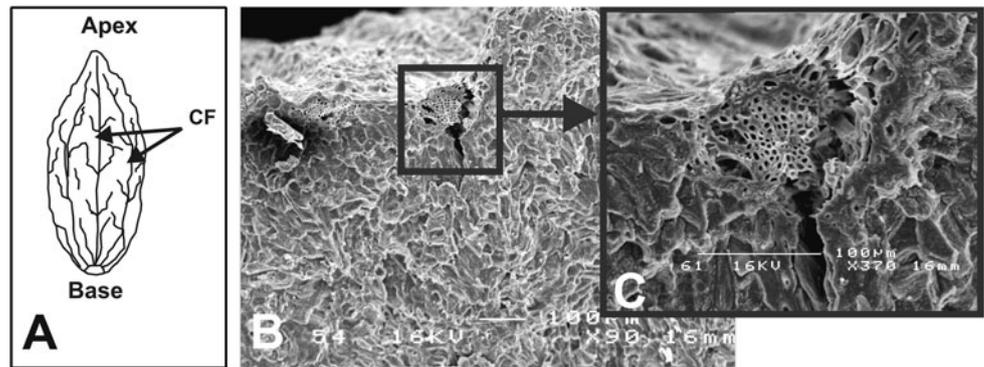
#### Ultrastructural analysis

The experimentally charred objects and pre archaeological specimens were selected by examining them under a stereomicroscope and subsequently a Zeiss Axioskop reflex microscope. Fragments showing the cross section of the olive stone were prepared for scanning electron microscopy (SEM) by sputter coating them with gold. A JEOL JSM 6400 scanning electron microscope was used for the actual analysis and digital pictures were generated with SemAfore.

Duration of heating was fixed at 3 h and was selected charred and archaeological olive stones, we first identified mainly to resemble the approximate duration of pre in the cell layers for recognizing the pre- and post charring domestic preplaces or furnaces. Of course longer durations breakage as described in Winton and Winton (1932). The such as all day might have been involved in the actual endocarp (Fig. 3a) consists of three layers: (1) outer stone cells, which are large, isodiametric or transversely elongated, often with a large lumen; (2) inner stone cells, which are narrow and transversely elongated; and (3) parenchyma more or less compressed (Winton and Winton 1932). The (temperatures, duration) were chosen because the archaeological layers of the seed coat, endosperm and cotyledons are more complex and are not subject of the current study. SEM that most of the studied samples originate from fuel used in a domestic context. This formation process has also been mentioned on experimentally charred olive stones. Another inferred in previous studies by Neel (1990) and Salavert (2008).

characteristic feature of the olive stones is the carpellar fascicle, which is a groove in the surface of the olive stone (Fig. 4). This pressure apparently continues to run through the entire cross section of the stone, and two of these extensions can join together as is the case here (Fig. 4). In the experimentally charred olive stones, the structures related to the carpellar fascicles (CF) are remarkably better preserved than the surrounding cells (Fig. 4). The impact of differing charring conditions (oxidising and non oxidising combined with changing temperature) on the morphological structures of the olive stone breakage (pre- and post charring) surface is illustrated in Fig. 4. At 230°C there is no visible difference between the pre- and post charring breakage. The cell structure is in both

Fig. 4 a Morphological structure of olive stone carpellar fascicle from experimental fractures, pre-charring breakage, at 330°C; c the same image giving detail of the carpellar fascicle



cases clearly distinguishable. This was observed for both marked by concretions which appear as particles on its oxidising and non oxidising charring. The cell structure is visible also in pre-charring broken surfaces heated to 330°C in non oxidising conditions, but not when the pre-charring breakages were heated to the same temperature in an oxidising environment.

Clear differences between the pre- and post-charring broken olive stones are often thought to be indicative of fractures are visible in olive stones charred at 330°C under both oxidising and non oxidising conditions and at 430°C under non oxidising treatment (Fig. 5). In the case of 330°C, the cell structure is still visible on pre-charring fractures, while the post-charring broken surfaces are more or less smooth, with cell structures no longer visible. At 430°C, a freshly cracked post-charring non oxidised surface is very smooth, with oval depressions as remnants of some cell cavities (Fig. 6a).

Generally by increasing the temperature, the dissimilarities between surfaces under non oxidising and oxidising conditions as well as between pre- and post-charring breakage become more pronounced.

Some archaeologically excavated olive stones from Tel Tweini were also analyzed by reflected light microscope and SEM. Several stone fragments that seemed to have been broken before charring were selected, based on their surface relief, which had smooth edges and coarse surfaces (examples in Figs 2, 7c). These fragments were more fragile than the experimentally charred ones, probably due to fluctuations during the exposure to heat in ancient times. Cell cavities of variable size were still visible on the fracture surface, though in some places they seemed to have been eroded. This resembles the experimental specimens with pre-charring breakage, charred at 330°C in oxidising conditions (Fig. 7b). The archaeological olive stone broken on purpose (Fig. 6b) looked entirely different from the former, and resembled the experimental specimens after charring to a remarkable extent, confirming the validity of reflected light microscope and SEM it is possible to identify exactly our experiment. Again this old stone, like the others, is porous and easily damaged, with many cracks, and altered (Figs 5, 6, 7). In this paper we have used SEM

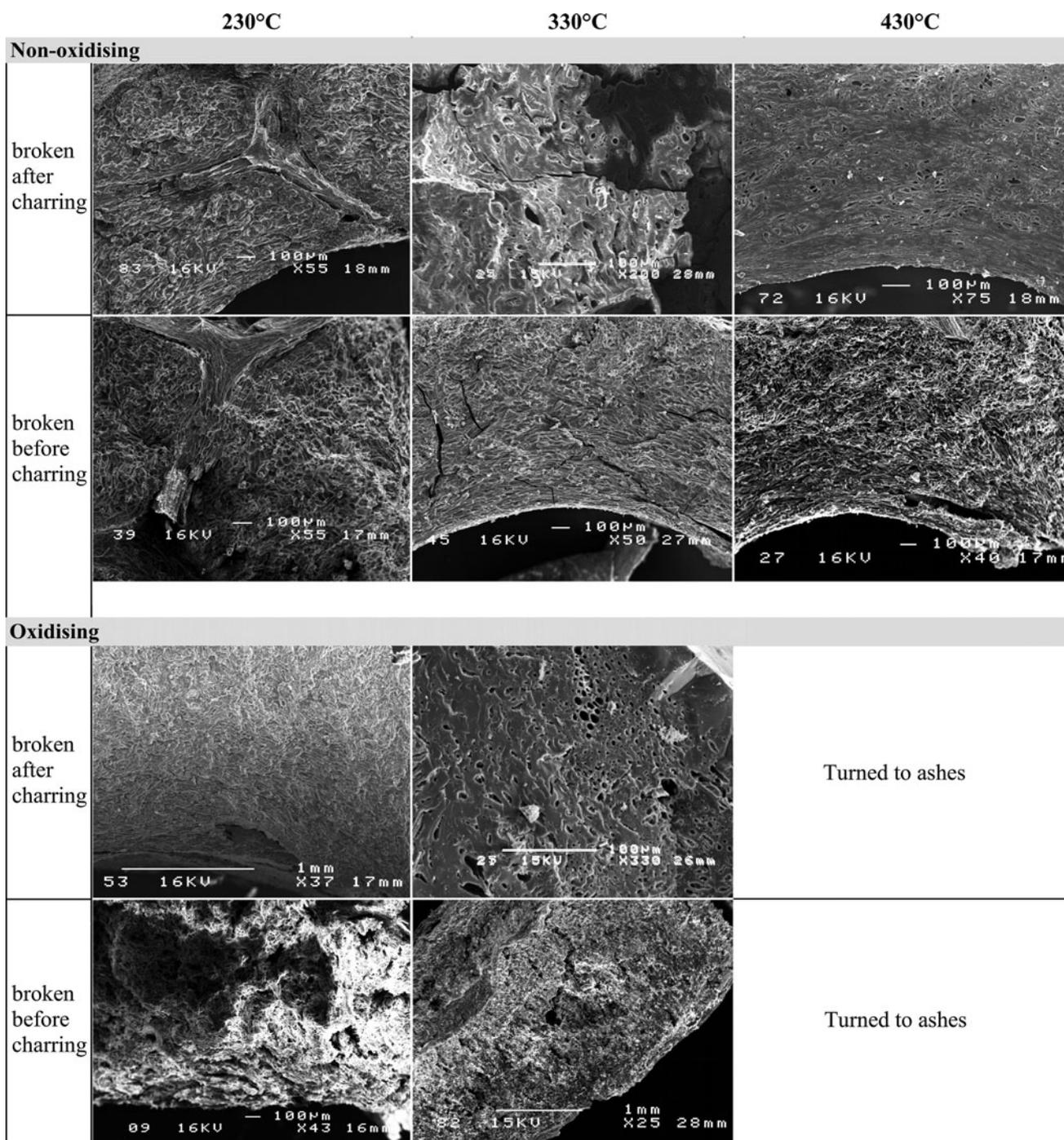


Fig. 5 Preservation of the cell structures in the fractured surfaces, at temperatures of 230, 330, and 430°C, under oxidising and non oxidising conditions

images because of their superior illustration qualities, but and post-charring breakage is negligible. Some of the larger fragments heated to 230°C in non oxidising conditions were not completely charred. This means that in sites with reflected light microscope.

Using this information, we also found indications for the dry (but not desiccated) preservation conditions, where the combined effect of temperature and oxygenation on the uncharred organic matter decomposes, such stones would have minimal chances to survive. These stones would then have minimal chances to bias the recognition of pre-dising treatments (compare Fig), as well between pre- and post-charring fractured olive stones in the

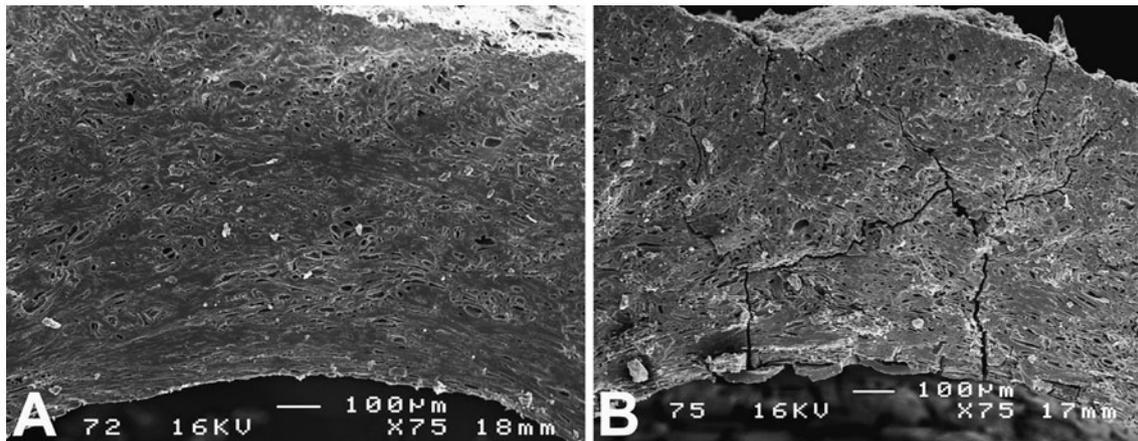


Fig. 6 a Overview of cross sections after experimental, post-charring breakage, non oxidising; 430°C; b Olive stone cross section surface, ancient, post-charring breakage, piece broken from whole olive from pit 942, Tell Tweini

archaeobotanical record. When temperatures rise to 330°C, later Iron II to Iron Age III period, when the site was under the cell patterns of oxygenated surfaces broken prior to Assyrian control. Processing installations consisting of charring become more disordered and start disintegrating. Large stone presses were found in nearly every house but are still visible compared to those broken after charring (Bretschneider and van Lerberghe 2008). (Fig. 3b). At 430°C, the majority of the complete and fragmented olive stones charred under oxygenated conditions even completely turned to ashes. Together with the observation that specimens become more brittle when charred at higher temperatures, it is clear that non oxidising charring has the highest preservation potential. Archaeological whole olive stones from Tell Tweini that were broken for further study by SEM showed morphologies very similar to those with breakage after charring at 330°C and 430°C in our experimental material (see Fig. 6). This indicates that the experimental results presented here may be applicable to archaeological material. On the other hand, the supposedly old archaeological fractures only showed certain similarities with the experimental materials, but also uneven surfaces and eroded cell walls, most probably as a result of post-depositional processes (Fig. 3). Given that it was not possible to make a distinction between pre-use and post-charring breakage at 260°C it is necessary that we explore this temperature with longer durations in the future.

Possible pressing installations at Tell Tweini, Field A, already studied samples with an average volume of 20 l are recorded in the Iron Age II/III levels 6 and 5 (ca. 800–550 B.C.). Six well preserved presses were excavated there (ESM 1). They were each made of a monolithic limestone block located in the corner of a room measuring around  $1.8 \times 1.3 \times 0.4 \text{ m}^3$  (ESM 2). The surface of the stone was hollowed out with small channels leading the liquids to the small ended sides of the presses. In combination with the stone presses, large round ceramic basins for collecting the liquid were preserved in front of them. The large scale industrial pressing possibly of olives and grapes at Tweini (Field A, ESM 1) is significant for the

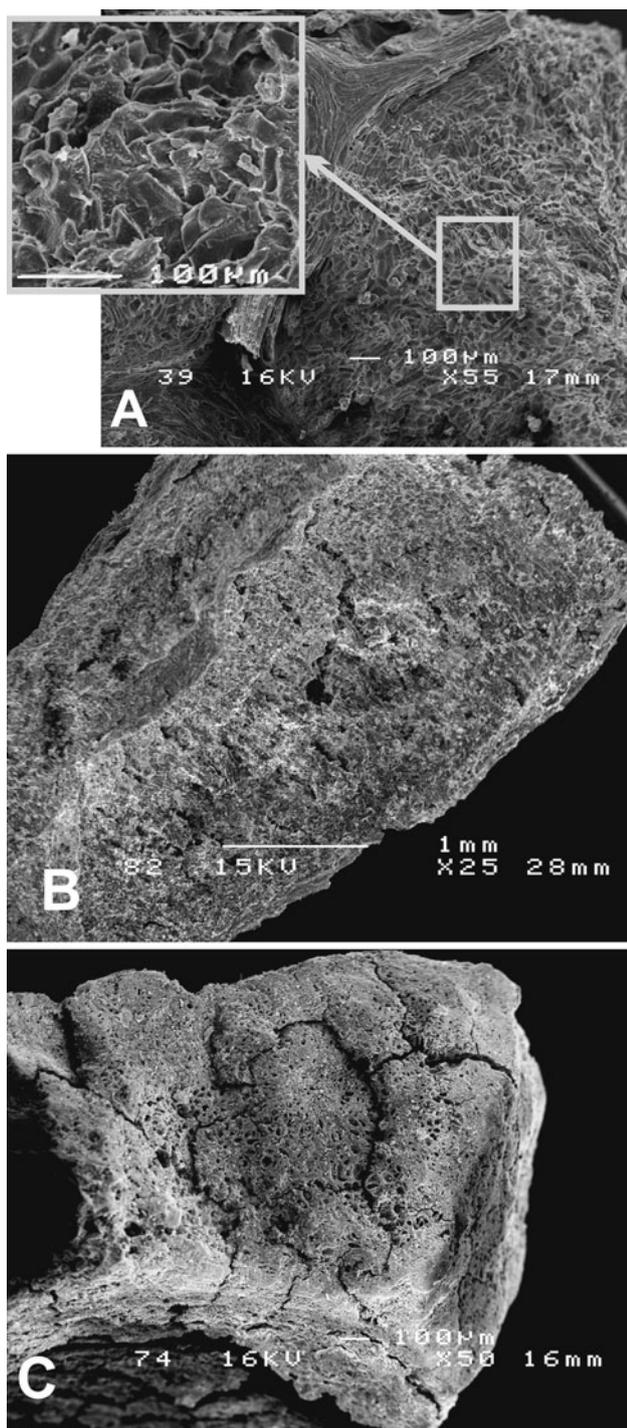


Fig. 7 a Overview and detailed image of an olive stone cross section surface, experimental, pre-charring breakage, non oxidising, 230°C; b overview image of an olive stone cross section surface, after experimental, pre-charring breakage, oxidising, 330°C. The piece is almost crumbling down to ashes; c ancient olive stone in cross section surface, ancient, supposedly pre-charring breakage. Note uneven surface with more relief. From middle Bronze Age tomb 1085, Tell Tweini

Factors which would have influenced the breakage surface of the olive processing residues include their use as fodder or fuel in the past. Most probably the use as fuel is the main factor for preservation of such remains in the archaeobotanical record (see the discussions in Neef and Salaver 2008). The use as fodder and subsequent use of the dung as fuel could also be the source of such fragments in the archaeobotanical record. Nevertheless the passing through the digestion of domestic herbivores would probably have a certain effect and also needs further experimental research.

Refining the experimental observation and examination of a greater amount of olive stone fragments from the different periods of the site using, among others, the methods presented in this paper, will give more answers to the question of olive oil production in the surroundings of Tell Tweini.

### Conclusions

The presented experiments confirmed that SEM allows the investigation of aspects of olive oil production, in particular those related to processing of olive stones and residues from them. The structures which were observed using a scanning electron microscope are also recognizable under a reflected light microscope, which makes the application of our results to other archaeobotanical assemblages simple and easy.

Differences between olive fragments broken pre- versus post-charring are not visible at lower charring temperatures of 200–230°C, and these need to be further explored by increasing charring duration at these temperatures. The pre- and post-charring breakage surfaces of olive stones charred by temperatures between 330 and 430°C could clearly be differentiated based on the changes which took place in the ultrastructure of the breakage surface.

This charring experiment permits a more rigorous interpretation of archaeologically recovered fragmented olive stones, and hence the formation of olive processing remains charred between 330 and 430°C. However, further parameters explored in the future through continued experimentation will enrich our methodology. The reliability of the method presented here in this pilot study and its real potential should be thoroughly tested on different archaeobotanical assemblages.

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