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ORIGINAL ARTICLE

Experiments on the effects of carbonization on some cultivated plant seeds

Tanja Märkle · Manfred Rösch

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Abstract Seeds and fruits of plants have different chances of getting carbonized in archaeological contexts. This chance depends on the one hand on the use of each plant; some plants are more likely to get in contact with fire than others, for example when they have to be roasted or cooked before eating. On the other hand this also depends upon the consistency and texture of the seeds themselves, which carbonize under different circumstances. The aim of our experiments was to reveal systematically the behavior of *Setaria italica*, *Panicum miliaceum*, *Papaver somniferum*, *Linum usitatissimum* and *Cannabis sativa* during carbonization. For this purpose we heated seeds of each species under both reducing and oxidizing conditions for 1–4 h in a muffle furnace to temperatures of 180–750°C. The results were striking: while reducing conditions usually enlarge the temperature range at which seeds carbonize without getting destroyed, broomcorn millet behaves exactly the opposite way. *Papaver somniferum* has only very little chance of becoming carbonized at all, because the temperature range at which this happens is very small. The chances of carbonization for *Linum usitatissimum* are quite good, those of *Cannabis sativa* even better.

Keywords Cultivated plants · Carbonization · Experimental archaeology

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T. Märkle (✉) · M. Rösch
Labor für Archäobotanik, Regierungspräsidium Stuttgart,
Landesamt für Denkmalpflege, Referat 114,
Arbeitsstelle Hemmenhofen, Fischersteig 9,
78343 Gaienhofen, Germany
e-mail: tanja.maerkle@rps.bwl.de

M. Rösch
e-mail: manfred.roesch@rps.bwl.de

Introduction

When organic material is exposed to heat it transforms mainly into gaseous components. Organic substances may oxidize partially and turn into elemental carbon only if circumstances are not optimal, for example if oxygen availability is too low for combustion. In the archaeological and archaeobotanical literature this process is called charring or carbonization without clearly defined differences between the two expressions. In other subject areas of science and technology definitions at least of "carbonization" exist, but differ between various fields of research, while there are no definitions for terms like "char" and "charcoal" (Braadbaart 2004; Braadbaart et al. 2004b; Poole et al. 2002). In a common sense "charred" means an incomplete combustion of organic material (Poole et al. 2002). Braadbaart prefers to use more objective terms like "heating" or "exposure to heat" for the treatment he applied to his samples (for example, Braadbaart 2004; but see also 2008). As the main subject of our analyses are not the chemical properties and reactions which occur in the seeds, in this paper "charring" and "carbonizing" will be used interchangeably meaning the conversion of biomass into a black solid residue by heat treatment. However, for archaeobotanists, this event is the ideal case for preservation of seeds and fruits in archaeological sites—apart from special sites, like waterlogged sediments or desiccated finds—because elemental carbon is not changed by chemical or biological processes in sediments (Jacomet and Kreuz 1999). Of course, seeds and fruits of plants have different chances of getting carbonized in archaeological contexts. It depends on the one hand on the use of each plant; some plants are more likely to be exposed to heat than others, for example, when they have to be roasted or cooked before eating. However, on the other hand it is also determined by the consistency and texture of the seeds themselves which carbonize under different circumstances. Therefore, it is necessary to know the conditions which are necessary for the carbonization of the seeds of various species. Thus we carried out a series of carbonization experiments in the laboratory. They are not supposed to reflect natural circumstances at prehistoric sites but rather provide documented and defined conditions and results taking three variables into consideration: temperature, time of exposure to heat and oxygen content of the surrounding atmosphere.

This study began with basic carbonization experiments which were part of a lecture by Manfred Rösch at the University of Innsbruck on the history and biology of cereals. While cereal grains of einkorn, emmer, wheat, rye, etc., yielded carbonized material with excellent preservation of morphological structures, flax seeds were less well preserved and broomcorn millet turned into a shapeless mass without any characteristic attributes. Potentially, the varying behavior of different seeds when carbonized could be one reason for the differing representation of taxa in archaeological contexts: millets, oil and fiber plant seeds always seem to be underrepresented in carbonized assemblages (Willerding 1991; Rösch et al. 1992; Jacomet et al. 1989, pp. 60; Bouby 2002; Wright 2003).

Previous analyses

While the first experiments on the carbonization of seeds were undertaken more than a 100-years-ago (Neuweiler 1905), other existing analyses have concentrated on cereals as the most important cultivated plants and some weeds (Braadbaart 2004, 2008; Braadbaart et al. 2004c, 2005; Braadbaart and Van Bergen 2005; Boardman and Jones 1990; Guarino and Sciarrillo 2004; Gustafsson 2000; Hillman et al. 1993; Hopf 1955, 1975; Schlichtherle 1983, pp 42ff; Wilson 1984). In addition, some experiments have been carried out on pulses (Braadbaart 2004; Braadbaart et al. 2004a, b; Braadbaart and Van Bergen 2005; Helbæk 1970; Guarino and Sciarrillo 2004; Hillman et al. 1993; Kislev and Rosenzweig 1991; Poole et al. 2002), grape pips (Smith and Jones 1990; Guarino and Sciarrillo 2004) and apples (Helbæk 1952) as well as some staple foods of North America (Wright 2003). Their main aim was the investigation of changes in size and shape of the seeds and fruits. Some more recent analyses include the study of physical and chemical alterations during the carbonization process (Braadbaart 2004; Braadbaart et al. 2004b, c; Hillman et al. 1993; Poole et al. 2002). Seeds of *Linum usitatissimum* and *Camelina sativa* were carbonized during an experiment in Sweden where a house was burned under less controlled circumstances (Gustafsson 2000). Apart from experiments on sunflower seeds (Wright 2003; Braadbaart et al. 2007; Braadbaart and Wright 2007) and olive stones (Terral et al. 2004), oil seeds have not been carbonized under standardized conditions until our own first analyses some years ago (Märkle and Rösch 2003). The same applies to millets, where carbonization of only African and Indian species of the genus *Eleusine* have yet been studied (Hilu et al. 1979).

Materials and methods

The carbonization experiments were accomplished with five different taxa, *Panicum miliaceum* (broomcorn millet), *Setaria italica* (foxtail millet), *Linum usitatissimum* (linseed), *Papaver somniferum* (opium poppy) and *Cannabis sativa* (hemp). While the first two are cereals rich in carbohydrates, the other three belong to the category of oil plants which are grown predominantly due to their high content of fat (Franke 1981). *L. usitatissimum* and *P. somniferum* as well as dehusked *P. miliaceum* were bought in a wholefood shop while *P. miliaceum* (not dehusked), *S. italica* and *C. sativa* were purchased directly from farms and gardens in southern Germany. For some repeat tests with *S. italica* and *P. miliaceum* we used seeds grown in the Botanical garden of Hohenheim University in southern Germany. Both millets were charred husked and dehusked. The seeds/fruits were not treated in any way before carbonization, which means not soaked, cooked or dried. From both millet species and from flax 100 seeds each were counted for one sample while only 50 seeds of hemp were used, taking into account the larger size of these seeds. All samples were weighed as well. Poppy seeds were not counted, but weighed. The samples were then put in small ceramic crucibles, two for each experiment and taxon—one was covered with aluminum foil, while the second was left open (Fig. 1).

One batch of each taxon was thus exposed to oxidizing conditions in the open crucible, and one to reducing conditions in the covered one. The crucibles were then put into a muffle furnace for 1, 2, 3 and 4 h with temperatures ranging from 180–750°C. The furnace was not pre-heated but the crucibles were placed into it at room temperature and then heated. The same heating rate was applied to all samples, so that after 20–40 min the predefined oven temperature was reached.

Fig. 1 The crucibles used, one covered by aluminum foil, the second left open

Fig. 2 Destruction of seeds, only some remain identifiable (*arrows*)

Results

Level of charring

The actual aim of this study was to determine the conditions under which prehistoric seeds and fruits have been able to survive in archaeological contexts until today. Therefore, the seeds were categorized after burning into three categories: uncharred, charred (thus able to be preserved), and destroyed. Soon it became clear that this distinction was not clear-cut. While Boardman and Jones (1990) during similar experiments used the alteration of the color of the seeds from brown to black as criteria for charring, this seems not to be the ideal feature in our analyses because there are millets which still show a brownish surface after treatment in the furnace, while their inner part is completely charred and black. Also by charring husked millets, the husks often still keep their brown color while the seeds themselves are already black. In contrast, the flax and poppy seeds became black after only short or low temperature charring, but they are still very oily and soft. These soft seeds were not added to the category "charred", because we think they would not be preserved in the soil.

Also there are samples, where one part of the seed is charred while another uncharred or destroyed (see Gustafsson 2000).

We decided to assign samples to the charred category when at least 5% of the remains exhibited typical characteristics of the species like shape or surface pattern while the biomass of the seeds had converted totally to a black solid residue (Fig. 2) because this leads to the possibility of preservation if they had been in an archaeological context. If less than 5% of the seeds were identifiable the sample was categorized as destroyed. In this case the seeds were burnt to ash or the carbonized residues had lost their shape and outer surface layers completely.

Millets

Millet seeds burst during the process of charring, their inner part pours out and the seeds may double in size like popcorn. However, the shape of the seeds often remains because the starch emerges only at one point while the rest of the surface remains identifiable.

The diagrams (Figs. 3, 4) are all based on the same principle: the *x* axis shows the time (1–4 h), the *y* axis the temperature. The gray line marks the minimal conditions for charring, the black one for the destruction of the seeds. This means that the zone between the two lines shows the zone of charred, and therefore, preservable seeds. Below the gray line seeds are still uncharred, above the black line they are destroyed.

Under oxidizing conditions *S. italica* and *P. miliaceum* start charring at similar temperatures. Foxtail millet needs 220°C regardless of the time, while seeds of broomcorn millet require only slightly higher temperatures of 235°C for 1–3 h and 225°C when exposure time is 4 h. But *P. miliaceum* is destroyed at temperatures between 400°C for 1 h and 335°C for 4 h, while *S. italica* stands up to 500°C for 1 h or 400°C for 4 h, respectively. So the foxtail millet seeds have better chances of being preserved at sites where enough oxygen is available during the charring process.

Even more obvious are the differences under reducing conditions. While *P. miliaceum* only becomes carbonized at higher temperatures of 270–245°C in 1–4 h, but is destroyed at lower temperatures (325–315°C, 1 or 4 h, respectively), than under oxidizing conditions, *S. italica* has an even greater range for getting charred; the seeds do not require higher temperatures for getting carbonized (just when the charring process is limited to 1-h duration they are slightly higher) but can stand even higher temperatures, 550–450°C during 1–4 h of charring. The seeds of broomcorn millet virtually explode if the oxygen content of

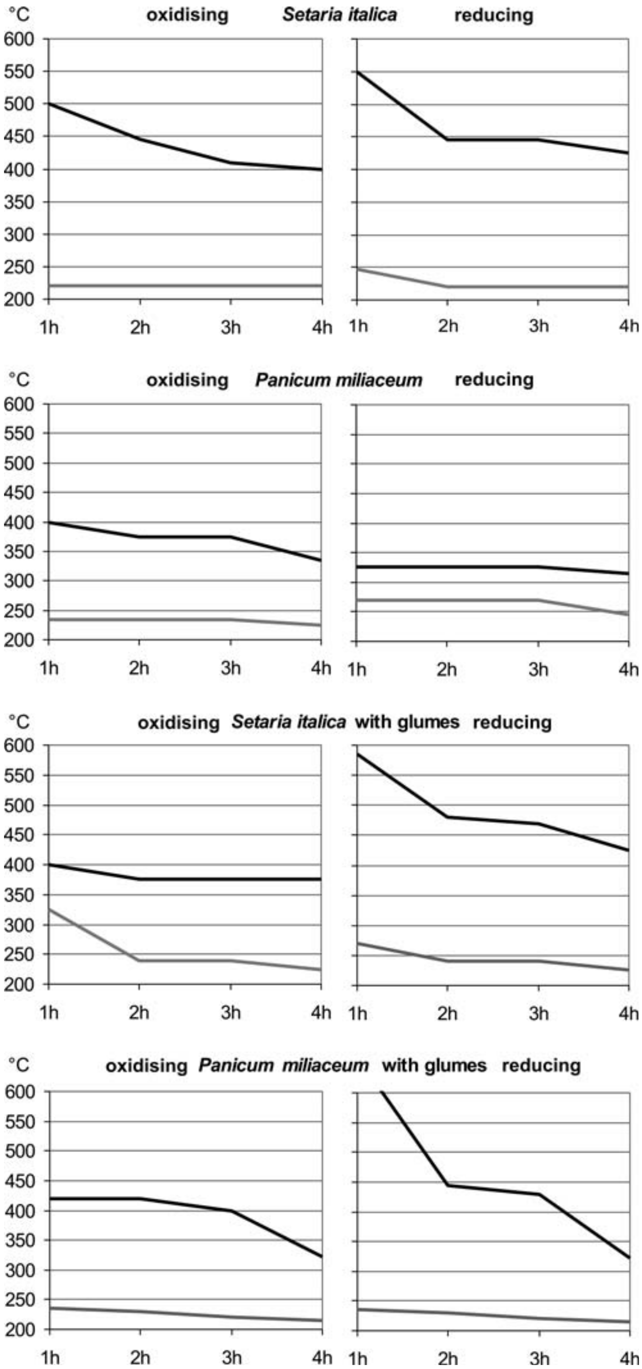

Fig. 3 Results of the charring experiments for the *Setaria italica*, *Panicum miliaceum* (millets); *x* axis, time (1–4 h), *y* axis, temperature; *gray line* minimal conditions for charring of seeds, *black line* minimal conditions for destruction of seeds

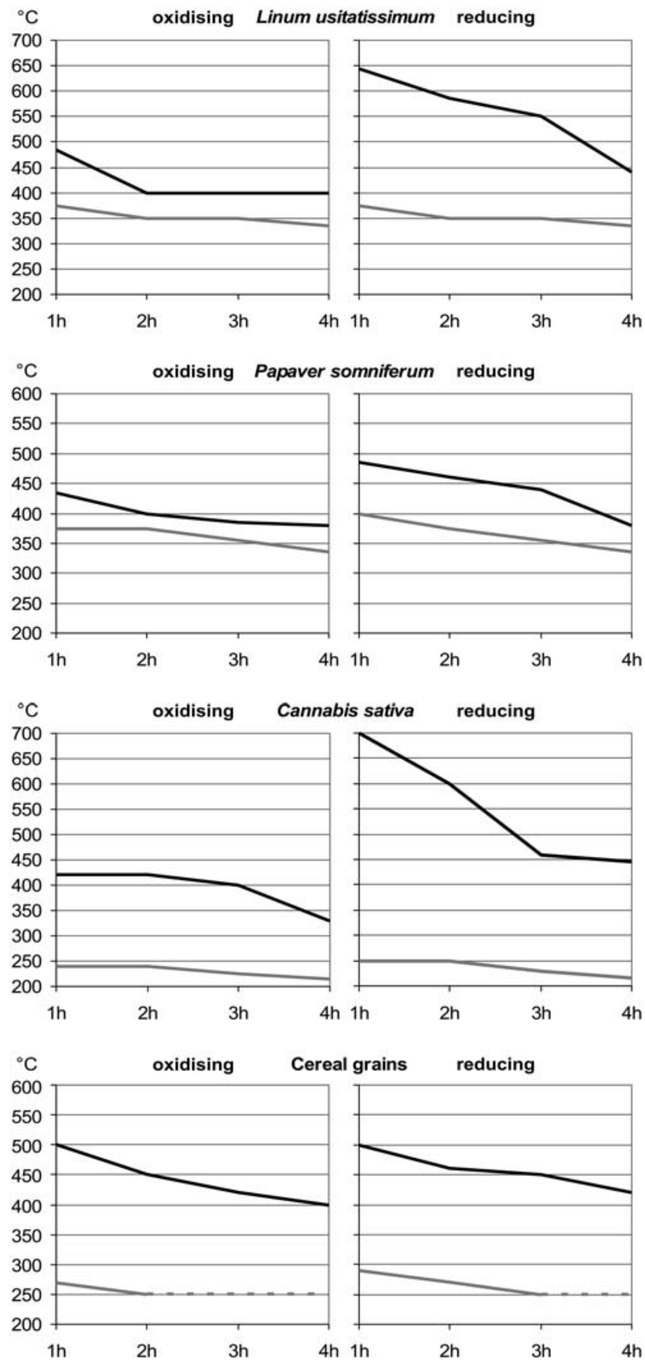

Fig. 4 Results of the charring experiments for oil seeds (*Linum usitatissimum*, *Papaver somniferum*, *Cannabis sativa*); in addition the results of the heating experiments of Boardman and Jones (1990) for cereals are shown; for further explanations, see Fig. 3

the surrounding atmosphere is low and they are not even recognizable as grains anymore, let alone as millets. Although the inner part of the seeds of foxtail millet pours out as well and forms a monotonous mass, the shape of some individual grains is still recognizable. The destruction does not seem to happen as explosively as in seeds of broomcorn millet. To check this peculiarity in the different behavior of *S. italica* and *P. miliaceum*, we conducted more heating experiments with dehusked seeds of both species from a different origin, which had been grown in the botanical garden at Hohenheim University. We heated both species for 1 and 3 h, respectively, under both oxidizing and reducing conditions at 300–420°C. The results showed the same difference in the performance under oxidizing versus reducing conditions of both species. *P. miliaceum* under reducing conditions carbonized in a much more explosive way than *S. italica*. The absolute temperatures leading to the destruction of the seeds vary somewhat: the grains of foxtail millet from the botanical garden at Hohenheim are not identifiable any more after heating at lower temperatures under oxidizing conditions, around 420°C after 1 h and around 375°C after 3 h, respectively. Anyway these temperatures are still higher than the destruction temperatures for the broomcorn millet grains. The foxtail millet samples carbonized under reducing conditions at the same times and temperatures still reveal some identifiable seeds and are therefore categorized as charred.

The results are different when the experiments are conducted with millets which are not yet dehusked, with the grains still embedded in their glumes (Fig. 3). The glumes seem to protect the grains of *S. italica*, so carbonization sets in at slightly higher temperatures. This effect is not seen with *P. miliaceum*: under oxidizing conditions charring temperatures are similar, under a reducing atmosphere even higher compared to dehusked grains. So the glumes do not impede carbonization but rather promote it. Another important difference is shown in the destruction temperatures. As expected, foxtail millet seeds with glumes are destroyed at higher temperatures under reducing conditions—and only under reducing conditions! If enough oxygen is in the atmosphere, the naked grains withstand even higher temperatures of 400°C and higher, while under reducing conditions they withstand only 400°C when charred for 1 h and less than 400°C when charred for a longer time. While the destruction temperatures for *P. miliaceum* under oxidizing conditions are somewhat increased—as expected, because of the protection of the glumes—under reducing conditions they are much higher! They are even higher than the destruction temperatures of the seeds (with glumes) charred under oxidizing conditions, at least when they are charred for 1–3 h; at 4 h the destruction takes place in both cases at around 320°C. So behavior of the millets during carbonization is considerably different if glumes surround the grains.

Oil plants

The seeds of *Linum usitatissimum* (Fig. 4) are classified as being charred when they show at least a thick black inelastic outer covering which cannot be bent when slight pressure is applied by tweezers. Before they reach this state they become deep black and look charred, but they are still very elastic and the inner part is brown. At the beginning of the charred state they get a glossy surface. Under oxidizing conditions this stage directly turns into the destroyed state at higher temperatures, while under reducing conditions there is an intermediary phase where the seeds become more deformed and the surface becomes bubbly and matt. Therefore, the chances of preservation are much better for this taxon under reducing conditions.

In both cases charring begins at the same temperatures (ca. 350°C) almost independently of the time applied. However, if there is enough oxygen in the atmosphere, the seeds are destroyed around 400°C or at somewhat lower temperatures when the charring lasts only 1 h. Under reducing conditions they preserve until approximately 600°C depending very much on the time. Hence the preservation of *L. usitatissimum* seeds is very much subject to conditions such as atmosphere and time of charring.

Papaver somniferum seeds also seem to carbonize after charring at low temperatures for a short time, but they are still soft and elastic. Properly charred seeds only occur at slightly higher temperatures than *L. usitatissimum*: carbonization starts after 2 h of charring at 375°C under reducing conditions, while under oxidizing conditions only 1 h at this temperature is needed (Fig. 4).

If charring is applied for only a short time and the oxygen content of the air is limited, the chances of preservation of poppy seeds are slightly better than in oxygen-containing surroundings: 485–440°C for 1–3 h, versus 435–385°C for 1–3 h. After 4 h of charring this advantage seems to diminish and both samples were destroyed at 380°C. During carbonization the seeds keep their shape and their surface with its characteristic pattern. However, they easily become very fragile and collapse at the lightest touch. In this case poppy seeds were classified as destroyed even if they were identifiable at first sight.

Cannabis sativa (Fig. 4) becomes carbonized at similar temperatures to the two analyzed millet taxa. One reason for these low temperatures seems to be the thick, hard seed coat, which gets black and hard quite rapidly, which is interpreted as charred. Destruction of the seeds starts in an atmosphere rich in oxygen after 1 h at 420°C or after 4 h at 330°C. If the oxygen content is limited, destruction temperatures are considerably higher, especially at short exposure times. In this case, hemp seeds are able to stand temperatures as high as 700°C. At even higher temperatures the shape of the seeds is still recognizable, but the texture consists of ash only and is so weak that it changes into dust by touching it (Fig. 5).

Cannabis sativa seems to have a good chance of getting carbonized. The range at which the seeds are preserved is quite high especially in reducing conditions but also to a lesser extent under oxidizing conditions. They have the best chances of preservation through time of all the taxa which were studied in the present experiments.

Fig. 5 *Cannabis sativa* heated for 1 h at 750°C

Discussion

Compared with oxidizing conditions, reduction usually enlarges the temperature range in which seeds carbonize without being destroyed. This has already been shown by previous analyses especially at lower temperatures (Boardman and Jones 1990, pp. 2; Wright 2003). The present analyses confirm this and show the same effect on *Setaria italica*, *Linum usitatissimum*, *Papaver somniferum* and *Cannabis sativa*. However *P. miliaceum* behaves exactly the opposite to what may be expected, at least the dehusked grains: seeds of commercially available broomcorn millets in covered crucibles were destroyed at temperatures around 320°C, while under oxidizing conditions they withstood temperatures as high as 400°C. This means that circumstances under which most other plant remains have good chances for getting charred are disadvantageous for *P. miliaceum*. This feature may contribute to the relative rarity of this species as charred grains in archaeological sites.

Papaver somniferum generally has little chance of getting carbonized. The range between carbonization and destruction of the seeds is very small and in a range of around 50°C–80°C depending on time and oxygen content of the air. Therefore, the possibility of this taxon surviving in an archaeological context is very limited.

Linum usitatissimum shows a great descrepancy depending on the oxygen content of the surroundings. It has a good chance of being carbonized under reducing conditions, while under oxidizing conditions the probability is low because of the wide difference of the destruction temperatures.

Cannabis sativa seems to have a better chance of getting carbonized than any other species in this experiment. So explanations for the underrepresentation of this taxon in dry deposits must have reasons other than the consistency and texture of the seeds themselves.

However, it is not the case that oil seeds lose their shape and surface pattern and thus are unidentifiable in the archaeobotanical material as is often cited in the literature (Schlichtherle 1985, pp. 26; Jacomet et al. 1989, pp. 60; Jacomet and Kreuz 1999, pp. 61).

To estimate the chances of millets and oily seeds becoming carbonized in comparison to other cereals, the results of Boardman and Jones (1990) are of interest (Fig. 4).

Cereal grains become carbonized at around the same temperatures as millets. Boardman and Jones did not apply temperatures lower than 250°C and are therefore represented by the dashed lines in the diagrams. However, destruction does not set in until 450°C and einkorn can even withstand 550°C; foxtail millet behaves similarly. In contrast, dehusked broomcorn millets are destroyed at temperatures clearly under 400°C.

Compared to the experiments of Boardman and Jones it can be asserted that cereal grains have better chances of charring than *P. miliaceum* or oily seeds—for *L. usitatissimum* and *C. sativa*, this is at least valid under oxidizing conditions. The chance of *S. italica* charring are similar to those of other cereal grains, with the exception of grains which are embedded in their glumes when carbonized under oxidizing conditions. In such cases the chance of charring is also worse than those of other cereal grains.

Conclusions

In the present experiments it has been shown that *Panicum miliaceum* and to a certain extent *Setaria italica* as well as oily seeds have significantly less chance of becoming carbonized in archaeological sites than other cereal grains. This may be due to the properties of the seeds themselves, probably because of their texture and consistency. However, other attributes of seeds used for experiments should be taken into account in future analyses as well, for example the moisture content of different breeds of cultivated plants. The chances of the carbonized seeds surviving embedding, excavation, washing and flotation are another aspect not considered in our experiments; the more fragile the seeds are in carbonized state, the fewer chances of survival and recovery they will have.

Especially interesting is the fact that reducing conditions are very disadvantageous for naked broomcorn millet grains, while the same conditions increase the chances of charring for all other taxa.

Finally, a muffle furnace in a laboratory cannot simulate fire events at prehistoric sites because charring takes place by means of hot air and not by flames. Furthermore, in the laboratory, constant temperatures and constant conditions exist while an open fire is subject to fluctuations. Considering that an open fire for the firing of pottery reaches 600°C (Lüdtke and Dammers 1990)—meaning most of the analyzed taxa would have been destroyed—it has to be assumed that these fluctuations existed at archaeological sites from where charred botanical macroremains have been found.

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