The Transformation of Phytolith Morphology as the Result of their Exposure to High Temperature

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ABSTRACT Phytoliths are an important component for interpreting the ancient botanical record. However, phytoliths can be altered through heating, either as the result of such activities as firing ceramics, clay molds use for casting metal or in hearths. Phytoliths can also be altered through heating as the result of creating comparative sample from living plants. By heating phytoliths at graduated intervals it was found that different types of phytoliths lost their diagnostic morphological characteristics at significantly different temperatures. The phytoliths used in this study are derived from economically important plants to Chinese archaeology and culture. Given the consistent results of the alteration of different type of phytoliths at specific temperatures it should eventually be possible to use phytolith alterations as a proxy measure of the original firing temperature of ancient objects and features. *Microsc. Res. Tech. 75:852–855, 2012.* © 2012 Wiley Periodicals, Inc.

INTRODUCTION

During the last decade, phytolith analysis has brought new insights into environmental and agricultural archaeology (Ball, 1992; Bowery, 2001; Horrocks et al., 2000; Pearsall, 1978, 1995; Piperno, 1988, 1993, 2006; Rosen, 1992; Rovner, 1992). A common method for the recovery of phytoliths from botanical specimens is a technique known as "dry-ashing" in which samples of plant material are heated to remove the organic matter from the walls of the plant cells. This study was conducted to examine the possible effects that heating might have on the morphology of the phytoliths from different species of plants that are of economic importance in China. By understanding the patterns of alteration of different types of phytoliths under different temperature regimes, these data can be used as proxy measures for the reconstruction of firing temperatures of archaeological artifacts and burned features.

THE EFFECTS OF HEATING ON PHYTOLITHS

Phytoliths are microfossils composed of amorphous opal structures deposited within the walls of plant cells. Just as the cells from different plants are different from one another, phytoliths take on a considerable variety of forms. Because of their species-specific variation, phytoliths provide significant taxonomic information based on their shape, size, and other anatomical features. Morphologically, distinct phytoliths are also derived from different parts of the same plant (e.g., Piperno, 1988, 2006; Rovner and Russ, 1992; Russ and Rovner, 1987; Twiss, 1969, 1987).

Dry-ash technique is one of the most common methods used to separate phytoliths from surrounding organic matter without using toxic chemicals under fume hoods (e.g., Piperno, 1988; Rovner, 1971). A number of researchers have examined the applicability of dry-ash method for the recovery of phytoliths from plants. (e.g., Bowdery, 1989; Piperno, 1988; Rovner, 1971). It is very important that the dry-ash method does not affect morphological or size characteristics of phytoliths. However, there is evidence that indicates temperatures in excess of 500°C can significantly alter the physical characteristics of phytoliths (Piperno, 2006). The dry-ash method can cause crystallization of some opal, an increase in the refractive index, and a decrease in surface area (Piperno, 2006). Runge (1998) observed dramatic changes of lightly silicified jigsaw-shaped epidermal and hair-cell phytoliths from African eudicots phytoliths that were broken into indistinguishable bodies when heater above 600°C. Otherwise, there are few published research works that systematically compare the morphological stability of different kinds of phytoliths under the condition of high temperatures.

In this work, the alteration of morphological characteristics of five important phytoliths types heated to different temperatures is documented. Two of the selected phytolith types are derived from rice husks and rice leaves. China is a country of the largest producer and consumer of rice in the world, with a very long history for rice cultivation (Crawford, 2006; FAO, 2004; Fujiwara, 1993; Khush, 1997). Phytoliths derived from rice (*Oryza sativa* L.) recovered from archaeological sites have provided strong evidence for the cultiva-

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TABLE 1. Change degree of phytolith morphology in different temperatures

Type	500°C	600°C	700°C	800°C	900°C	1000°C	1100°C
Rice husk phytolith	N	N	N	N	N	N	S
Rice leaf phytolith	N	N	N	S	D	—	—
M. alba leaf phytolith	N	S	L	D	—	—	—
P. tatarinowii leaf phytolith	N	S	L	D	—	—	—
C. bungeana leaf phytolith	N	L	D	D	—	—	—
C. koraiersis leaf phytolith	N	L	D	D	—	—	—

N, no change; S, slightly changed; L, largely changed; D, destroyed.

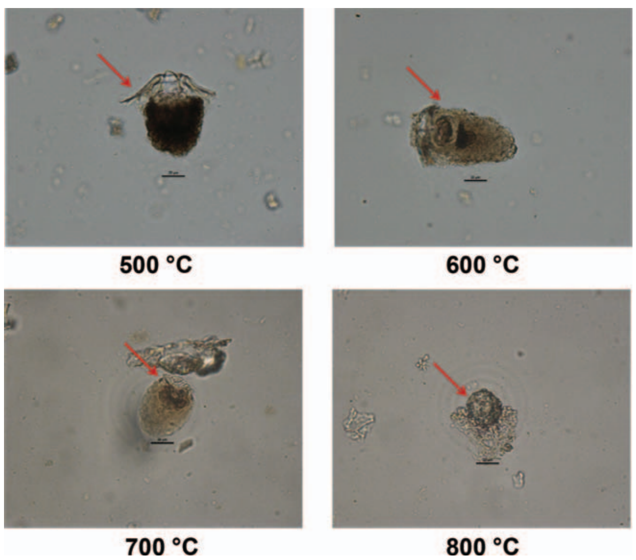

Fig. 1. *C. bungeana* leaf phytolith morphology in 500, 600, 700, and 800°C. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

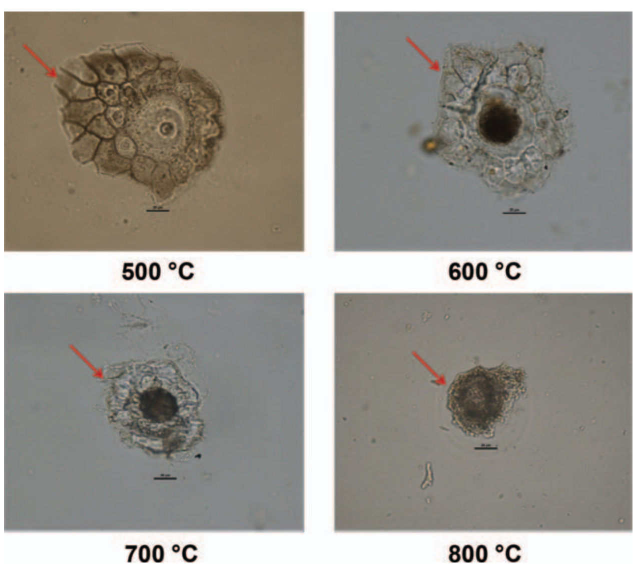

Fig. 2. *M. alba* leaf phytolith morphology in 500, 600, 700, and 800°C. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

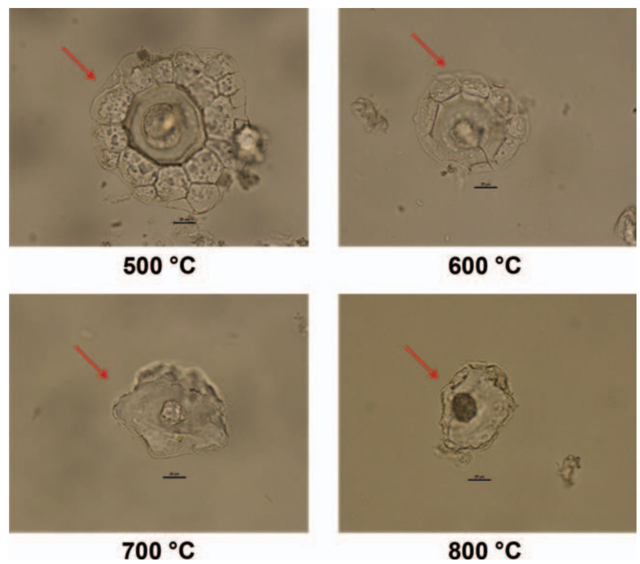

Fig. 3. *P. tatarinowii* leaf phytolith morphology in 500, 600, 700, and 800°C. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

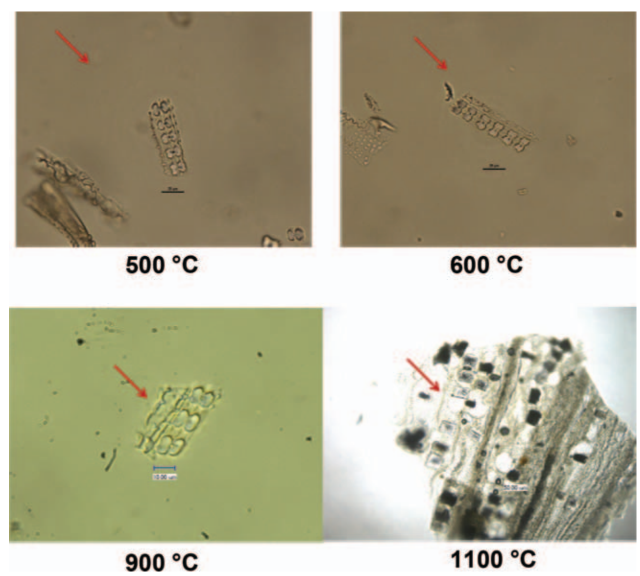

Fig. 4. *Rice Leaf* phytolith morphology in 500, 800, 900, and 1100°C. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

tion of rice in ancient times (Zhao, 1998). The shape of phytoliths is considered an effective criterion for determining subspecies of ancient rice (Zhao, 1998; Zhang, 2002). The third is *Pteroceltis tatarinowii* M., which is an important material used for the production of Xuan work, a special kind of work used for Chinese painting

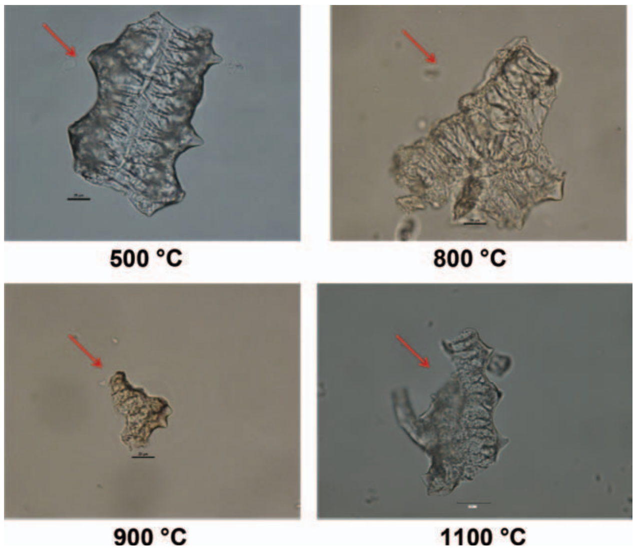

Fig. 5. *Rice Husk* phytolith morphology in 500, 800, 900, and 1100°C. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

and traditional calligraphy (Mullock, 1995). The other two plants used in this study are *Celtis bungeana L.* (Ulmaceae) and *Morus alba L.* (Moraceae), which are typical vegetation types found in and around Zhoukoudian (Kong et al., 1985).

MATERIALS AND METHODS

The plant materials were obtained from different places within China, including the provinces of Anhui, Henan, Jiangxi, Zhejiang, and the city of Beijing. Phytoliths were extracted from the incineration of plant materials by the dry-ash technique in oxidizing conditions. Samples were placed in porcelain crucibles in a muffle furnace and heated for 8 h over a range of temperatures ranging from 500 to 1100°C. Rice phytoliths were heated over a temperature range between 500 and 1100°C at 100°C intervals. The phytolith bodies of *P. tatarinowii, C. bungeana and M. alba* leaf were indistinguishable when fired above 800°C. Samples from these plants were only heated to a temperature range between 500 and 800°C at 100°C intervals. The ashing experiments were repeated three times.

Then the samples heated were analyzed on their-morphology and physiology. The resulting slides were scanned for diagnostic phytoliths using a Nikon ECLIPSE LV100 POL at 500×. Phytoliths were counted according to the number of identifiable phytoliths.

RESULTS

Based on the morphological analysis, there are following findings: the alteration of the morphology of phytoliths from the different plant families took place at different temperature ranges (Table 1). As shown in Figure 1, the morphology of *C. bungeana* leaves was preserved well at 500°C. When the temperature reached 600°C, its shape was broken, and it lost its original characteristic form (Figure 1). The morphology of phytoliths from leaves of *P. tatarinowii* and *M. alba* is well preserved at 600°C (Figures 2 and 3). Both species lose their characteristic form at 700°C as shown in Figures 2 and 3. In comparison, although some crystallization may occur, the morphological characteristics of rice husk and rice leaf phytoliths resist significant change if temperature do not exceed about 900°C (Figs. 4 and 5). As shown in Figures 4 and 5, the physical characteristics of rice leaf phytoliths are significantly altered when the temperatures exceed 900°C. Alternation of phytoliths from rice husks phytoliths does not occur until they are heated to a temperature greater than 1000°C.

CONCLUSION

This study has demonstrated that the alteration of the morphology of Phytolith of *Oryza sativa L.* (Poaceae), *Pteroceltis tatarinowii* Maxim. (Ulmaceae), *Celtis bungeana L.* (Ulmaceae), and *Morus alba L.* (Moraceae) occurs at different temperatures. In particular, the alteration of phytoliths derived from rice occurred at a higher temperature relative to the other phytoliths examined during this study. Research is currently underway to understand the mechanisms that control the variation in the alteration in the morphology of phytoliths when subjected to elevated temperatures.

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