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SIMPLIFIED PROCEDURE FOR HAND FRACTURING, IDENTIFYING, AND CURATING SMALL MACROCHARCOAL REMAINS

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SUMMARY

Charred plant remains are common and significant components of many archeological assemblages, and the proper identification of these remains is essential for an excavation team to gather the maximum amount of information. Identification of charred plant remains, especially of small pieces, can be difficult due to the brittle characteristics of charcoal and changes in anatomical structure due to charring. Charcoal must be snapped, which is difficult for small specimens, or sectioned with time consuming resin embedding procedures. This study presents an alternative procedure in which small (0.7 mm thick) charcoal specimens are produced, attached to specimen mounting stubs used in scanning electron microscopy (SEM), and then hand snapped. This procedure consistently produced flat viewing surfaces. It also reduced the air evacuation time in SEM and facilitated the production of replicas.

Key words: Methods, wood charcoal, charcoal replicas, epoxy, archeological woods, sample preparation.

INTRODUCTION

Recovering macrobotanical remains from archeological sites generates valuable data on the past relationships between populations of humans and plants in the Pacific (Fankhauser 1986; Weisler & Murakami 1991; Hather & Kirch 1991; Hather 1991, 1992; Kolb & Murakami 1994; Allen & Murakami 1999) and elsewhere. The initial sorting of samples with geological sieves into size fractions, usually greater than 2.0 mm, is common practice among paleoethnobotantists (Di Piazza 1998; Hastorf 1999; Orliac 2000; Pearsall 2000). Difficulties often arise when working with size fractions less than 2.0 mm. The information available about plant type from small-size fractions is often more limited than from larger-size fractions; but it can still be of value (Guarino & Sciarrillo 2004). Interpreting information must always be tempered by consideration of biases: depositional bias (what enters into the site in the first place), preservation bias (which deposited materials survive), and recovery bias (what comes out of the site) (Wagner 1982; Pearsall 2000; Piqué & Barcelo 2002; Scheel-Ybert 2002; Ferguson 2005; Lepofsky & Lertzman 2005).

During the charring and related taphonomic processes, many features used for identifying taxa are altered or distorted (Pearsall 2000; Ferguson 2005), which has led to

laboratory studies of the charring processes and to controlled productions of charcoal reference collections (Hather 1991; Guarino & Sciarrillo 2004; Orvis *et al*. 2005). The taxonomic identification of charcoal can be facilitated by embedding these burnt remains in resin or hand fracturing them (Smith & Gannon 1973; Leney & Casteel 1975; Pearsall 2000). However, both of these methods have some problems (Angeles 2001), including the reflective nature of fractured surfaces, which affects observations made using light microscopy. Scanning electron microscopy (SEM), on the other hand, produces clear images but is costly. Additionally, if thin sections are not used, long air evacuation times in SEM further add to the operating costs. Resin imbedding procedures alter the samples and take a total of 10 days until complete solidification occurs (Angeles 2001). A new technique using correction fluid, which is commonly applied to paper to mask errors in text, to observe vessel grouping and distribution, and indirect replicas of nail polish to observe intervessel pit apertures and ray size produced good results with a light microscope (Angeles 2001). This paper presents improvements to this method through the use of dental impression medium and gives suggestions for handling small-sized charcoal samples.

The purpose of this study was to develop an efficient means of producing thin, flat, hand fractured charcoal specimens ready for microscopy and the production of replicas. We hand fractured charcoal specimens produced under laboratory conditions having a thickness of 0.7 mm and a maximum diameter of 3.7 mm. The procedure is easily replicated with reusable and inexpensive materials. Replicates of the fractured charcoal were made from vinyl polysiloxane bite registration creme (Exabite™ II NDS used in the field of dentistry).

MATERIALS AND METHODS

Charcoal production

The charcoals used in this study were produced by wrapping fresh woody and herbaceous specimens in aluminum foil and charring them sequentially in a barbeque grill or muffle furnace. A total of 18 charcoal specimens were prepared for examination (Table 1). The taxa selected for charring were ones similar to those found in earth oven features on Rapa Nui (Orliac 2000; manuscript in preparation) and were selected to test a range of plant tissues. In the barbeque grill, all samples were buried under the bed of hot brickets for approximately three hours; in the muffle furnace, samples were heated at 300 °C until the production of smoke ceased. The time needed to complete charring in the muffle furnace was variable (Orvis *et al*. 2005; Pearsall 2000).

Preparing charcoal for SEM stubs

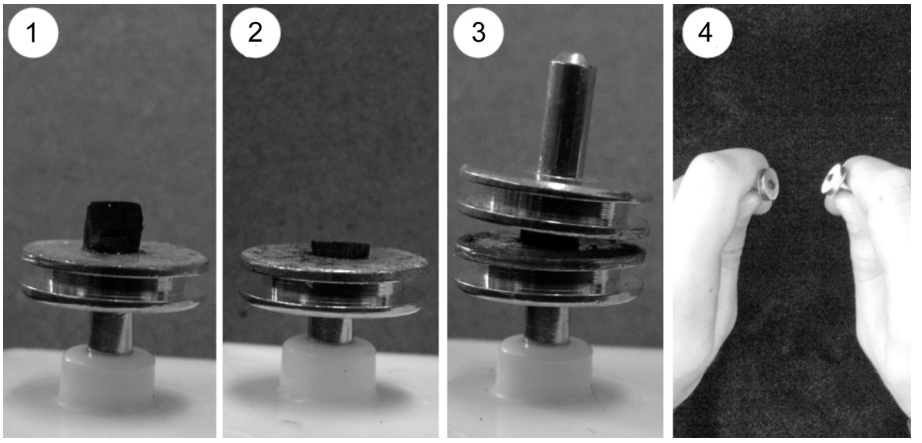
Charcoal samples were prepared by removing a small piece from the original specimen with a fine-toothed jeweler's saw (blade from Ted Pella, Inc., product #54472). A surface on each sample was scraped with a razor, rubbed with fine-grit sandpaper, or both, to flatten one side. Afterwards, air was blown onto the flattened surface of each sample with a reusable, compressed air computer duster to remove the remaining particles that may prevent adhesion to the surface of the SEM stub specimen mount (Ted Pella, Inc., product #16111).

Table 1. Charcoal specimens produced by controlled charring in a barbeque grill and in a muffle furnace. Nomenclature follows the Missouri Botanical Garden TROPICOS database (2010; http://www.tropicos.org/).

Taxa	Family	Plant part	Section		
			Cross	Tangential	Radial
Eucalyptus citriodora Hook.	Myrtaceae	stem	x	x	x
Melia azedarach L.	Meliaceae	stem	x	x	x
*Pittosporum tobira (*Thunb.) W.T. Aiton	Pittosporaceae	stem	x	x	x
Psidium cattleianum Sabine	Myrtaceae	stem	x	x	x
Saccharum officinarum L.	Poaceae	stem	x		
Cordyline fruticosa (L.) A. Chev.	Asparagaceae	stem; rhizome	x x		
Ipomoea batatas (L.) Lam.	Convolvulaceae	tuber	x		
Colocasia esculenta (L.) Schott	Araceae	corm	x		
Curcuma longa L.	Zingiberaceae	rhizome	x		

Preparing SEM stubs for charcoal

SEM stubs were prepared by roughing the top surface with 100-grit sandpaper and then rinsing in absolute ethanol. After the ethanol was blotted dry with a Kimwipes® and allowed to evaporate completely, a thick layer of "5 minute epoxy gel" (ITW Performance Polymers of Riviera Beach, Florida, part number 46409/20845; http://www.itwconsumer.com/catalog.aspx?prodID=117) was applied to the top surface of



Figures 1–4. Abbreviated stepwise method for the simplified procedure for hand fracturing small macrocharocoal remains. – 1: Adhere the flattened charcoal specimen to the thin layer of epoxy on roughed SEM stub. – 2: With small washer to serve as a cutting guide, use a fine-toothed jeweler's saw to cut the charcoal specimen to the standard thickness of the washer. – 3: Adhere another SEM stub applied with epoxy applied on top of the thin sample of charcoal, making sure the epoxy does not run down the sides of the thin section. – 4: After the minimum curing time of the epoxy (~ 24 hours), hand fracture the charcoal specimen by holding the base of the SEM stub pins like handles.

each sample. Excess epoxy was removed with a small dowel applicator. This procedure leaves a thin film of epoxy within the groves produced by the sandpaper.

Mounting charcoal on a SEM stub

The flattened surface of a charcoal specimen was placed onto the thin film of epoxy remaining on its stub (Fig. 1). After 5 minutes, a small washer with the thickness of 0.7 mm and an inside diameter of 3.7 mm, was placed around the specimen to serve as a cutting guide. Then, with the fine-toothed jeweler's saw, the charcoal sample was cut to the standard thickness of 0.7 mm (Fig. 2). The surface of the charcoal sample that remained attached to the SEM stub was examined under a Zeiss binocular dissecting microscope to ensure that a flat surface had been produced. In most cases the fine-toothed saw produced an adequately flat surface. Occasionally, irregularities occurred. When this happened, the specimen was further flattened by lightly scraping with a razor blade and, subsequently, the prepared surface was cleaned with a blast of air to remove the remaining charcoal particles.

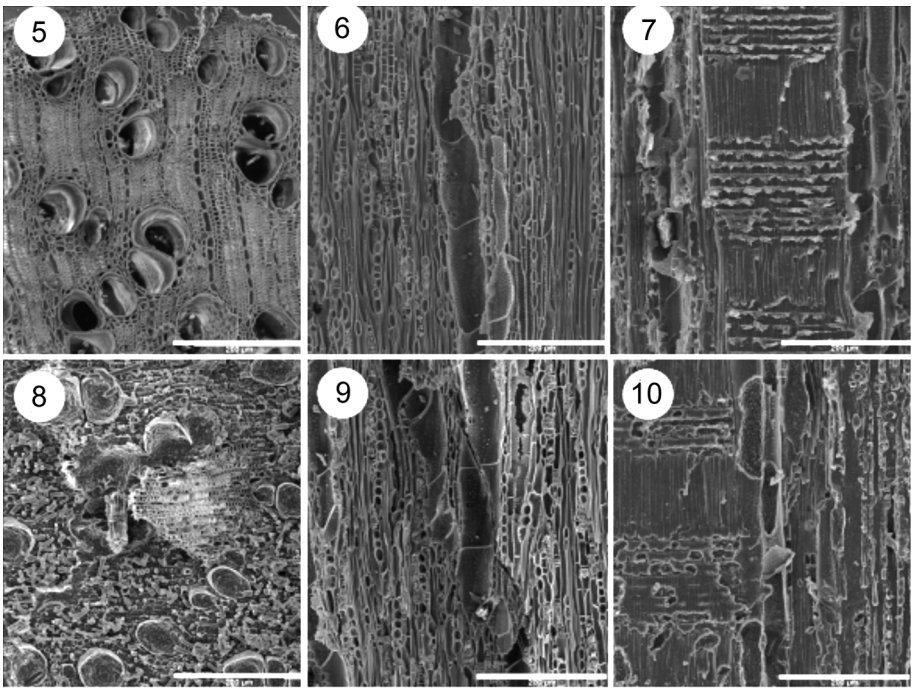
Hand snapping charcoal with SEM stubs

Another SEM stub was prepared and applied with epoxy, as previously described (see *Preparing SEM stubs for charcoal*). This stub was then placed on top of the thin sample of charcoal, making sure the epoxy did not run down the sides of the thin section (Fig. 3). Once the epoxy was allowed to cure completely, for a minimum of 24 hours, the specimen was hand fractured by holding the base of the SEM stub pins as handles (Fig. 4).

After the prepared specimens were snapped, the SEM stubs and attached charcoal were cleaned by sonication in 95% ethanol. In some cases, the excess epoxy around the edges of the stub loosened, but in no case was the charcoal damaged or released from the stub. The fractured charcoal surface was observed under a Zeiss binocular dissecting microscope and a Hitachi S-800 field emission scanning electron microscope. Samples were stored in SEM specimen storage boxes (Ted Pella, Inc., product #16718) labeled with the Latin binomial and plane of section included in each box for replica production.

Preparation of replicas

Replicas were prepared using dental impression medium (Exabite™ II NDS; http://www.gcamerica.com/gcxbite2.html), as previously reported by Angeles (2001). To produce the replica, a quantity of impression medium sufficient to cover the sample was placed onto a glass microscope slide. This step created a flat surface which aided the attachment of the replica to the top of the SEM stub. A snapped charcoal specimen, prepared as described above (see *Hand snapping charcoal with SEM stubs*), was then placed onto the unhardened dental impression medium with light pressure to ensure the entire surface was embedded with material. The impression medium was allowed to cure completely before removal with forceps from the charcoal specimen and the glass backing. Afterwards, the replica was sonicated in 95% EtOH. The clean replica was adhered to a pre-sonicated SEM stub using carbon conductive sheets cut to fit the



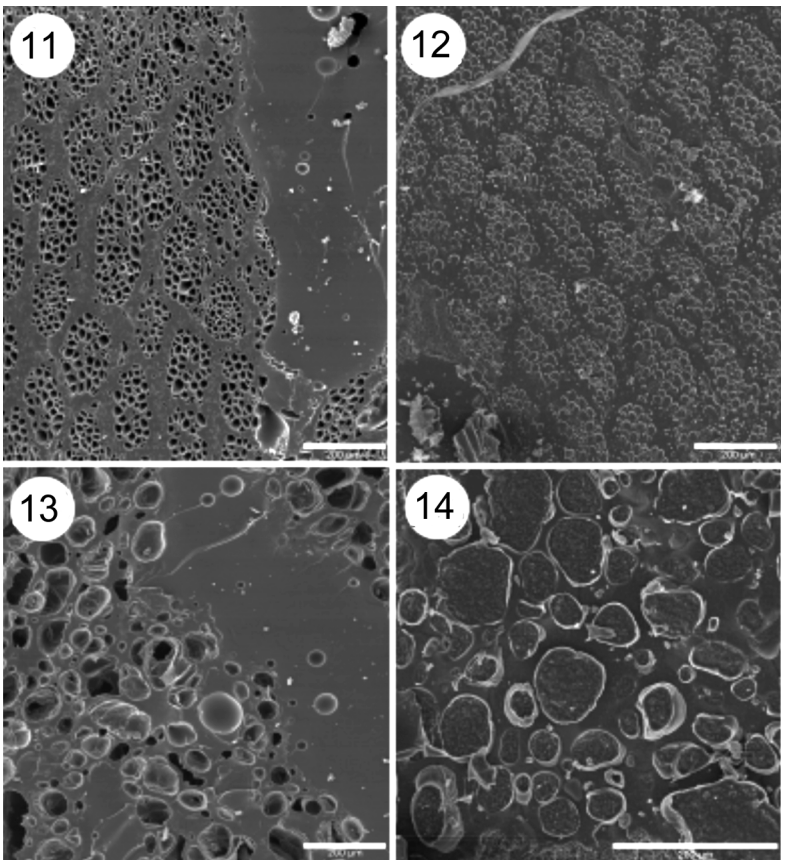
Figures 5–10. *Eucalyptus citriodora* Hook. – 5: Charred cross section. – 6: Charred tangential section. – 7: Charred radial section. – 8: Replica cross section. – 9: Replica tangential section. – 10: Replica radial section. — Scale bars = 200 μm.

SEM stub (Ted Pella, Inc., product #16085-1). Replica surfaces were observed using a Hitachi S-800 field emission scanning electron microscope at the Pacific Biomedical Research Center at the University of Hawai'i at Mānoa.

RESULTS

Among the 18 charcoal sections prepared (Table 1), the anatomical features of *Eucalyptus citriodora* Hook., *Melia azedarach* L., and *Pittosporum tobira* Dryand. were clearly visible in all planes (Fig. 5–10). Unfortunately, *Psidium cattleianum* Sabine detached from one of the SEM stubs rather than fracturing through the specimen, possibly due to the high density of the wood charcoal. In the cross section of the *Cordyline fruticosa* (L.) A.Chev, the secondary amphivasal vascular bundles, composed of centrally located phloem surrounded by xylem as described in Mauseth (1988) were clearly visible (Fig. 11 & 12). The atactostele in *Saccharum officinarum* L. was well preserved and clearly visible. Even though the sections of *Ipomoea batatas* (L.) Lam. and *Colocasia esculenta* (L.) Schott were mostly composed of isodiametric parenchymatous cells, regions showing key anatomical traits were visible (Fig. 13 & 14).

In a few instances, the epoxy penetrated deeply into or around the specimens. As a result, the duration of gas pump down times on the SEM were decreased by the epoxy



Figures 11–14. Cross sections of *Cordyline fruticosa* (L.) A.Chev. and *Ipomoea batatas* (L.) Lam. – 11: Charred aerial rhizome of *C. fruticosa*. – 12: Replica aerial rhizome of *C. fruticosa*. – 13: Charred cross section of *I. batatas*. – 14: Replica cross section of *I. batatas*. — Scale bars = 200 μm.

infiltrating the thin specimens. The epoxy surfaces are readily distinguishable because of their homogeneous nature (Fig. 11 & 13).

Overall, the replicas of each species sampled consistently revealed almost identical cell shape and distribution as observed in the original fractured charcoal specimen. In some instances a very thin, almost translucent layer of charcoal remained on the replicas (Fig. 8 & 9). *Curcuma longa* L. was fractured very unevenly, making clear anatomical traits difficult to distinguish.

The majority of the specimens prepared for this study had regions where the anatomical details required for identification could be observed. However, the overall quality of the specimen varied with hardness of the charcoal. No instrumentation was used that could quantitatively measure the evenness of a surface; as a result, the following observations were made:

– More homogeneous charcoal produced the smoothest viewing surfaces;
– Charcoal with pronounced differences between hard and soft regions broke less evenly and often separated along the natural plane of weakness, instead of fracturing through the thin charcoal specimen;
– If the charcoal is homogeneous, then replicas consistently show almost identical cell shape and distribution as the original fractured charcoal specimen.

DISCUSSION

In a pilot study consisting of trial-and-error attempts to optimize this technique, 5-minute and 30-minute set time epoxies were tested. The 30-minute epoxy gave excellent results with hard, wood charcoal; however, soft, porous charcoal was often completely saturated. When the saturated charcoal was hand fractured, the sample regularly broke free from one of the two SEM stubs rather than producing a clean fracture through the specimen. Consequently, we opted for using high strength epoxy with a 5 minute set time to give more consistent results for a range of charcoal samples.

Additionally, after charcoal samples are fractured, the reliability of the replica to show almost identical cell shape and distribution as that of the original fractured charcoal is enhanced when the charcoal is evaluated for clarity of anatomical features before applying dental impression medium. Replicas should be a vital source added to herbaria and archeological collections. Moreover, investigations of the effects of different water content prior to charring and different charring temperatures may show which conditions are best for producing charcoal that show clear anatomical features.

Techniques designed to alleviate some of the difficulties of charcoal processing should make macrocharcoal data accessible to more scientists, thus expanding the importance of paleoethnobotany in education and research (Mehrotra 2005). With the amount of botanical remains recovered from archeological assemblages increasing in recent years, new methods and ideas will allow the trend to continue. Accordingly, scholars trained in multiple disciplines must attempt to provide a coherent synthetic theory linking culture and botany (Ford 1988; Wilson 1998, 2006).

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