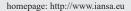


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Charred Organic Material, Heated by Anthropogenic Fires and Hot Volcanic Products from the Minoan Eruption, Excavated from the Bronze Age Site of Akrotiri on the Cycladic Island of Thera (Greece)

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ARTICLE INFO

Article history:

Received: 24th July 2018 Accepted: 16th December 2019

DOI: http://dx.doi.org/ 10.24916/iansa.2019.2.3

Key words:
Thera – Akrotiri
Cycladic Period
Bronze Age
charcoal analyses
reflectance analyses
burning temperature
tephra

ABSTRACT

The Bronze Age settlement site of Akrotiri, situated on the island of Thera in the Aegean Sea (Greece), developed during a period of over 1500 years into a flourishing city. This process started from the Late Neolithic through the Early and Middle Cycladic periods to the beginning of the Late Cycladic period when at a date in the late seventeenth century BC the city was buried and at the same time preserved by four phases of hot tephra released from the Minoan eruption. The investigations covering the archaeological excavations showed the remains of the fuels used for the fires lighted by the occupants of the city i.e. charred organic materials (COM) and ash. The volcanological part of the investigations studied the influence of the heat generated by the hot tephra on the wooden construction material, incorporated in the buildings, when they were covered and heated by the tephra. By measuring the reflectance on the charred organic material, the temperatures at which they were heated in the past were determined by applying the existing calibration curves. The results provided very interesting information about the function of the fires and the type of fuel resource selected by the occupants. The elemental analyses and the opal phytoliths from the ash provided additional information. The emplacement temperatures measured for the various phases of the hot tephra ranged from 310-340 °C for phase one, from 370-410 °C for phase two and is around 500 °C for phase four. It is interesting to note that the black charred material appeared not always to be charcoal.

1. Introduction

The Bronze Age settlement of Akrotiri is situated on the southern coast of Thera, a volcanic island, belonging to the Cyclades, in the Aegean Sea (Greece). From an archaeological point of view the Bronze Age is in this region divided into three major periods *i.e.* Early Cycladic (EC) (3000–2100 BC), Middle Cycladic (MC) (2100–1600 BC) and Late Cycladic (LC) (1600–1100 BC). The huge Plinian (Minoan) eruption buried and at the same time preserved the settlement by layers of hot tephra ejected from the volcano. The date of the eruption has been much debated in the literature and has led to a dispute between the approaches of conventional archaeology and scientific archaeology. Based

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on recent information a late seventeenth BC date seems now to be accepted (see among others: Manning *et al.*, 2014, p.1176; MacGillivray, 2014). The ejected tephra layers are divided into a precursory phase followed by four main phases of the eruption each with its characteristic composition (Figure 1). Only the precursory and the two following phases are present in Akrotiri due to their erosion. Many studies deal with these phenomena and the reader is referred to these studies and the references cited therein (Bond and Sparks, 1976; Heiken and McCoy, 1984; Druitt *et al.*, 1989; Heiken *et al.*, 1990; Sparks and Wilson, 1990; Friedrich *et al.*, 1990; McCoy and Heiken, 2000; Friedrich and Heinemeier, 2009; Friedrich, 2013).

Systematic archaeological excavations at Akrotiri started in 1967 by Marinatos and are continued to this day by Christos Doumas. The data shows that already since, at least, the Late

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STRATIGRAPHY ERUPTION ACTIVITY AND PHASE

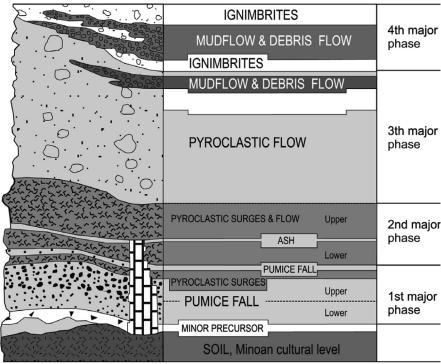


Figure 1. Stratigraphy of the Minoan eruption deposits. The wall shown in the lower part of the section represents the human settlements of Akrotiri archaeological site that interacted only with the first and second phases of the eruption (figure redrawn from McCoy, Heiken, 2000).

Neolithic, and continuously in the EC and MC periods the settlement was occupied and gradually developed into a flourishing city at the end of the MC (Doumas, 2012). At the time of the eruption and its destruction, in the beginning of the LCI period, the city was inhabited by a very affluent society that built multi-story buildings with magnificent wall-paintings, drainage systems, paved street and even toilets.

The archaeological excavations showed that during all the periods of occupation charred organic material (COM) was present and recovered from the settlements. During the EC and MC periods the heat necessary to char the organic material was generated by fires induced by the occupants for heating, cooking or other pyro-technological purposes, such as metal-working. The fires and heating continued in the LC I period before the city was buried by the volcanic hot tephra of the Minoan eruption. However, around 50 years preceding the Minoan eruption seismic activities in the form of earthquakes had ravaged the city several times in the MC period causing severe destructions to the buildings (Palyvou, 2015; McCoy and Heiken, 2000). As a result, the then present streets and ground floors were covered with a layer of debris consisting of building material: the volcanic destructing level (VDL), consisting of building material, with a thickness in the order of 1 to 2 metres. (Figure 2), and the city underwent major architectural changes as they

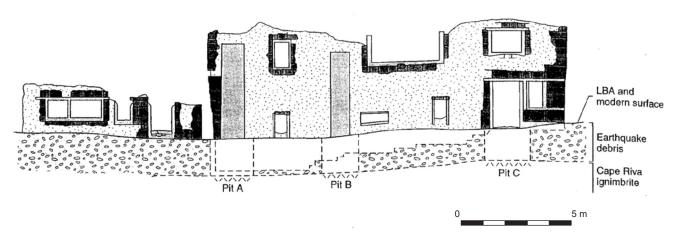


Figure 2. West House after the Minoan eruption with underneath the Volcanic Destruction Level (VDL).



incorporated the rubble and the streets became higher than the ground floors. This made ground floors of some buildings, especially along Telchines Road, become basements or semibasements, after the architectural restructuring of the town in MC III. Subsequently, the thin layer of precursory material from the Minoan eruption, consisting of hot pumice, covered and penetrated the VDL. Moreover, the buildings and their many wooden construction parts (Palyvou, 2005) became covered by the hot tephra. The result was that the wooden construction parts became heated and since they were shut off from air became charred as well. There is no indication that the city would have been on fire by the combustion of the wooden material, which confirms the charring. Thus, in addition to the heat sources of the fires made by the occupants of the city for different pyro-technological purposes, now a second heat source, in the form of hot pumice, afflicted them.

The important point is that charred organic material (COM) heated by two heat sources provides a unique opportunity to investigate questions that may arise from archaeological as well as volcanological origin. Archaeological information about the types of fires and fuel type, used by the occupants, could be obtained. Emplacement temperatures could be measured from the different phases of tephra ejected from the volcano.

For this purpose, the heated and subsequently charred materials are studied under reflected light, which makes it possible to determine the type of material and by measuring the reflectance, the temperature at which it had been exposed to, in the past. As fuel for the fires various organic materials could have been used including wood, charcoal, olivepressing residue and, probably, animal dung (Braadbaart and Poole, 2008; Sarpaki and Asouti, 2008). The material excavated from the older periods, i.e. EC and MC, was not affected by the heat of the hot volcanic material, so here fuel type and fire type can be investigated, per se, in those levels. However, the charred material found on the occupation/ destruction floor, in the LCI period, could have been heated by both heat sources. The temperatures measured though on the charred wood used as building material may provide the possibility to measure the emplacement temperature of the relevant tephra.

To facilitate this type of research, reference materials were used of experimentally-charred, modern samples (Braadbaart, 2004) all heated at a range of temperatures, with and without air, under controlled conditions in the laboratory. Hereby are the calibration curves of angiosperm and conifer wood prepared by Braadbaart and Poole (2008) and those prepared for olive stones (Braadbaart *et al.*, 2016) showing the reflectance (%Ro) as a function of the temperature (Figure 3). To get more information related to the type of fuel ash from different modern fuel resources this was investigated (Braadbaart *et al.*, 2016). For this purpose, the elemental composition was analysed and the presence of silica phytoliths was further investigated.

The archaeological objective of this study is to investigate if more information can be obtained about the fire structures lit by the inhabitants: such as their function and the type of

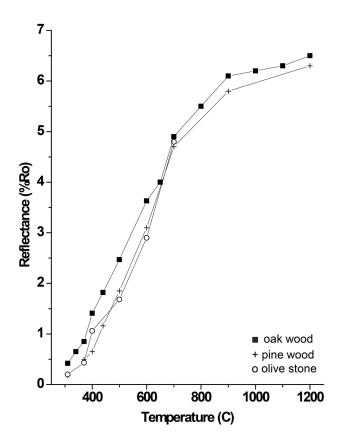


Figure 3. Mean reflectance measurements (%Ro) taken from modern olive stones (open circles) compared to modern oak (solid squares) and pine wood (crosses) samples experimentally heated under reducing conditions. Plotted as a function of the final temperature (°C).

fuel resource used. The results may further provide insights into the workings of a particular society and the landscape it operated in and will provide information pertaining to the control and application of heat (pyro-technology) as used in past societies.

The objectives for volcanology are the emplacement temperatures measured on the charred material that will be compared with investigations performed on lithics and pottery by palaeo-magnetic methods. It is believed that the results of the investigations for these two disciplines *e.g.* volcanology and archaeology, will provide additional information about the site of Akrotiri in general.

2. Material

The material for this study consists for the greater part of samples of charred organic material (COM), *i.e.* charred wood (charcoal) and charred olive stones. In addition, two samples of fuel ash and two samples of pumice were investigated. Regarding the charred material, a distinction has to be made between samples heated by human fires and those heated by hot volcanic material, the tephra. Samples heated by human fires were selected from the EC and MC periods as being representative of the whole collection of



Table 1. Charred wood (charcoal) and charred olive stones from EC and MC periods heated by anthropogenic fires. The temperatures are based on calibration curves by Braadbaart, and Poole, 2008.

Sample No.	Taxonomic status	Excavation sample code	Mean reflect (%Ro)	SD	N	Temp. after 60 min heating (°C)	Period
178CC	Charcoal	M4/64N005	1.000	0.179	100	380	EC
667CC	Charcoal	M4/64(AEN015	1.266	0.233	100	390	MC
860CC	Charcoal	M12/64NN015	0.850	0.050	100	370	MC
1183CC	Charcoal	M22/64N033	1.060	0.176	100	380	EC
580CC	Charcoal	M13/61N025	0.970	0.180	100	380	EC
746CC	Charcoal	M26/61N048	0.957	0.102	100	380	EC
178OS	Olive stone	M4/64N005	0.982	0.169	100	390	EC
667OS	Olive stone	M4/64(AE)N015	0.937	0.076	100	390	MC
806OS	Olive stone	M5/64N006	1.366	0.090	80	470	MC
1183OS	Olive stone	M22/64N033	1.481	0.112	100	490	EC
580OS	Olive stone	M13/61N025	0.919	0.124	100	390	EC
745OS	Olive stone	M25/61N047	1.093	0.104	100	390	EC

Table 2. Charred organic material heated in LC period by anthropogenic fires and occasionally heated again by hot material ejected from the Minoan eruption. The temperatures are based on calibration curves by Braadbaart, and Poole, 2008.

923	Olive stone	M14/65N024	0.977	0.079	100	400	LC
715	Olive stone	M8/65N007	0.696	0.090	100	380	LC
7-10	Olive stone	Rm#5 WH	0.284	0.084	100	330	LC
8-12	Charcoal	M7/54N003	0.583	0.131	88	340	LC
5-10	Charcoal	M11/6NO43	0.685	0.085	100	340	LC
1-11	Charcoal	M12/65N87	0.250	0.086	100	300	LC
18-12	Charcoal	M6/68N034	2.485	0.372	100	500	LC
2-11	Barley seed	M11/1BN	0.279	0.077	100	300	LC
6-10	Seeds	Rm#6 WH	0.368	0.115	100	300	LC
4-10	Dung	M35/43NO49	0.350	0.600	100	310	LC

Table 3. Reflectance measurements on charcoal samples recovered from imprints in walls of buildings above the ground level. Samples have been affected by the hot tephra of the Minoan eruption. For situation see Figure 4. Taxonomic status of wood: angiosperm. Dates of excavation 25-5-2010 and 05-10-2012.

	Location of sample in wall	Mean %Ro	SD	N	Temp. (°C)
1-10	Sector gamma Rm #1 or 2	0.850	0.122	100	410
2-10	House of beautiful pottery (gamma)	0.800	0.102	100	400
3-10	Sector gamma Rm #2a	0.760	0.098	100	390
1-12	Triangle square	0.906	0.126	100	370
2-12	West House Rm #3a	0.327	0.217	100	300
3-12	West House stairs to 1st floor	0.457	0.097	100	320
4-12	Arvanites square (Sector Alpha) Rm #2	0.473	0.130	100	320
5-12	House of the Ladies	1.206	0.189	100	390
6-12	House of the Ladies south	0.941	0.158	100	370
7-12	House of the Ladies Rm #8	0.583	0.131	100	330
9-12	Delta Rm # 16	1.038	0.149	100	380
10-12	Beta Rm#1	0.559	0.168	100	330
11-12	Beta Rm#2	0.368	0.115	100	300
13-12	Delta Rm#1a	0.479	0.086	100	290



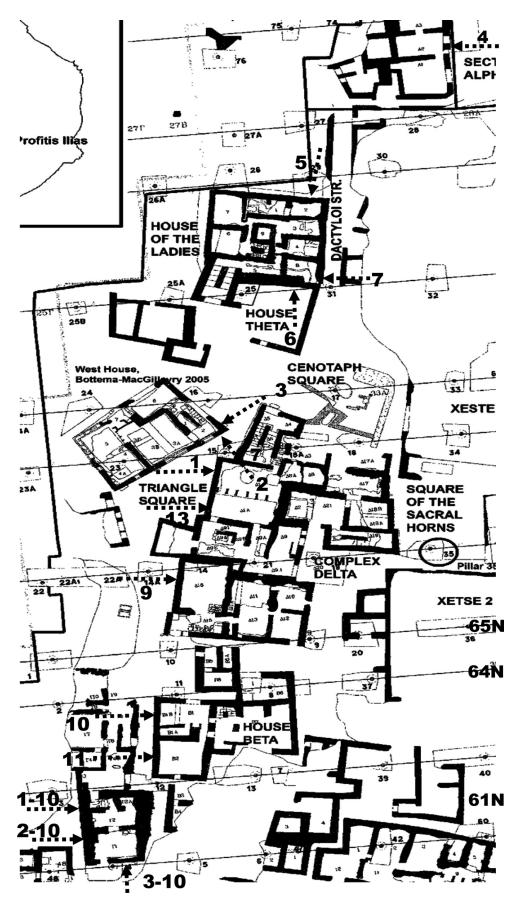


Figure 4. Situation of the investigated imprints plotted on a map of Akrotiri. For the samples 1-12, see Table 3. Sample nr.12 is not shown on the map. Black arrows indicating the situation (2-10).



Figure 5. Example of two imprints (arrows) in the wall of a building, being the original place of the wooden construction beams.

excavated samples that is kept in the store room at the site (Table 1). For the LC period, samples were also selected from the store room that could have been heated by human fires (charred olive stones) as well as by hot tephra (seeds in pots) (Table 2). The ash samples from the store room are from the LCI period. Samples of wooden construction parts heated by the hot tephra were located and collected by the authors from imprints in the walls of the buildings (Table 3, Figure 4), holes that originally kept the wooden beams or other wooden construction material. For the situation of these buildings see Figure 5. These imprints and the charcoal samples found in these hollows were situated at a height of around 1.50 m above the actual floor, but it is not clear if this was the VDL or the original living floor (Figure 5). Pumice samples were taken from sector gamma by the authors.

In addition, samples were obtained from outside the city of Akrotiri: two charred olive wood samples from the Caldera wall (see Friedrich, 2013 for exact location) collected by the authors and two charcoal samples from the site of Megalochori donated by Mr. Lefteris Zorzos.

3. Methods

3.1 Reflectance on charred organic material

The charring process and its governing variables, as well as the application of reflectance measurements, have been described extensively in earlier studies and the reader is referred to the relevant publications (Braadbaart, Poole, 2008; Braadbaart, Wright, 2007). However, all vitrinite reflections for establishing the maximum heating temperature of the



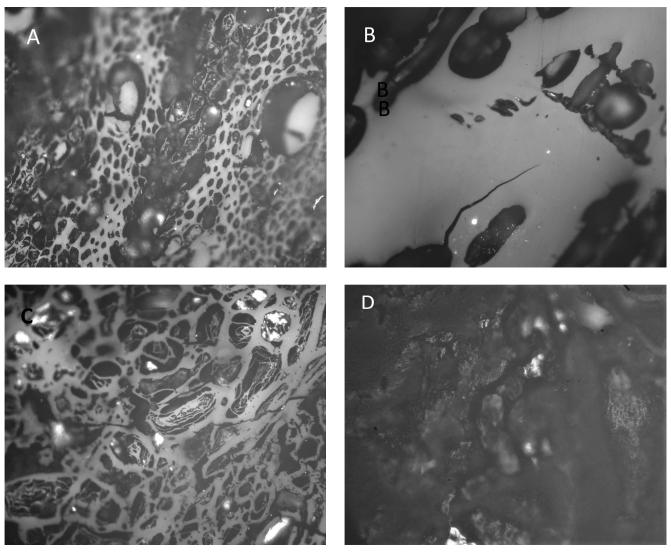


Figure 6. Microphotographs under reflected light microscopy of charred organic material (COM) heated under reducing conditions. Cell walls and other charred material: A=178CC angiosperm wood; B=178CC, no cells homogeneous material; C=806OS olive stone; D=M35/43NO49, animal dung.

samples were measured according to ISO 7404-5 (Methods for the petrographic analysis of coals – Part 5: Method of determining microscopically the reflectance of vitrinite). All measurements were individually calibrated against the proper reflectance standards. (N.E.N. 2009).

Temperature and time of exposure determine the charring process. For wood and olive stones the effect of the temperature has been studied in earlier studies and therein a time of exposure of 60 minutes was applied (Braadbaart *et al.*, 2016). For this study the effect of time was studied on charred angiosperm wood (Figure 7). It shows that until around 450 °C the time of exposure does not have an important effect on the reflectance and thus the temperature. And it was therefore decided to continue the application of an exposure time of 60 minutes for this study, as was used in earlier studies.

The accuracy of the reflectance measurements is determined by the standard deviation of (at least) 50 measurements per sample. The given reflectance value is the average of these 50 measurements. The standard deviation generally ranges between 0.05%R. The accuracy of the temperature is determined by the accuracy and stability of the oven in which the original material was heated (Laura *et al.*, 2009; Pensa *et al.*, 2015; Veal *et al.*, 2016).

It is noted that the reflectance measured on the samples represents the highest temperature at which they were ever exposed to in the past. This means that reheating at a lower temperature does not affect the reflectance.

3.2 Elemental analyses

From each ash residue, five samples were measured, and the mean of the normalized (corrected for organic compounds) results was calculated. The equipment used was the HH x-ray fluorescence (XRF) Thermo Scientific Niton XL3t device with GOLDD detector equipped with a silver anode operating at a maximum of 50 kV and 40 μ A. This handheld device was used in the laboratory, and the samples were measured on a stable flat surface. This device is well suited for measuring up to 25 elements simultaneously in the analytical range between sulphur (atomic number 16) and

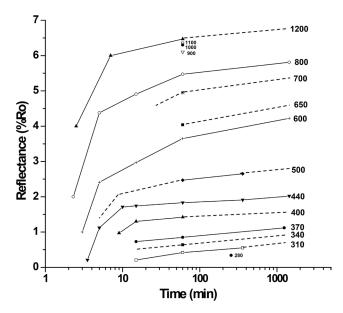


Figure 7. Mean reflectance (%Ro) of modern charred organic material (COM) as function of time.

uranium (atomic number 92). Light elements (magnesium, aluminium, silica and phosphorous) can also be measured with the same detector using a helium purge. The samples were measured in bulk mode. The device was factory calibrated.

3.3 Opal phytoliths

For the microscopic study, 4 mg of each ash sample was thoroughly mixed with 2 mL of deionized water. From each

mixture, 0.05 mL was mounted on a glass slide. In this way, each slide contained an equal amount of ash, that is, 0.1 mg, which made comparison between the different ashes possible. The slides were examined using trans-illumination under a Leica DM6000 M microscope.

3.4 pH measurement on pumice

The pH measured on the two pumice samples followed the specifications outlined in NEN 5750, 1989 and for the measurements a Consort D514 digital pH meter was used.

4. Data

4.1 Material excavated from the EC and MC periods

Samples of charcoal and charred olive stones from the new trenches N61 and N64 were analysed under reflective light.

For the charred wood, the characteristic cell walls of angiosperm wood were observed (Figure 6A). It is noted that in some samples, the cell-wall material from numerous cells seemed to have been transformed into a homogeneous mass showing no cells and clearly different from the characteristic wood-cell structure (Figure 6B). The mean values of the reflectance readings for these fragments ranged from 0.850 to 1.266 %Ro (SD = 0.05–0.18 %Ro). These values correspond to temperatures in the range of 370–390 °C (Table 1).

The charred olive stones show their characteristic cells (Figure 6C). The mean values of the reflectance readings for this material range from 0.919 to 1.481 %Ro (SD = 0.076-0.169 %Ro). These values correspond to temperatures ranging from 390 to 490 °C (Table 2).

Table 4. Reflectance measurements on charcoal samples from locations outside the city of Akrotiri. Samples have been affected by the hot tephra of the Minoan eruption. The temperature is based on calibration curves by Braadbaart, and Poole, 2008.

Sample No.	Location of sample	Mean %Ro	SD	N	Temp. (°C)
OT 1	Olive tree in Caldera wall	0.430	0.600	100	290
OT 2	Olive tree in Caldera wall	0.255	0.087	100	280
O1	Megalochori DR/ER/3	1.626	0.309	100	420
O2	Megalochori DR/ER/5	3.149	0.354	100	580

Table 5. Mean normalized elemental analyses of five specimens of 2 samples of chemically untreated ashes in wt%.

Sample excavation code	M7/65N009	M6/68N034	
SiO2	34.7	33.5	
CaO	9.6	2.2	
P2O5	3.2	0.7	
K2O	2.3	2.0	
Al2O3	5.6	6.0	
TiO2	0.3	0.3	
Fe2O3	3.3	3.6	
Balance	39.8	51.3	
SiO2/CaO ratio	3.5	18	



4.2 Material from the LC period

4.2.1 Reflectance

Different samples of COM, including wood, olive-pressing residue, seed and dung were analysed (Table 2). These samples showed their particular characteristic cell walls under reflective light. For charred wood the variation in reflectance is large and varies from 0.250 to 2.485 %Ro (SD = 0.085–0.372 %Ro) resulting in temperatures ranging from 300 to 500 °C. The samples of charred olive stones also show this variety and the temperatures vary from 330 to 400 °C. The two seed samples were heated at around 300 °C. Among the available samples only one sample of possible charred dung was found and analysed, whereby a temperature of 310 °C was measured (Figure 6D).

4.2.2 Elemental analyses

The only two ash samples we analysed contained 34.7 and 33.5 wt% SiO2, while the calcium content was 9.6 and 2.2 wt% CaO, respectively (Table 5). This resulted in a silica/calcium ratio of 3.5 and 18.

4.2.3 Opal phytoliths

In both samples the dendritic long cells strongly dominate each slide (Figure 8). These can be considered as the

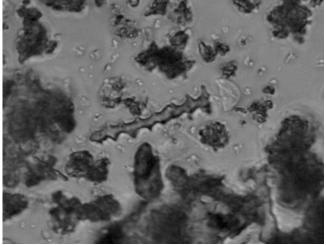
characteristic morphotypes of cereals (Ball *et al.*, 2009). Further, the presence of phytoliths originating from olive stones is possible, but were not observed (Braadbaart *et al.*, 2016). No typical grass phytoliths were observed. Detailed analyses about the number of phytoliths or the taxa of cereals were not performed.

4.2.4 Material recovered from wood imprints in the walls of buildings

These samples show the characteristic cell walls of angiosperm wood as well as conifer wood under reflective light. Based on the measured temperatures two separate groups can be distinguished. For one group of samples the reflectance was around 1.000 %Ro with temperatures ranging from 370to 410 °C. A second group showed a reflectance of around 0.500 and the corresponding temperatures range from 310 to 340 °C (Table 4).

4.2.5 Samples from the island of Thera, but beyond the site of Akrotiri

The two samples of charred olive wood from the Caldera wall and deposited in pumice showed reflectances of 0.430 and 0.255 %Ro (SD=0.600 and 0.087 %Ro), which correspond to a temperature of around 300 °C (Table 4). The two charred samples from the excavation of Megalochori



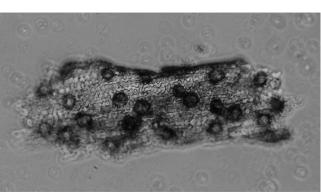
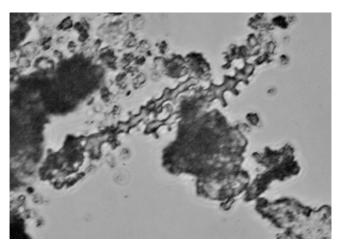
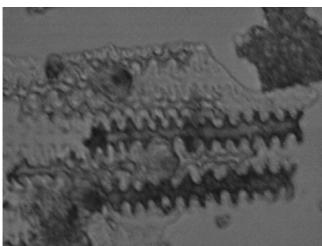


Figure 8. Microphotographs of examples of dendritic morphotypes of opal phytoliths of cereals recovered from archaeological ash. (See also Ball *et al.*, 2009, Figure 1).







show the characteristic cell walls from angiosperm wood. Here the reflectances were rather high with values of 1.626 and 3.149 %Ro (SD=0.309 and 0.354 %Ro), giving rather high temperatures of 420 to 580 °C, respectively.

4.2.6 *pH of the pumice recovered from sector gamma* The results of the two samples showed values of 8.5 and 9.05.

5. Discussion

This study is mainly focused on the charred organic materials (COM) excavated from the site of Akrotiri and although it is a rather small data set, it reveals interesting information which would need to be further explored and enriched in the future. For example, at Akrotiri, to char organic material like wood or olive stones, it has to be exposed to heat under reducing conditions. On this site, two heat sources, generating the required heat, made this possible. To begin with, small fires ignited by the occupants of the site, such as hearths, which they used for their daily need to prepare food, to light up, or induced by other pyro-technological sources, we name: the **anthropogenic heat**. A second source of heat was the hot tephra ejected from the volcano during the Minoan eruption, which buried the site: the volcanic heat. The charred wooden construction material within the architecture makes it possible to measure the reflectance of this charred material and accordingly the emplacement temperatures of the various volcanic materials from the relevant phases, as ejected by the volcano.

5.1 Anthropogenic heat source

In the EC, MC and LC periods, charred wood or charcoal and charred olive stones were excavated (Sarpaki and Asouti, 2008). In such archaeological sites it seems that these materials were often charred when used as fuel in fireplaces. To initiate a fire, an interaction has to take place between the fuel, air and the heat generated by an external heat sourcethe three basic elements of the fire triangle (Emmons and Atreya, 1982). Fuel is composed of water, an inorganic ash fraction and an organic fraction, the latter providing the potential energy that can be transformed into the required heat energy. Once a heat source has been introduced to the fuel, heat is absorbed and the fuel begins to increase in temperature. When a temperature of 280-300 °C is reached with no air, a complex chemical reaction starts that leads to the thermal degradation of the organic constituents of the fuel, producing volatile gases and a carbon rich or charred residue (Rein, 2009; Braadbaart et al., 2012). When enough air is available at this temperature and higher, both new products will oxidize, a highly exothermic process, meaning that heat will be generated. As long as the temperature remains above 300 °C and with enough air, the volatile gases will produce flames and the carbon-rich char will be converted into carbon dioxide (CO₂) and a residue of ash, both reactions releasing heat. When enough air is not made available, the temperature will decrease and the fire will extinguish. Under these conditions not only the char, but also the ash provide indications of the original type of fuel and possibly the heating conditions of the fire, which in turn may give an indication of the function of the fire. The results show that wood, olive stones and animal dung must have been available to meet the fuel demands of the occupants. The question now arises as to whether there were enough of these fuels available on the island. Based on the available literature it is not clear how the pre-eruption surface of the island looked like. In Assouti (2003, p.472), a wooded landscape and early olive cultivation has been described. However, in the work of Sarpaki and Asouti (2008, p.370) and Bottema-MacGillivray (2005), a certain level of thrift, or rather of economizing, in wood has been indicated. On the other hand, wood was used extensively as a building material, although it only survived in very few places in Akrotiri (Palyvou, 2005, p.111). It is also possible that some timber was imported, in turn, perhaps, suggesting that not enough suitable wood was available on the island (Palyvou, 2005, p.112). Therefore, the fuel issue, as further discussed, is only based on the investigations on the material and their samples used in this paper.

In an earlier paper it was shown that the olive-pressing residue (OPR) is an excellent fuel (Braadbaart et al., 2016). After pressing the olives, two types of fuel can be prepared: (1) after air drying OPR can be used directly as a fuel and (2) OPR is thermally degraded by heating, in the absence or near absence of air, in the range of 400-500 °C. The charred OPR is now upgraded into a more efficient type of fuel (COPR) with a heating value that increases by 50% (Braadbaart et al., 2017). The weight (mass) of the fuel is also considerably reduced, which facilitates transportation. Thus, a hotter and smoke-free fire is created, compared to a fuel that is just air dried and not thermally degraded as explained above. The temperatures of around 400 °C measured on the charred olive stone samples could be a confirmation that COPR was indeed used as fuel (Table 1a). In the case of the charcoal samples the temperature is 380 °C, somewhat lower compared to the olive stones in spite of the fact that both types of samples were retrieved from approximately the same spot, i.e. 178CC and 178OS in the EC period (Table 1b). Yet, even at this temperature, a similarly efficient fuel like COPR could have been produced from wood, although the quality might have been less in the sense of its physical strength. Since the reflectance just shows the highest temperature at which the charred material was heated, it can be argued that olive stones and wood were thermally upgraded or charred under the described conditions outside the city before being used as fuel by its occupants. Thus, the measured temperatures of 380 to 400 °C are not the temperatures necessary for the purpose of the fires. They are too high for fires that were probably only used for cooking where a temperature of around 300 °C or even less is normally more than sufficient, which would mean that less fuel would be used. It is, therefore, tentatively suggested that the more efficient thermally-upgraded COPR and wood had been used as fuel. It could have been upgraded at Akrotiri, or on Thera itself, where enough olives and/or



woody material were available. Subsequently, the light-weight, thermally-upgraded materials would have been transported to the city from its vicinity and, perhaps, along with the possible importation of wood from the macroenvironment of the island and/or from other areas further afield, used for the construction of houses (Palyvou, 2005, p.112). On the line of these arguments, one notes that already in the EC period this system would have been applied for the production of fuels, a rather advanced technology. However, it cannot be excluded that the higher temperatures were used for other activities where higher heat was needed, such as metalworking, faience and so forth.

For the LC period, in principle, the same explanation is valid as discussed above, but the process becomes more complicated to explain, since the material was suddenly buried by hot pumice, *i.e.* the volcanic heat source. In order, to study this process a number of characteristic samples of different materials were selected and analysed (Table 2). The results show that the olive stone samples 715 and 923 were heated at temperatures of 400 °C, which is comparable to the samples from the EC and MC periods. Apparently, these samples were not affected by the heat of the hot pumice, which must have had a lower temperature. The temperatures of the other samples are much lower and are further discussed in a following section describing the volcanic heat source.

5.2 Ash

In addition to the charred material, two samples of ash from the LCI period were investigated (Table 5). The results show that hardly any calcium was present, indicating that wood was not burned as fuel (Braadbaart et al., 2017). Moreover, sample 4-10 found in the vicinity did not show the typical cell walls of wood and is attributed to, probably, animal dung (Sarpaki and Asouti, 2008). As far as the opal phytoliths are concerned, the dendritic morphotypes strongly dominate the samples and are characteristic of cereals (Ball et al., 2009). Assuming that animal dung could have been used as one of the fuels, it is suggested that harvest residues of cereals were used as feed for goats and in turn goat dung could have been used as fuel (Owen, 1994), especially as cereals were grown on the island. The use of animal dung as fuel would be in accord with the possible findings in Sarpaki and Asouti (2008). No typical grass phytoliths were found, so dung from other animals that feed on grass were not used as fuel in that particular hearth. However, the study of fuel remains an open question at Akrotiri, which needs to be further studied more closely in the future (Shahack-Gross, 2011).

5.3 Volcanic heat source

After the thin layer of precursory material, the first phase of the Minoan eruption deposited a layer of hot pumice on the city. It buried the VDL layer and spilled through windows and doors. The roofs were loaded with the pumice and some collapsed. The second phase consisted of hot pyroclastic surges and flows. This caused probably less damage, since it may have flowed along the buildings through the streets. The third phase does not seem to be present in Akrotiri, but there

are indications that the fourth phase consisting of ignimbrites is present in Akrotiri (Druitt, 2014).

The organic material got buried by the hot tephra and accordingly was heated and also charred since no air was present between the reacting surfaces of the tephra and the organic material. The temperatures based on the reflectance measurements of the charred wood samples recovered from the post holes show that two groups of temperatures can be distinguished (Table 3). One group was heated and charred at 310 to 340 °C and a second group at 370 to 410 °C. Apparently, these groups represent the first two phases of tephra, each with different emplacement temperatures. Samples presented in Table 2 also show temperatures that are comparable to the samples of the first group excavated from the post holes. Olive stone sample 7-10 and the two samples of seeds (2-11 and 6-10) were recovered from pots found on the ground floor of the West House building and it is not very likely that they were charred before being deposited in the pots. Since they were charred it can be concluded that they were heated by the hot pumice at a temperature of around 320 °C. The beam from shaft 65N (sample 1-11) must have been part of the building material and was heated at its outside by the hot pumice at a temperature of 300 °C. The two charcoal samples (5-10 and 8-12) were charred at 340 °C. The samples of charred olive wood recovered from the wall of the Caldera and covered by pumice showed a temperature of 310 °C. This temperature is comparable to the samples from Akrotiri and may suggest that the emplacement temperature of the first phase consisting of the pumice was in the range of 300–340 °C. It is suggested that the second group of charred material from the post holes represents the second phase of tephra with temperatures of 370 to 410 °C. In relation to their height, the exact location of the samples from the post holes is confusing, since the original level is not clear. Charcoal sample 18-12 was charred at 500 °C, which is considerable higher than the samples heated by the tephra of the first two phases. However, the two charcoal samples from the site of Megalochori show comparable temperatures of 420 and 580 °C. Temperatures like these are also measured in the ignimbrites of phase 4 (McCoy, Heiken, 2000, p.55 and Druitt, 2014). This would mean that phase four of the volcanic deposits must have had some effects in Akrotiri.

Earlier studies that have measured emplacement temperatures used magnetic methods (Bardot, 2000 and references cited therein). The measurements were applied on lithics found on various places on the island of Thera. For Akrotiri, magnetic methods have been applied on lithics as well as pottery (Tema *et al.*, 2013; 2015). The results show measurements that were in the order of 260 to 280 °C for material covered by the pumice of the first phase, but higher temperatures to the order of 310 °C were also shown at a few places. The temperatures, therefore, measured by magnetic methods on the inorganic material are lower than the temperatures found on charred organic material charred by tephra studied in this paper. For organic material to be charred the temperature has to be at least 280 to 300 °C and



these materials become, as a result, always black. Since the reflectance measure only the highest temperatures, the samples that were even exposed to a possible cooling of the tephra after deposition, will not change the reflectance and the temperatures. It is therefore tentatively suggested that the emplacement temperatures of the tephra of phases 1, 2 and 4 are as described above. The pH values of the pumice are such that no corrections for the reflectance are necessary (Braadbaart *et al.*, 2009).

In the literature no descriptions could be found of fires that have raged the city during or after the eruption. On the other hand, hardly any wooden construction material used for the buildings was recovered. Only wood imprints were all that was found, indicating where wood must have been present (Palyvou, 2005, Figure 5). An explanation for this phenomenon could be that the buildings and thus also the wooden material were buried by substantial layers of hot pumice without air. Therefore, as the wooden material was heated without air, as a result it became charred. Probably this process occurred over a limited time and only had an effect on the outside of the wooden beams (sample 1-11) and other architectural wooden parts, which means that the inside of the beams were not charred and have not survived the natural disintegrating processes that usually occur after deposition. Only the char formed on the outside was left along with some remnants that could still be recovered from the imprints.

6. Conclusions

The settlement of Akrotiri developed from the Late Neolithic through the Early and Middle Cycladic period into a flourishing city up to the beginning of the Late Cycladic period, covering a period of over 1500 years. Then, at a late seventeenth century BC date, as a result of the Minoan eruption, the city was buried and at the same time preserved by four phases of hot tephra. During this whole Cycladic period, fire structures were found and excavated that were most probably used for activities primarily concerned with the preparation of food. However, the practice of other pyrotechnologies should not be excluded. The charred organic materials recovered from these fire structures showed that the temperatures at which they must have been exposed to range from 380 to 490 °C (Table 1a). It is argued that such temperatures are too high for an activity such as cooking, as these temperatures consume too much fuel and the food may well burn. When olive-pressing residues and wood are thermally upgraded by charring at 400 to 500 °C, excellent fuels are produced (Braadbaart et al., 2016). This process could have been performed outside the city in places where enough olive trees and woody material were present. It probably explains both the presence of the charred olive stones and charcoal showing high temperatures. It has been noted from this study that already since the EC period this advanced technology was applied in the settlement. Another residue of burned fuel is the ash. Unfortunately, the very summary work and the few samples indicate, through some elemental analyses, the presence of opal phytoliths (Shahack-Gross, 2011) and some observations under reflective microscopy that animal dung could also have been used as a fuel resource.

When the city was buried by the first main phase of tephra, this being the hot pumice, the buildings and all the material they contained were exposed to this heat source. The wooden parts of the architecture were heated and covered and since no air was present anymore at the reacting surfaces, they became charred. The lack of air may explain why in the literature no indication of fires in the city after the eruption was found. By using this charred organic material, the temperature at which the material had been exposed to can be measured using the reflectance method. In this way the emplacement temperatures of the various phases of tephra can be determined. The results show that: for the first phase, the pumice, the temperature must have been between 310 and 340 °C; for the second phase, between 370 and 410 °C; and samples showing temperatures of around 500 °C are attributed to the ignimbrite of phase four of the tephra. These temperatures are somewhat higher than the earlier obtained temperatures using magnetic methods measured on lithics and pottery, both being inorganic materials. The applied methods are especially valid for the circumstances encountered in this archaeological and volcanological site. It was observed that not all black material was charcoal, but could be charred dung or other charred organic material. It is suggested that, apart from botanical identification, another important part of an archaeological investigation should be, as suggested in this study, the analysis of charred organic material and ash, as well as the remains of the various used fuels. This could add much valuable information related to not only how ancient societies selected the wide spectrum of their fuels for their pyro-technical needs, but also open up avenues of research into the technological know-how and those aspects of the environment and economy that can only be indirectly inferred.

Acknowledgements

We thank Ch. Doumas for allowing us to work and take samples at the site Akrotiri on Thera (Greece). For the preparation of phytolith microphotographs we thank Ton van Brussel of the Faculty of Science, Institute of Biology, Leiden University (The Netherlands). We would also like to thank Sampson Katsipis who walked with us and indicated the site on the edge of the caldera where Prof. Friedrich had found the olive tree trunk, as well as Lefteris Zorzos who provided the Megalochori samples. Last but not least, we thank Mieke Verheem, Freek Braadbaart's widow, for she has been instrumental in finding leads which helped with complying with the reviewers' suggestions and the completion of this work.



References

- ASOUTI, E., 2003. Wood charcoal from Santorini (Thera): new evidence for climate, vegetation and timber imports in the Aegean Bronze Age. *Antiquity*, 77, 471–484.
- BOND, A., SPARKS, R.S., 1976. The Minoan eruption of Santorini, Greece. J. Geol. Soc. London, 132, 1–16.
- BALL, T.B., EHLERS, R., STANDING, M.D., 2009. Review of typologic and morphometric analysis of phytoliths produced by wheat and barley. *Breeding Science*, 59, 505–512.
- BARDOT, L., 2000. Emplacement temperature determination of proximal pyroclastic depo sits on Santorini, Greece, and their implications. *Bulletin Volcanologica*, 61, 450–467.
- BOTTEMA-MACGILLIVRAY, J.N., 2005. Wood of the West House, Akrotiri, Santorini (Greece). *Palaeohistoria*, 45/46, 95–120.
- BRAADBAART, F., 2004. Carbonization of Peas and Wheat A Window into the Past: A laboratory Study. Leiden: Universiteit Leiden. Available at: https://amolf.nl/publications/
- BRAADBAART, F., WRIGHT, P.J., VANDERHORST, J., BOON, J.J., 2007. A laboratory simulation of the carbonization of sunflower achenes and seeds. *J. Anal. Appl. Pyrolysis*, 78, 316–327.
- BRAADBAART, F., and POOLE, I., 2008. Morphological, chemical and physical changes during charcolification of wood and its relevance to archaeological contexts. *Journal of Archaeological Science*, 35, 2434–2445.
- BRAADBAART, F., POOLE, I., and VAN BRUSSEL, A.A., 2009. Preservation potential of charcoal in alkaline environments: an experimental approach and implications for the archaeological record. *Journal of Archaeological Science*, 36, 1672–1679.
- BRAADBAART, F., POOLE, I., HUISMAN, H.D.J., and VAN OS, B., 2012. Fuel, Fire and Heat: an experimental approach to highlight the potential of studying ash and char remains from archaeological contexts. *Journal of Archaeological Science*, 39, 836–847.
- BRAADBAART, F. MARINOVA, E., SARPAKI, A., 2016. Charred olive stones: Experimental and archaeological evidence for recognizing olive processing residues used as fuel. *Vegetation History and Archaeobotany*, 25, 415–430.
- BRAADBAART, F., VAN BRUSSEL, T., VAN OS, B., EIJSKOOT, Y., 2017. Fuel remains in archaeological contexts: Experimental and archaeological evidence for recognizing remains in hearths used by Iron Age farmers who lived in peatlands. *The Holocene*, 27(11), 1682–1693.
- DOUMAS, CH., 2010. Akrotiri. In: Eric H. Cline, ed. *The Oxford Handbook of the Bronze Age Aegean*. New York: Oxford University Press, pp. 752–760.
- DRUITT, T.H. MELLORS, R.A., PYLE, D.M., SPARKS, R.S.J., 1989. Explosive volcanism on Santorini, Greece. *Geol. Mag.*, 126, 95–126.
- DRUITT, T.H., 2014. New Insights into the Initiation and Venting of the Bronze-Age Eruption of Santorini (Greece), from Component Analysis. *Bulletin of Volcanology*, 76, 794. https://doi.org/10.1007/s00445-014-0794-x.
- EMMONS, H., and ATREYA, A., 1982. The science of wood combustion. *Proc. Indiana Acad. Sci.*, 5, 259–268.
- FRIEDRICH, W.L., WAGNER, P., TAUBER, H., 1990. Radiocarbon dated plant remains from the Akrotiri excavation on Santorini, Greece. In: D.A. Hardy, and A.C. Renfrew, eds. *Thera and the Aegean World III*, Vol.3, London: The Thera Foundation, pp. 188–96.
- FRIEDRICH, L.W., HEINEMEIER, J., 2009. The Minoan eruption of Santorini radiocarbon dated to 1613±13 BC: geological and stratigraphic considerations. In: D.A. Warburton, ed. *Time's Up! Dating the Minoan Eruption of Santorini*. Aarhus: Monographs of the Danish Institute at Athens, pp. 57–63.
- FRIEDRICH, L.W., 2013. The Minoan Eruption of Santorini around 1613 B.C. and its consequences. *Tagungen des Landesmuseums für Vorgeschichte Halle*, 9, 37–48.

- HEIKEN, G., MC COY, F., 1984. Caldera development during the Minoan eruption, Thera, Cyclades, Greece. *J. Geophys. Res.*, 89(B10), 8441–8462.
- HEIKEN, G., MC COY, F., 1990. Precursory activity to the Minoan eruption, Thera, Greece. In: D.A. Hardy, J. Keller, V.P. Galanopoulos, N.C. Flemming, T.H. Druitt, eds. *Thera and the Aegean World III, Vol. 2*. London: The Thera Foundation, pp. 79–88.
- International standard ISO 7404-5:2009 (E): Methods for the petrographic analysis of coals —Part 5: Method of determining microscopically the reflectance of vitrinit.
- LAURA, C., MCPARLANT, M., COLLINSON, E., SCOTT, A.C., CAMPBELL, G., 2009. The use of reflectance values for the interpretation of natural and anthropogenic charcoal assemblages. *Archaeol. Anthropol. Sci.*, 1, 249–261.
- MANNING, S.W., HOFLMAYER, F., MOELLER, N. DEE, M.W. BRONK RAMSEY, C., FLEITMANN, D., HIGHAM, T., KUTSCHERA, W., WILD, E., 2014. Dating the Thera (Santorini) eruption: archaeological and scientific evidence supporting a high chronology. *Antiquity*, 88, 1164–1179.
- MACGILLIVRAY, A., 2014. A disastrous date. Antiquity, 88, 288-89.
- MC COY, F., and HEIKEN, G., 2000. The Late Bronze Age explosive eruption of Thera (Santorini), Greece: regional and local effect. *Geological Society of America*, Special paper, 345, 43–70.
- N.E.N. 5750, 1989. Netherlands Norm Soil.- Determination of pH in soil samples (in Dutsch).
- N.E.N. ISO 7404-5, 2009. Methods for the petrographic analysis of coals-Part 5: Method of determining microscopically the reflectance of vitrinite (ISO 7404-5:2009, IDT) (in English).
- OWEN, E., 1994. Cereal crop residues as feed for goats and sheep. *Livestock Research for Rural Development*. Volume 6(1), 1–15.
- PALYVOU, C., 2005. Akrotiri, Thera: An Architecture of Affluence 3,500 Years Old. Philadelphia Pennsylvania: INSTAP Academia Press.
- PENSA, A., PORRECA, M., CORRADO, S., GIORDANO, G., CAS, R., 2015. Calibrating the pTRM and charcoal reflectance (Ro%) methods to determine the emplacement temperature of ignibrites. Fogo A sequence, São Miguel, Azores, Portugal, as a case study. *Bulletin of Volcanology*, 77(3), 1–19.
- REIN, G., 2009. Smouldering combustion phenomena in science and technology. *International Review of Chemical Engineering*, 1, 3–18.
- SARPAKI, A., ASOUTI, E., 2008. A glimpse through a kitchen key-hole at Late Bronze Age Akrotiri: the organic remains. In: N. Brodie, J. Doole, G. Gavalas, C. Renfrew, eds. *Horizon: A colloquium on the prehistory of the Cyclades*. Cambridge: McDonalds Institute Monographs, pp. 370–376.
- SHAHACK-GROSS, R., 2011. Herbivorous livestock dung: formation, taphonomy, methods for identification, and archaeological significance. *Journal of Archaeological Science*, 38, 205–218.
- SPARKS, R.S.J., WILSON, C.J.N., 1990. The Minoan deposits: a review of their characteristics and interpretation. In: D. Hardy, J. Keller, V.P. Galanopoulos, N.C. Flemming, T.H. Druitt, eds. *Thera and the Aegean World III.* Vol. 2, The Thera Foundation, pp. 89–99.
- TEMA, E. PAVLIDES, S., KONDOPOULOU, D., 2013. Late Bronze age pottery as indicator of the deposition temperatures of the Minoan pyroclastic products, Santorini, Greece. In: *Bulletin of the Geological Society of Greece*, 43(3), Proceedings of the 13th International Congress, Chania, September 2013, pp. 1289–1297.
- TEMA, E., ZANELLA, E., PAVÓN-CARRASCO, F.J., KONODOPOULOU, D., PAVLIDES, S., 2015. Palaeomagnetic analysis on pottery as indicator of the pyroclastic flow deposits temperature: new data and statistical interpretation from the Minoan eruption of Santorini, Greece. *Geophysical J. International*, 203(1), 33–47. https://doi.org/10.1093/gji/ggv267.
- VEAL, R, O'DONNEL, L., MCPARLAND, L., 2016. Reflectance- Current state of research and future directions for archaeological charcoal: results from a pilot study on Irish Bronze Age cremation charcoals. *Journal of Archaeological Science*, 75, 72–81.