

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

Elena Marinova · Jan M. A. van der Valk ·  
Soultana Maria Valamoti · Joachim Bretschneider

Received: 26 September 2010 / Accepted: 21 April 2011  
© Springer-Verlag 2011

**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

Communicated by G. Willcox.

Electronic supplementary material The online version of this article (doi:10.1007/s00334-011-0298-y) contains supplementary material, which is available to authorized users.

E. Marinova (✉)  
Center for Archaeological Sciences, Katholieke Universiteit Leuven, Celestijnenlaan 200E, 3001 Leuven, Belgium  
e-mail: elena\_marinova@gmx.de;  
elena.marinova@bio.kuleuven.be

J. M. A. van der Valk  
Bioarchaeology Unit, Royal Belgian Institute for Natural Sciences, Vautierstraat 29, 1000 Brussels, Belgium

S. M. Valamoti  
Department of Archaeology, Aristotle University of Thessaloniki, 541 24 Thessaloniki, Greece

J. Bretschneider  
Department of Near Eastern Studies, Katholieke Universiteit Leuven, Blijde Inkomstraat 21, 3000 Leuven, Belgium

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 Rech 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

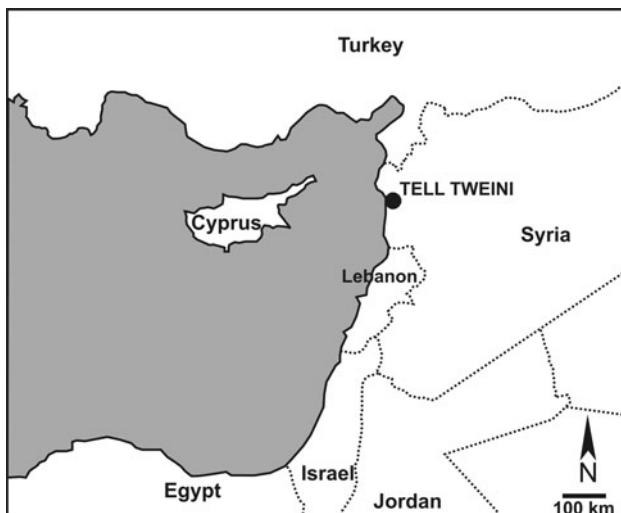


Fig. 1 Map of the study area with the location of Tell Tweini

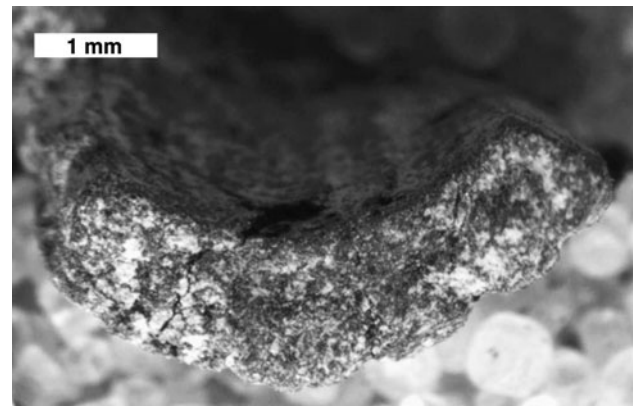


Fig. 2 Fossil olive stone from Tell Tweini, pit 942, early Bronze Age

and Iron Age layers at the site. The palynological research 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).

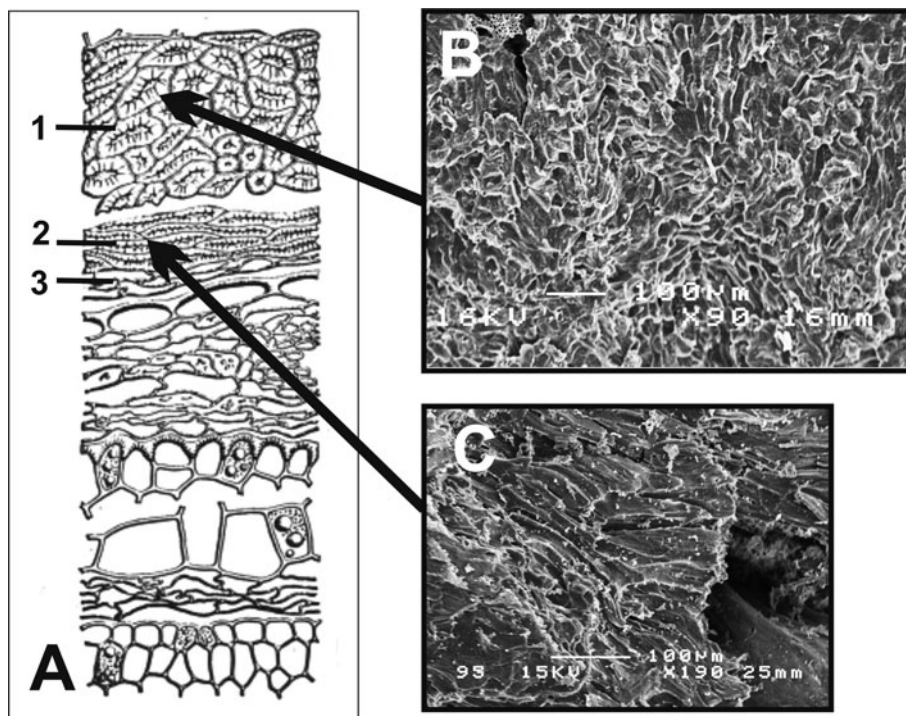
Previous studies were primarily restricted to macroscopic observations on olive remains. Adding to the observations of the authors we mentioned above, the current study aims to explore whether differences between pre- and post-charring breakage surfaces could also be detected at an ultrastructural level. 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 in strictly non oxidising conditions (see Braadbaart et al. 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 preheating in the cell layers for recognizing the pre- and post charring domestic preheating places or furnaces. Of course longer durations of heating at low temperatures, could be explored in the future. The currently presented approach and conditions (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 overviews are given (Fig. 3b, c) to illustrate the features 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).

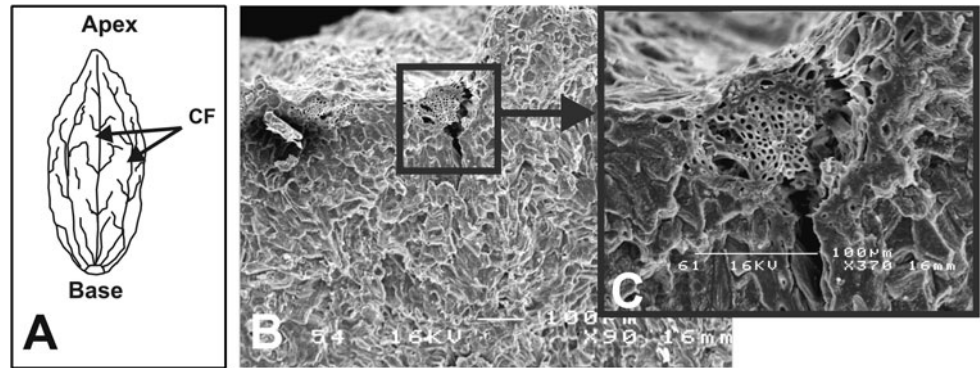
#### Ultrastructural analysis

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.

Results  
 Duration of heating was fixed at 3 h and was selected to resemble the approximate duration of preheating in the cell layers for recognizing the pre- and post charring domestic preheating places or furnaces. Of course longer durations of heating at low temperatures, could be explored in the future. The currently presented approach and conditions (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 overviews are given (Fig. 3b, c) to illustrate the features 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 fascicleb SEM image 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 differences 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 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 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

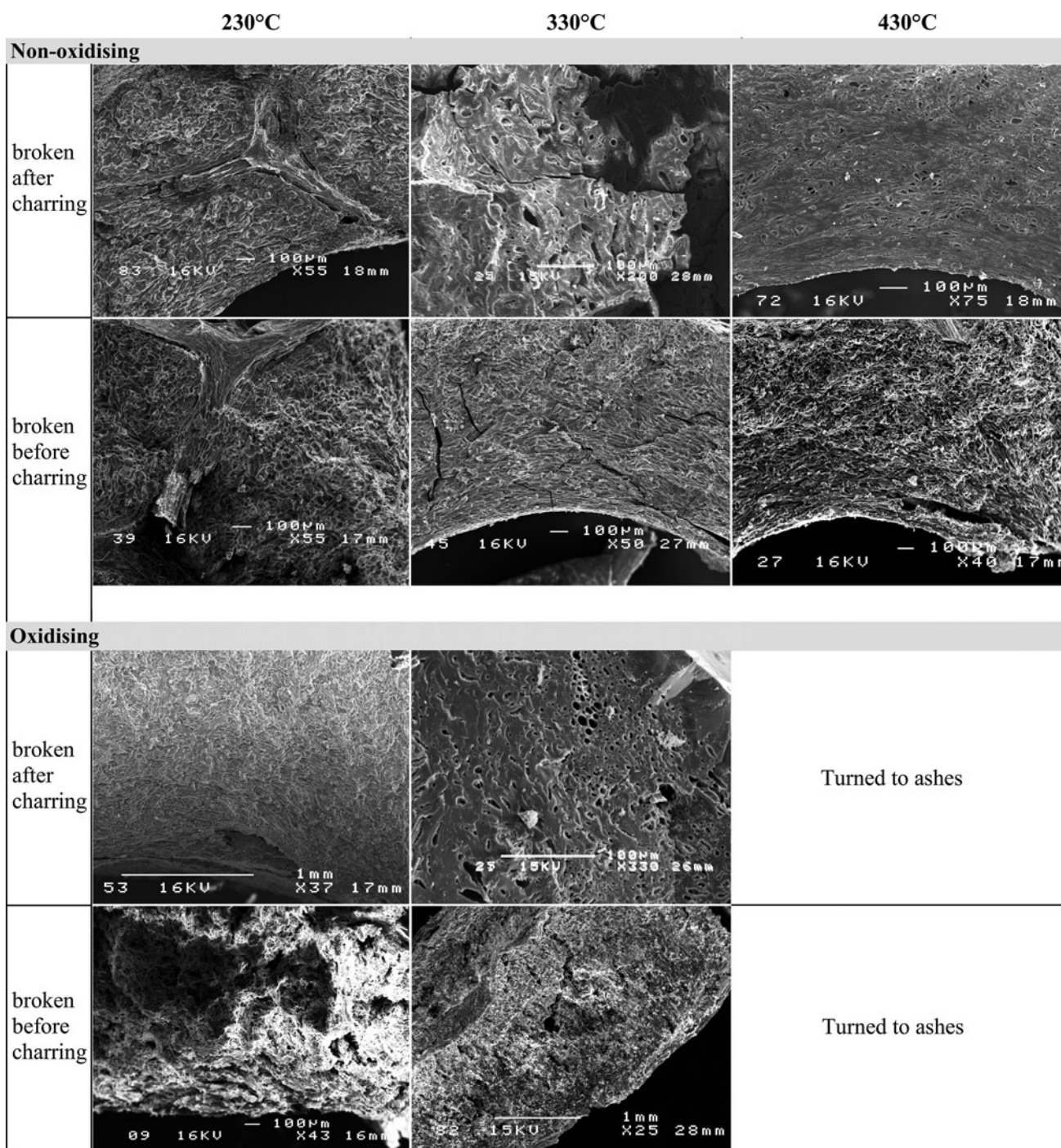


Fig. 5 Preservation of the cell structures in the fractured surfaces, at temperatures of 230, 330, and 430°C, under oxidising and non oxidising conditions

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



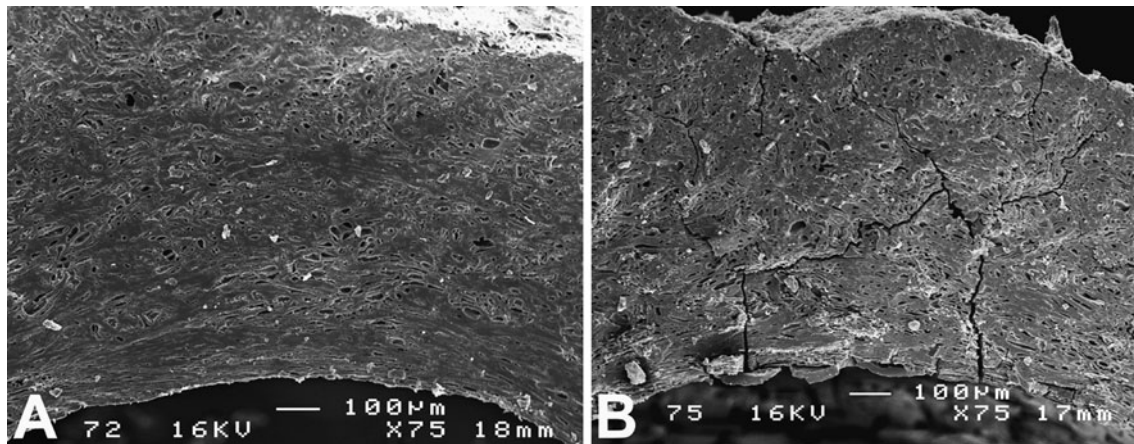


Fig. 6 a Overview of cross sections after experimental, post-charring breakage, non oxidising; 430°C; b Olive stone cross section surface, ancient, post-charring breakage, piece broken from whole olive from pit 942, Tell Tweini

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

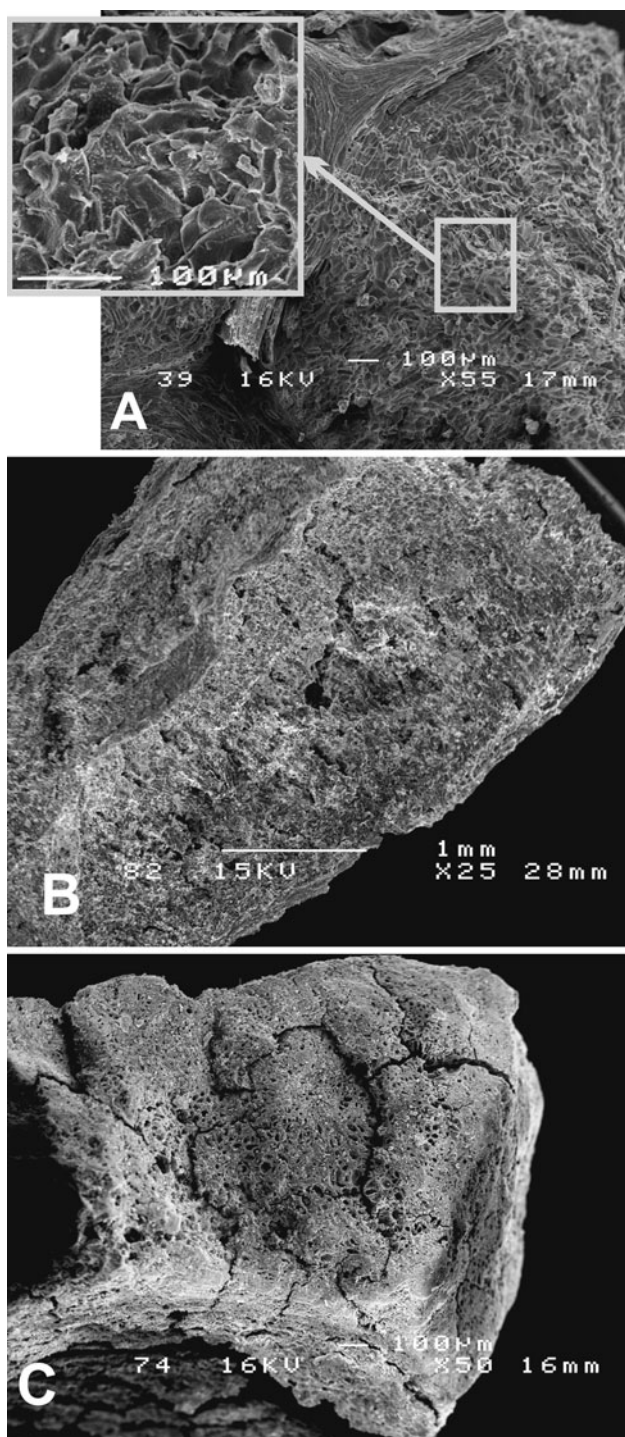


Fig. 7 a Overview and detailed image of an olive stone cross section surface, experimental, pre-charring breakage, non oxidising, 230°C; b overview image of an olive stone cross section surface, after experimental, pre-charring breakage, oxidising, 330°C. The piece is almost crumbling down to ashes; 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.

**Acknowledgments** The authors are grateful to Dagmar Wielgosz-Rondolino for collecting Italian olives. We also would like to thank to Freek Braadbaart for discussing with us the results of the experiments and to both anonymous reviewers for the very useful comments, which helped us to improve the paper.



## References

- Adam-Veleni P, Mangafa M (1996) Archeo electrivio sta Vrasna nomu Thessalonikis [Ancient olive factory at the site of Vrasa Thessalonikis, in Greek ELIA KE LADI. AE Elais, Athens, pp 92–104]
- Boardman S, Jones G (1990) Experiments on the effects of charring on cereal plant components. *J Archaeol Sci* 17:1–11
- Braadbaart F (2004) Carbonization of peas and wheat: a window into the past: a laboratory study. Dissertation, Universiteit Leiden. <http://www-old.amolf.nl/publications/theses/braadbaart/braadbaart.html>
- Braadbaart F, Poole I (2008) Morphological, chemical and physical changes during charcoalification of wood and its relevance to archaeological contexts. *J Archaeol Sci* 35:2,434–2,445
- Braadbaart F, Wright JP, van der Horst J, Boon JJ (2007) A laboratory simulation of the carbonization of sunflower achenes and seeds. *J Anal Appl Pyrolysis* 78:316–327
- Bretschneider J, van Lerberghe K (eds) (2008) In search of Gibala: an archaeological and historical study based on eight seasons of excavations at Tell Tweini (1999–2007) in the A and C fields. (Aula Orientalis-Supplementa 24) Editorial AUSA, Barcelona
- Foxhall L (2007) Olive cultivation in ancient Greece: seeking the ancient economy. Oxford University Press, Oxford
- Galili E, Stanley DJ, Sharvit J, Weinstein-Evron M (1997) Evidence for the earliest olive-oil production in submerged settlements off the Carmel Coast, Israel. *J Archaeol Sci* 24:1,141–1,150
- Gustafsson S (2000) Carbonized cereal grains and weed seeds in prehistoric houses: an experimental perspective. *J Archaeol Sci* 27:65–70
- Helbæk H (1970) The plant husbandry of Hacilar. In: Mellaart J (ed) Excavations at Hacilar. Edinburgh University Press, Edinburgh, pp 189–244
- Hopf M (1955) Formveränderungen von Getreidekorn beim Verkohlen. *Ber Dt Bot Ges* 68:191–193
- Kaniewski D, Paulsen E, van Campo E, Bakker J, van Leirberghe K, Waelkens M (2009) Wild or cultivated *Olea europaea* L. in the eastern Mediterranean during the middle-late Holocene? A pollen-numerical approach. *Holocene* 19:1,039–1,047
- Kislev ME, Rosenzweig S (1989) Influence of experimental charring on seed dimensions of pulses. *Acta Interdisciplinaria Archaeologica* 7:143–157
- Korber-Grohne U, Pienning U (1980) Microstructure of the surfaces of carbonised and noncarbonised grains of cereals as observed in scanning electron and light microscopes as an additional aid to determining prehistoric findings. *Flora* 170:189–228
- Mangafa M, Kotsakis K (1996) A new method for the identification of wild and cultivated charred grape seeds. *J Archaeol Sci* 23:409–418
- Margaritis E, Jones M (2006) Beyond cereals: crop processing and *Vitis vinifera* L. Ethnography, experiment and charred grape remains from Hellenistic Greece. *J Archaeol Sci* 33:784–805
- Margaritis E, Jones M (2008a) Crop processing of *Olea europaea* L.: an experimental approach for the interpretation of archaeobotanical olive remains. *Veget Hist Archaeobot* 17:381–392
- Margaritis E, Jones M (2008b) Olive oil production in Hellenistic Greece: the interpretation of charred olive remains from the site of Tria Platania, Macedonia, Greece (fourth-second century B.C.). *Veget Hist Archaeobot* 17:393–401
- Markle T, Rosch M (2008) Experiments on the effects of carbonization on some cultivated plant seeds. *Veget Hist Archaeobot* 17:S257–S263
- Neef R (1990) Introduction, development and environmental implications of olive culture: the evidence from Jordan. In: Bottema S, Entjes-Nieborg G, van Zeist W (eds) Man's role in the shaping of the eastern Mediterranean landscape. Balkema, Rotterdam, pp 295–306
- Riehl S (2009) Archaeobotanical evidence for the interrelationship of agricultural decision-making and climate change in the ancient Near East. *Quat Int* 197:93–114
- Salavert A (2008) Olive cultivation and oil production in Palestine during the early Bronze Age (3500–2000 B.C.): the case of Tel Yarmouth, Israel. *Veget Hist Archaeobot* 17:S53–S61
- Sarpaki A (1999) The archaeobotanical study of Tzambakas House, Rethymnon, Crete. In: Tzedakis Y, Martlew H (eds) Minoans and Mycenaeans: flavours of their time, Greek Ministry of Culture. National Archaeological Museum, Athens, pp 40–41
- Sievers C, Wadley L (2008) Going underground: experimental carbonization of fruiting structures under hearths. *J Archaeol Sci* 35:2,909–2,917
- Simchoni O, Kislev ME (2006) Charred by-products of olive-oil production in the Iron Age. In: Mazar A (ed) Excavations at Tel Beth-Shean 1989–1996, vol 1. The Israel Exploration Society and the Institute of Archaeology, Jerusalem, pp 679–685
- Valamoti SM (2002) Food remains from Bronze Age Archondiko and Mesimeriani Tomba in northern Greece? *Veget Hist Archaeobot* 11:17–22
- Valamoti SM, Samuel D, Bayram M, Marinova E (2008) Prehistoric cereal foods from Greece and Bulgaria: investigation of starch microstructure in experimental and archaeological charred remains. *Veget Hist Archaeobot* 17:S265–S276
- Warnock P (2007) Identification of ancient olive oil processing methods based on the olive remains. BAR International Series S 1635. Archaeopress, Oxford
- Wilson DG (1984) The carbonization of weed seeds and their representation in macrofossil assemblages. In: van Zeist W, Casparie WA (eds) Plants and ancient man: studies in paleoethnobotany. Balkema, Rotterdam, pp 201–206
- Winton AL, Winton KB (1932) Fruit of the olive family. The structure and composition of foods, vol 1. Cereals, starch, oil seeds, nuts, oils, forage plants. Wiley, New York, pp 587–597
- Wright PJ (1998) The making of the carbonized macrobotanical record. Dissertation, Washington University
- Wright PJ (2003) Preservation or destruction of plant remains by carbonization. *J Archaeol Sci* 30:577–583