Experimental Micromorphology on Burnt Shells of *Anomalocardia brasiliana* (Gmelin 1791) (Bivalvia, Veneridae) and Its Potential for Identification of Combustion Features on Shell-Matrix Sites

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Received
31 July 2013
Revised
20 December 2013
Accepted
13 March 2014

Scientific editing by Francesco Berna

Published online in Wiley Online Library
(wileyonlinelibrary.com).

doi 10.1002/gea.21486

The identification of *in situ* or reworked hearths in archaeological sediments is currently one of the main topics in archaeological micromorphology. In the case of shell-matrix sites (shell mounds and middens), the clast-supported matrix and high porosity can hamper the clear identification of combustion features. Previous experimental approaches were useful to identify in thin section the transformations induced by heating in mollusk shells, which are related to its mineralogy. Since different species of mollusks contain diverse mineralogy (aragonite, calcite, or both), it is expected that each species follows a specific thermal behavior, although similar general trends exist. In this short contribution, a set of valves of the mollusk *Anomalocardia brasiliana* (Gmelin 1791) (Bivalvia, Veneridae), the most important component of Brazilian shell mounds, was heated in a muffle furnace at temperatures from 200°C to 800°C. Thin sections were made and their description is presented for the identification of burnt shell of *A. brasiliana*. This information can help to infer the temperature of prehistoric fires and can assist in the identification of *in situ* versus reworked hearths in shell-matrix sites.

INTRODUCTION

Experimental approaches in archaeological micromorphology provide references for the interpretation of archaeological samples. Recent work in experimental and ethnoarchaeological micromorphology (see Macphail et al., 2003; Mallol et al., 2007; Miller et al., 2009; Villagran et al., 2011a; Miller & Sievers, 2012; Mallol et al., 2013) proves the benefit of this approach for understanding archaeological microfacies (mF).

In the case of shell-matrix sites (shell mounds and shell middens), experimental approaches were used to infer the temperature of prehistoric hearths from the optical and textural characteristics of burnt shell in thin section (Villagran et al., 2011). The experimental heating of the species *Mytilus edulis* (Linnaeus) helped to differentiate between hearths with good and poor combustion. This, combined with faunal and isotopic analyses, could be correlated with the season of occupation of the site. Identifying the temperature of heating in archaeological shells also has implications for taphonomic studies (e.g., fragmentation indexes, trampling) and can give insights into prehistoric cooking practices (Stiner et al., 1995; Stiner et al., 2003).

The bivalve mineralogy, as well as the temperature and duration of heating, will influence the transformations induced in the shell (Gaffey et al., 1991). Some bivalves, such as *M. edulis*, have an outer calcite layer and inner aragonite layer (Burgoin, 1988), while others are exclusively made of aragonite or calcite or even show an intercalation of aragonite with calcite layers within the shell. The diverse mineralogy of carbonate-secreting organisms accompanies differences in the mineral structure and in the arrangement of the organic matrix (Lowenstam, 1981; Weiner et al., 1983; de Paula & Silveira, 2009). The destruction of the organic matter is one of the key transformations induced by heating, together with the loss of water and release of CO_2 (Gaffey et al., 1991). Therefore, variations in mineralogy can determine specific thermal behaviors for different species of mollusks (see Faust, 1950; Schifano, 1982; Balmain et al., 1999; Bourrat et al., 2007; Zolotoyabko & Pokroy, 2007; Nemliher et al., 2009; Ren et al., 2009). Since mollusk shells vary in shape, size, and mineralogy, it is important to continue developing the experimental collections by including a wider diversity of species.

Along the coast of Brazil, shell mounds of diverse dimensions are the most conspicuous archaeological remnant of precolonial populations (Gaspar et al., 2008). The most frequent mollusk species in these sites is *Anomalocardia brasiliana* (Gmelin, 1791). The shell of this species is entirely made of aragonite (Bezerra et al., 2009) and is covered by an organic external sheet (the periostracum). In this short note, shells of *A. brasiliana* were burnt at temperatures ranging from 200°C to 800°C to identify the heat-induced changes in the shell optical properties. Results can be used in micromorphological studies of archaeological sediments to infer the temperature reached by prehistoric fires, to recognize reworked versus *in situ* combustion features, and to understand the taphonomy and potential processing of archaeological shells.

MATERIALS AND METHODS

Shells of *A. brasiliana* were heated in a muffle furnace at 200°C, 300°C, 400°C, 500°C, 600°C, 700°C, and 800°C. The maximum temperature was set at 800°C because above that temperature the shell is highly friable, lacks any organic bounding, and is made of powdery calcium oxide (CaO), which makes thin sectioning impossible. A total of 21 mollusks was used for this experiment, in order to have three burnt individuals per temperature interval. Samples were put into crucibles and placed into the muffle furnace until the temperature reached 200°C. After 30 minutes, the samples burnt at 200°C were removed from the muffle. Then, the temperature in the muffle was set to 300°C and after 30 minutes, the second pair of samples was removed. This process continued through five more steps until reaching 800°C. At every step and after removal from the muffle, samples were put into a desiccator to cool. Thin sections were made out of two of the three heated mollusks. The third mollusk was used for macroscopic evaluation of the changes induced by heating.

One set of samples was impregnated under vacuum with a mixture of epoxi resin, hardener, and diluent. The second set of samples was impregnated using polyester resin. Thin sections were analyzed with a Zeiss Stemi 2000-C stereomicroscope, and Zeiss Axioplan and Zeiss Axio Imager A2 petrographic microscopes, under plane-polarized light (PPL) and cross-polarized light (XPL).

RESULTS

Table I summarizes the macroscopic and microscopic observations of the heated shells of *A. brasiliana*. The changes induced by heating are easy to distinguish both macro- and microscopically. In the hand specimen, the thermal alteration of the shell starts at 200°C, with a general darkening of the outer surface. This darkening goes from dark brown at 200°C (Figure 1A1), black at 300–400°C (Figure 1B1 and C1), until gray and white at temperatures above 500°C (Figure 2A1 and B1, Figure 3A1 and B1). These color changes are related with the combustion of the periostracum, carbonization of the shell surface, and the sequential transformation of aragonite into calcite and calcite into calcium oxide.

Under the microscope, the darkening of the shell valve starts above 500°C (Figure 2A2–5). Below this temperature, it is difficult to distinguish clear optical differences related with heating (Figure 1A2–5, B2–5 and C2–5). At 600°C and 700°C, the shell is dark brown (Figures 2B2–5, 3A2–5), turning gray at 800°C (Figure 3B2–5). Parallel to the darkening of the valve, there is the carbonization and volatilization of the periostracum. This external organic sheet is carbonized at 400–500°C (Figures 1C3–5, 2A5) and completely disappears above 500°C. From 500°C, the shell starts to fissure following the growth lines of calcium carbonate secretion (Figure 2A3–4). Fine longitudinal fissures and major cracks appear at above 600°C (Figures 2B2–5, 3A2–5). The formation of pseudo-vacuoles, characteristic of the physical deformation of the shell, starts at 700°C (Figure 3A3–5).

DISCUSSION

After disappearance of the periostracum, the most evident effects of heating of *A. brasiliana* in thin section are darkening of the valve and formation of longitudinal and transversal fissures and cracks. The fissures result from the loss of water during burning of the organic matrix (Gaffey et al., 1991), which commonly starts at 240–500°C in bioaragonite shells. However, as also seen in the experiment, the most important changes happen above 500°C, when most of the organic matrix is lost and CO_2 is released from the mineral phase (Balmain et al., 1999).

Another consequence of heating bioaragonite involves its transformation into calcite. The change in mineral phase happens at 300–400°C (Faust, 1950; Passe-Courtin et al., 1995; Balmain et al., 1999; Nemliher et al., 2009), or even at lower temperature due to the large number of lattice defects and water inclusions in shells (Gaffey et al., 1991). However, the transformation of aragonite into calcite would not result in major changes in weight or severe destruction of the organic matrix, since the organic

Table I Alterations of *Anomalocardia brasiliana* valves burnt at temperatures from 200°C to 800°C.

Temperature	Description	Figure
200°C	Darkening of the external surface of the valve, reddish dark brown color	1A1
	No major alteration of the internal surface of the valve, despite the black carbonized rim	1A2,3
	Well-preserved valve with visible growth lines and preserved orange (PPL) periostracum	1A4,5
	No major textural alterations	
300°C	Black external surface	1B1
	Slight darkening of the shell with dark spots	1B2—5
	Well-preserved dark-orange periostracum (PPL)	1B4,5
400°C	Black external surface, very friable	1C1
	Darkening of the shell and more abundant dark spots. Visible growth lines	1C2,3
	Complete carbonization of the periostracum, detaching from the valve in some areas (PPL)	1C3–5
500°C	Grayish external and internal surfaces	2A1
	Dark-brown shell (PPL) with low birefringence (XPL) and less-visible growth lines	2A2–4
	Fine longitudinal fissures following the growth lines of carbonate secretion (PPL)	2A2–4
	Black carbonized periostracum (PPL)	2A5
600°C	Grayish external and internal surfaces	2B1
	Blackish-brown shell (PPL) and dark reddish brown (XPL) with complete loss of the periostracum	2B2–5
	Increase in the number of longitudinal fissures	2B4,5
	Appearance of transversal fissures developed from the surface to the inner portions of the valve	
700°C	Dark-gray and white external and internal surfaces	3A1
	Intense darkening of the valve, dark grayish brown (PPL) with opaque rim and low birefringence (XPL)	3A2–5
	Increased longitudinal fissures	3A3–5
	Very diffuse growth lines (PPL, XPL)	
800°C	General deterioration of the valve and high friability	3B1
	White external and internal surfaces	
	Gray valve with no visible growth lines and black opaque rim (PPL)	3B2
	Formation of pseudo-vacuoles (PPL, XPL)	3B3–5
	Rough surface and visible CaO crystals (PPL, XPL)	3B4,5

matrix in bioaragonite is not fully destroyed below 500°C (Bourrat et al., 2007; Zolotoyabko & Pokroy, 2007; Nemliher et al., 2009) or even 800°C (Dauphin et al., 2006). In fact, neither the hand specimens nor the thin sections of *A. brasiliana* heated at 300–400°C showed visible changes that could be related with the transformation of aragonite into calcite.

The transformation of calcite into calcium oxide, which happens between 500°C and 600°C, was clearly visible both macro- and microscopically in *A. brasiliana*. This change happens after a major release of CO_2 that produces a weight loss of about 50% in the shells. Above 900°C, calcite is completely replaced by CaO (Balmain et al., 1999).

As observed for the heating experiments of *M. edulis*, fissuring and deformation of the valve are the most evident features of temperature-induced weight loss of *A. brasiliana*. The physical alterations, together with combustion of the organic and mineral matrix that provokes a general darkening of the shell, happen above

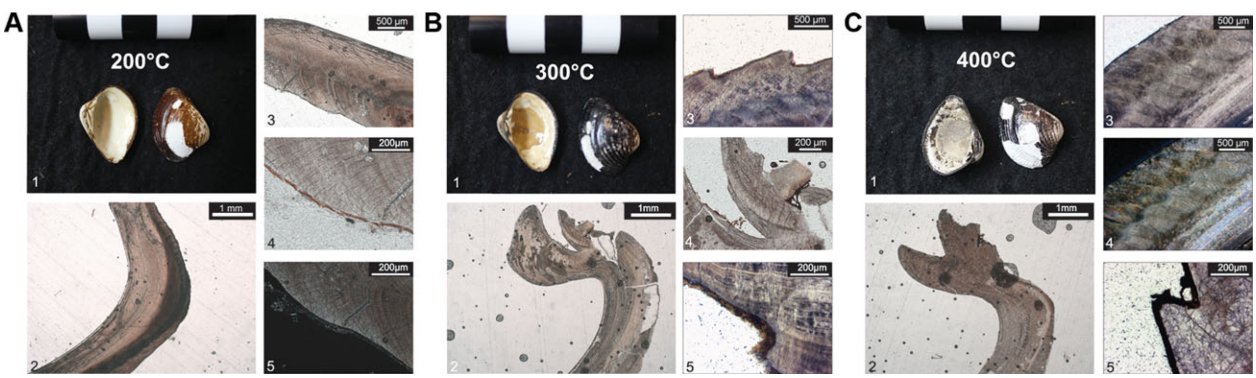

Figure 1 Transformation processes of burnt valves of *Anomalocardia brasiliana* at 200°C (A), 300°C (B), and 400°C (C), described in Table I.

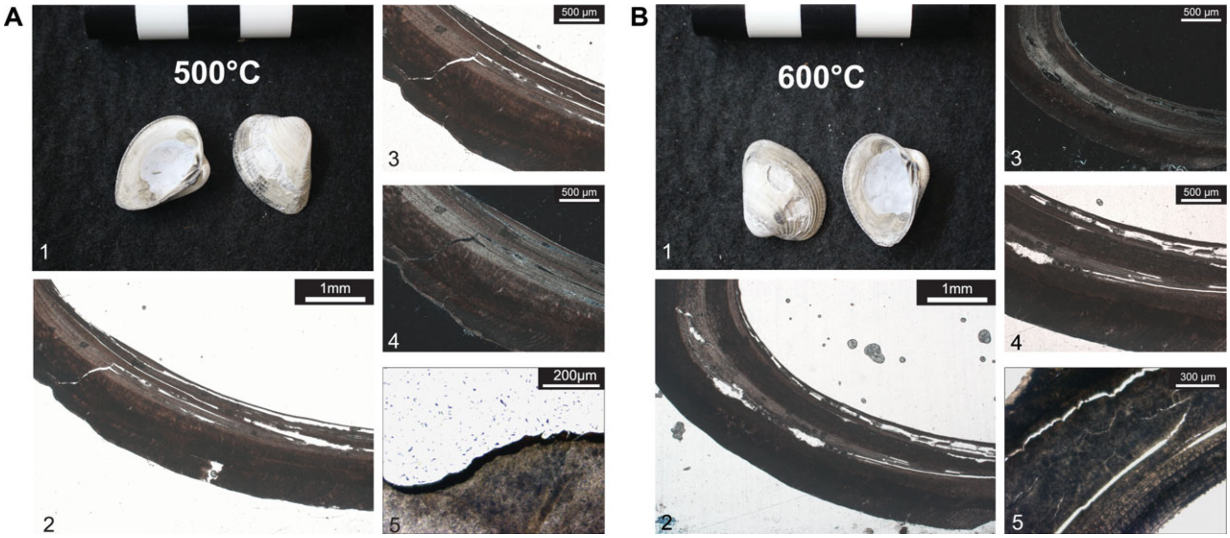

Figure 2 Transformation processes of burnt valves of *Anomalocardia brasiliana* from 500°C (A) to 600°C (B), described in Table I.

500°C, setting a clear temperature boundary where shell burnt below and above this threshold can be easily identified in thin sections. The higher the temperature, the more transformations the shell goes through, enabling more straightforward estimations of the burning temperature.

Figure 4 contains two examples of thin sections collected from a shell mound located in the southern coast of Brazil. The Cabeçuda site (22J 0712601/6852170 UTM) is one of the oldest shell mounds in the State of Santa Catarina, located in an area known for its monumental sized shell mounds (Gaspar et al., 2008). Radiocarbon dating on charcoal and shell samples collected from the base and top of the mound, respectively, indicate an occupation time span of more than 2000 years (5280–3930 to 1510–670 cal. yr B.P.) (Giannini et al., 2010). The site occupies an area of 900 m^2 and has been almost completely destroyed (Mendonça de Souza, 1995). Its stratigraphic profiles show an intercalation of centimetric to decimetric layers made of variable proportion of mollusk shells (mostly *A. brasiliana*), fish bone fragments, charcoal, and terrigenous sediments. Micromorphology samples were collected from two different locations within the site where possible combustion features were identified during the field examination of two vertical successions (Figure 4A and B).

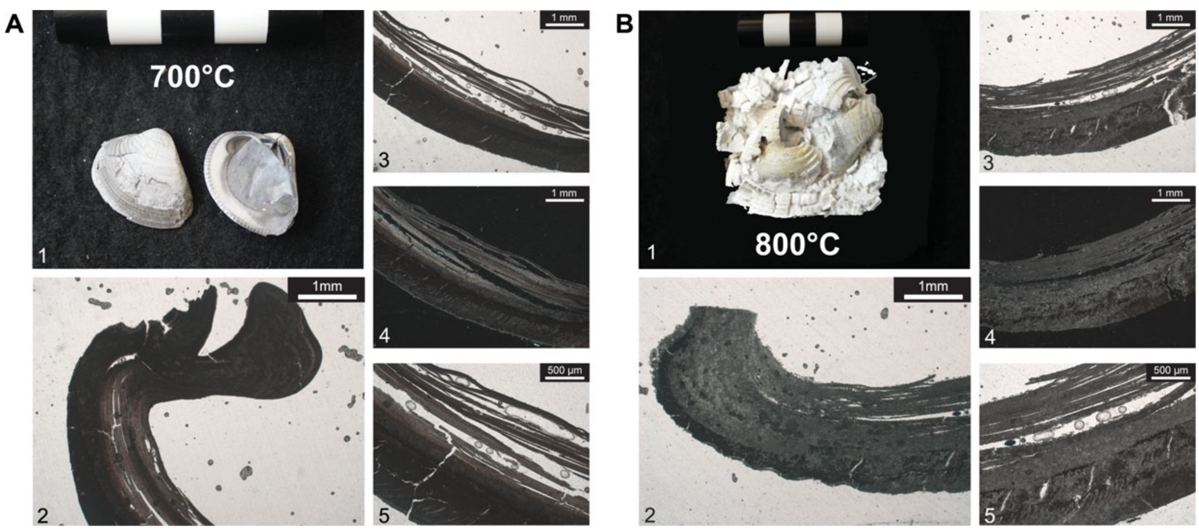

Figure 3 Transformation processes of burnt valves of *Anomalocardia brasiliana* from 700°C (A) to 800°C (B), described in Table I.

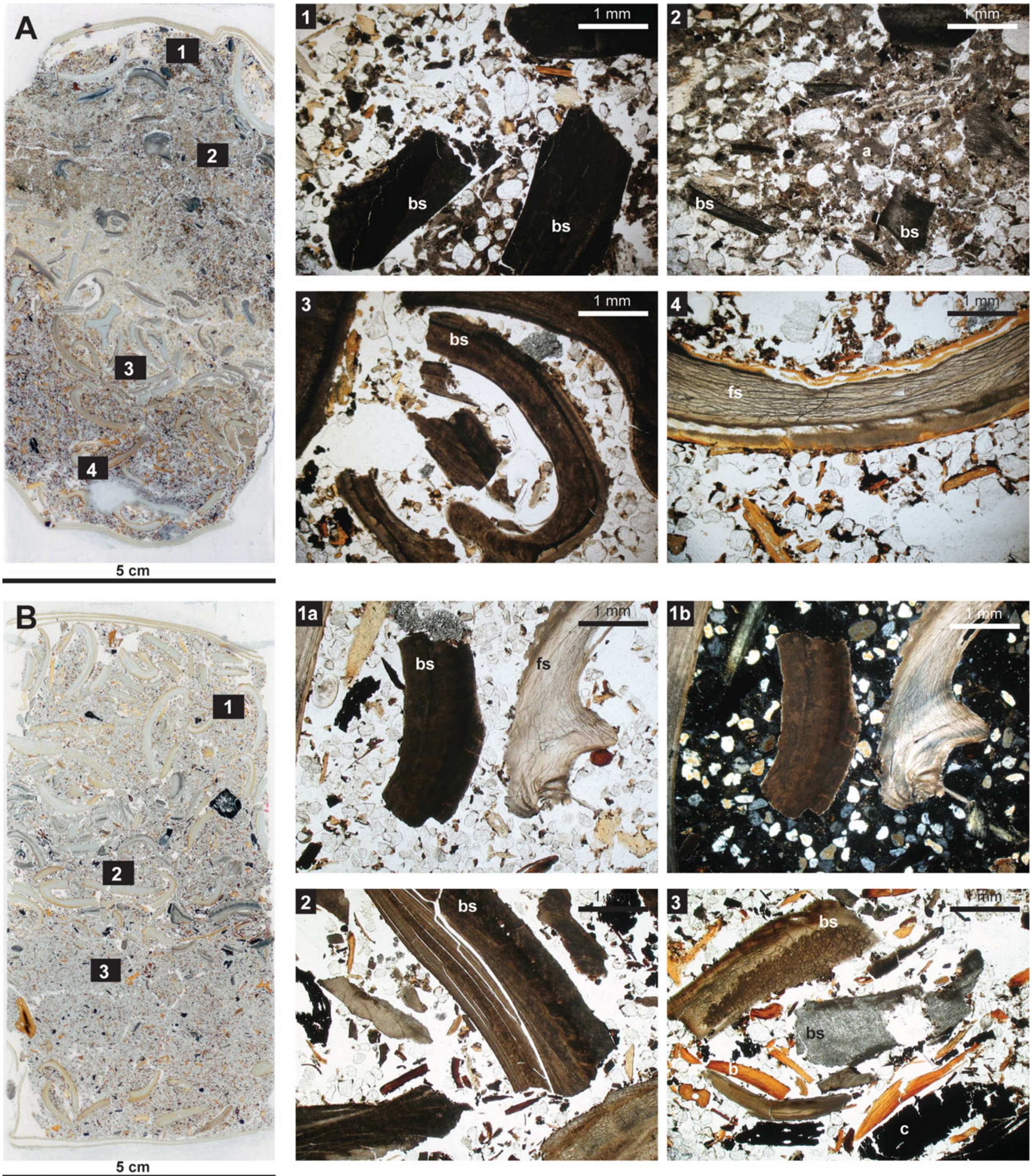

Figure 4 Thin sections collected from two stratigraphic successions at the Cabeçuda shell mound (Santa Catarina, Brazil). Scanned thin section of *in situ* combustion feature, identified through micromorphological analyses and comparison with the heating experiment of *Anomalocardia brasiliana* (A). Photomicrographs of sample A (PPL): shells burnt at temperatures between 600°C and 700°C (bs) (1); micromass made of ashes (a) and small fragments of shell burnt at above 700°C (2); shells burnt at temperatures around 500°C (3); fragment of shell with no heat-induced changes (fs) and phosphate coating (4). Scanned thin section of reworked combustion feature (B). Photomicrographs of sample B: shell fragment burnt at 500–700°C next to nonheated shell fragment, in PPL (1a) and XPL (1b); shell fragment burnt at 200–400°C (2); shell fragments burnt at 200–300°C next to fragment burnt at above 800°C, bone fragments (b), and charcoal (c) (3).

The microscopic evaluation of the thin section, compared to the heating experiments presented here, help to distinguish between an *in situ* hearth and reworked hearth components. Sample A (Figure 4A) showed three distinct mF: upper mF made of burnt shell fragments (Figure 4A1), burnt bones, charcoal, and quartzose sand grains, with micromass made of wood ashes (Figure 4A2); middle mF with burnt shells (Figure 4A3), bone fragments, and quartzose sand with organic microaggregates; lower mF with shell fragments, bones, and quartzose sand with organic microaggregates (Figure 4A4). By comparing the mollusk assemblage in each of the three mF with the experimental heating of *A. brasiliana*, it is inferred that the upper and middle mF are part of a combustion feature, given the widespread presence of shells with signs of burning. Shells with clear signs of burning make up almost 90% of the shell assemblage in the upper and middle mF. This and the close association of burnt shells with burnt bones and wood ash in the upper mF suggest the presence of an *in situ* combustion feature. Similar observations have been made for other hearths in shell middens from Tierra del Fuego (Villagran et al., 2011b), where thin sections made from combustion features (identified during excavation of the site) always contain burnt shell and ashes. In the Cabeçuda site, the optical evaluation of shells under the microscope indicates heating temperatures of 500–700°C. The absence of burnt shell in the lower mF, where shells show no signs of heat-induced changes (Figure 4A4), indicates that the surface fire did not affect the substrate. The middle mF would represent the transition between the fire and the intact shell mound sediments. In the highly porous sediments of the shell mound, with low quantities of clay minerals in the micromass, reddening of the substrate would not necessarily be present as an indicator of *in situ* fire, as described for other contexts (see Canti & Linford, 2000; Schiegl et al., 2003; Berna et al., 2007; Mallol et al., 2007; and review by Mentzer, 2012).

Sample B showed similar attributes as sample A in the field examination of the profile. However, micromorphological analyses indicate it contains the components of reworked combustion features. The mollusk assemblage in sample B shows a mixture of shells with no signs of heat-induced changes (Figure 4B1a and 1b) with shell burnt at temperatures between 200°C and 400°C (Figure 4B2), 500°C and 700°C (Figure 4B1a and 1b), and above 800°C (Figure 4B3). The random mixture of different temperatures and nonburnt shells, the high porosity of the sediments, high fragmentation of the bones, and absence of ashes in the micromass indicate association with combustion-related activities, but not an intact combustion feature as seen in sample A. Similar observations have been made on the bones in thin section to differentiate between *in situ* fireplaces and dumping areas (Schiegl et al., 2003; Miller et al., 2009). Although in these cases the authors also cite the absence of a rubified substrate, reddening of the substrate does not always happen in a shell site context if clay minerals are not present in the micromass. In this case, burning of the coarse fraction in the substrate of the fire, such as shells and bones, would indicate a primary context, like observed in the middle and upper mF of sample A.

CONCLUSIONS

Shells of *A. brasiliana* burnt at different temperatures can be identified in thin section. The physical transformations that shells go through are due to the weight loss and destruction of the organic matrix as a result of heating. Above 500°C, the textural and mineralogical effects of heating are clearly visible, since shell darkens and fissures along the growth lines, deforms with pseudo-vacuole formation and turns into powdery calcium oxide. In archaeological sediments, the precise identification of burnt shell in micromorphological analyses can give clues on the temperature reached by prehistoric fires. Furthermore, the identification of burnt shells can potentially help to distinguish between *in situ* or reworked hearths, especially in shell midden contexts. It also provides insights into taphonomy of the mollusk assemblage and can help to understand fragmentation indexes.

The author would like to thank the financial support of Fundação de Amparo à Pesquisa do Estado de São Paulo (FAPESP proc. 08/51264-0) and the Ciência sem Fronteiras program (CNPq/ CAPES proc. 245774/2012-2); Professor Madu Gaspar (Museu Nacional, UFRJ), Professor Claudia Rodrigues Carvalho (Museu Nacional, UFRJ), and Professor Paulo DeBlasis (Universidade de São Paulo) coordinators of the field work at the Cabeçuda site; Professor Paulo C. F. Giannini and the Sedimentology laboratory of the Institute of Geosciences (Universidade de São Paulo); Professor Christopher Miller, Panagiotis Kritikakis, and the Micromorphology Laboratory of the Institute for Archaeological Sciences (University of Tübingen).

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