METHODS FOR THE STUDY OF SOIL CHARCOAL AS AN INDICATOR OF FIRE AND FOREST HISTORY IN THE APPALACHIAN REGION, U.S.A.

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Abstract—Charcoal particles in soils and sediments of the Appalachian region provide evidence of long-term fire history relevant to resource management and to studies of paleoclimate, vegetation history, and the effects of prehistoric and historic humans on the environment. Charcoal records of fire history are of low resolution in comparison to dendrochronological records, but reach well beyond the oldest trees in most areas, providing evidence of fires thousands or tens of thousands of years ago. We focus here on fire history reconstruction from soil charcoal, which provides site-specific evidence of past fires and potentially forest composition. Charcoal > 2 mm may be large enough for taxonomic identification, and of sufficient mass to enable AMS radiocarbon dating of individual particles. Soil mixing due to physical and biological factors creates soil profiles in the southern Appalachians in which charcoal age is not predictable from charcoal depth; soil charcoal records thus require many radiocarbon dates.

INTRODUCTION

The incomplete combustion of plant material during vegetation fires produces charcoal fragments of various sizes. Some of these fragments are incorporated into soils of the burn site, while other fragments are carried away by wind or water, in some cases to later settle on the surface of a lake or wetland. Researchers study charcoal in soils and in sediments of lakes and wetlands at sites around the world to document past fires and to understand long-term relationships between fire, climate, and human activity (Berg and Anderson 2006, Hart and others 2008, Horn and others 2000, League and Horn 2000, Sanford and Horn 2000, Whitlock and Larsen 2001). We focus here on the analysis of macroscopic charcoal in soils of the Appalachian region as an indicator of fire and forest history. The records of past fires that can be obtained from the study of soil or sediment charcoal are coarse (low resolution) in comparison to fire histories developed from dendrochronological analyses of fire-scarred trees, which permit the identification of exact fire years (Flatley and others 2013, Lafon and others 2014). However, the evidence of fire provided by charcoal studies reaches well beyond the oldest trees and tree-ring records in the Appalachian Mountains, providing evidence of fire thousands or even tens of thousands of years ago. For periods of time prior to about 300 years ago, charcoal in soils and sediments is the only evidence we have of wildland fires in the Appalachian region.

Interest is growing in soil charcoal as a proxy for fire history in the Appalachian region. Welch (1999) examined macroscopic charcoal in forests dominated by yellow pines on the Cumberland Plateau. She looked only at the presence or absence of macroscopic charcoal in soil increments, and did not obtain radiocarbon dates. The presence of charcoal in 85 percent of the samples from seven sites documented the importance of fire. Hart and others (2008) quantified macroscopic charcoal in soils of mixed hardwood forests on the Cumberland Plateau. Radiocarbon dates were obtained on five charcoal samples, and several charcoal samples were identified to be from trees with diffuse porous growth rings, possibly maple, beech, or tulip poplar. The weighted means of the calibrated probability distributions of the five radiocarbon dates ranged from 6735 to 174 cal yr BP (calibrated years before present). No overlap occurred within the 2-sigma calibrated age ranges of the dated charcoal samples, indicating a minimum of five unique fire events.

Fesenmyer and Christensen (2010) reconstructed a stand-level fire history in the Nantahala National Forest in western North Carolina from soil charcoal. They sampled a broad array of forest types that included pine forest, xeric-oak hardwood forest, and mesic cove forest. The median probabilities of the calibrated ages of 81 soil charcoal fragments ranged from 4000 to 0 cal yr BP, with one sample returning a date of 10,570 cal yr BP. The prevalence of charcoal from 4000 to 0 cal yr BP demonstrated that fires occurred regularly in the study area during the Late Holocene.

Our soil charcoal work in the Appalachian region has focused on pine and mixed forests in eastern Tennessee and western North Carolina, mainly within Great Smoky Mountains National Park. Within the park we have quantified, identified, and dated charcoal fragments in Table Mountain pine stands and in other stands

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historically dominated by lower-elevation yellow pine trees (Underwood 2013) to complement and extend analyses of fire-scarred trees in the same study sites (Lafon and others 2014). We have also examined charcoal in soils surrounding a wetland in which we have examined sedimentary charcoal.

CHARCOAL TAPHONOMY AND SOIL CHARCOAL COMPARED TO OTHER EVIDENCE OF PAST FIRE

To use charcoal in soils or sediments as an indicator of past fire requires knowledge of the processes that create, move, and preserve charcoal in different environments (Scott and Dablon 2010). Many of these processes are not yet completely understood, but they are attracting increasing research attention as charcoal in soils is part of the carbon pool and of interest from the perspective of carbon sequestration and cycling (Licata and Sanford 2012, Massielo and Louchouarn 2013), as well as for its value as a paleoenvironmental indicator. The increased use of prescribed fire in Appalachian forests provides an opportunity to test ideas about the production and fate of charcoal in fires. Recent work by Scales (2011) on charcoal distribution following a prescribed fire in Virginia provides a good example of what can be learned from post-fire sampling of charcoal in burn sites.

Although our focus here is large (≥ 2 mm) macroscopic charcoal in soils, consideration of the fate of smaller particles during and after burns provides context for comparing soil charcoal evidence to evidence from studies of charcoal in sediment profiles in lakes and wetlands, which focus on smaller charcoal particles. Studies of charcoal in sediment cores include studies of microscopic charcoal in pollen preparations, sometimes called pollen-slide charcoal, and of larger particles that are sieved from sediments. Charcoal on microscope slides prepared for pollen analysis is generally < 125 or 180 μm, as sieves of this size are commonly used to concentrate pollen. Charcoal that is extracted from sediment cores by sieving is usually ≥ 125 μm, as this is typically the smallest sieve size used in such studies, which often employ nested sieves of 125, 250, and 500 μm, for example (Whitlock and Larsen, 2001). During fires, microscopic charcoal and small particles of macroscopic charcoal are lofted into the air by convection currents. Once aloft, the smallest of these particles may be carried tens of kilometers or more away from the fire. Many charcoal particles present on microscope slides prepared for pollen analysis are < 50 μm and may derive from regional sources (Clark 1988, Whitlock and Larsen 2001), such that their variable abundance through a lake core may reflect the history of burning not just in the lake watershed, but also in a large area upwind from the lake.

In contrast, macroscopic charcoal particles sieved from lake sediments provide evidence of fires within the lake watershed or nearby. If samples for charcoal analysis are taken at a high sampling density, for example in contiguous 1-cm intervals, detailed fire history records can be developed. Depending on the sedimentation rate and the frequency of fires, peaks in charcoal (sediment levels with high charcoal abundance) may represent single past fires, or periods of high fire activity (Whitlock and Larsen 2001). Microscopic charcoal is rarely sampled contiguously in cores, so records can miss fires; thus these records are of lower temporal and broader spatial resolution than macroscopic charcoal records from lake sediments. For both microscopic and macroscopic charcoal records from sediment cores, the ages of charcoal peaks can be estimated from radiocarbon or other dates on bracketing sections of the core. For example, a peak in charcoal half-way between two dated horizons could be interpreted to have an intermediate age. In sediment core studies, radiocarbon dates are sometimes obtained for individual pieces of charcoal, but often dates are obtained for uncharred macrofossils, pooled charred or uncharred organic material, or bulk sediment.

Charcoal that is not blown or washed out of burn sites becomes incorporated in soil horizons by various processes of mixing, or is buried by geomorphic processes. Radiocarbon analyses have demonstrated that charcoal in modern soils and paleosols (buried soils) can persist for thousands to tens of thousands of years, in some cases to beyond the limit of radiocarbon dating, which for typical samples is around 45,000 years ago. In studies of soil and sediment charcoal, a fragment that is too old for radiocarbon dating will have a reported "date" with a greater-than symbol. For example, charcoal in a paleosol in Costa Rica returned a date of > 43,630 years (Driese and others 2007). However, most charcoal particles do not persist this long; if they did, we would be wading through snow-drift like piles of charcoal in fire-prone forests of the world, and soils in these and other frequently burned ecosystems would be black in color.[3] Recently Jaffé and others (2013) demonstrated that charcoal may account for a large proportion of dissolved organic carbon in ecosystems, and suggested that the persistence of charcoal fragments in soils may depend on the material burned and charring temperature, as well as on soil conditions and fauna. These findings deserve more study in the analysis of soil charcoal as they may function as a filter on the information we can obtain from such studies.

[3]We credit the idea that fire-prone forests would harbor snow-drift like piles of charcoal, if charcoal was resistant to breakdown, to our colleague Ken Orvis. R. Jaffé was quoted as saying that soils would be black if charcoal in soils was resistant to breakdown in a press release from Florida International University posted by J. Adkins upon publication of the article by Jaffé and others referenced.

Although large charcoal particles can be moved short distances downslope by gravity or overland flow, most of the large charcoal in soils located away from stream courses and floodplains likely reflects the burning of vegetation at or very near the sampling location. Like fire scars on standing trees, charcoal in soils provides evidence of past fire that is highly site-specific, more so than macroscopic charcoal records from lake or wetland sediments, which can document fires within a watershed but not on a particular portion of the watershed. Another advantage of studies of soil charcoal is that charcoal fragments are often massive enough that a radiocarbon date can be obtained on an individual fragment. However, because soils are mixed by biological and geomorphic activity, the age of charcoal cannot be reliably estimated from dates on other charcoal particles in the soil profile.

FIELD METHODS

Researchers obtain soil charcoal samples from the walls of excavations or road cuts, or by collecting soil cores. The number of sites to sample, and the number and arrangement of pits or cores at each site, depends on the specific aims of the research. Collecting cores rather than digging pits in the field offers time efficiency, the ability to work in light rain, and less environmental disturbance, but it is not feasible if the volume of contiguous soil material to be examined is greater than the volume that can be obtained in a single core (e.g., Di Pasquale and others 2008). We favor a cylindrical soil-coring device manufactured by Eijkelkamp© (http://en.eijkelkamp.com/) that allows the collection of successive samples in 10-cm increments to a depth of 1 m or the depth of refusal (Horn and others 1994; fig. 1). The device we use, known as a "single root auger," was developed for use in studies of root growth and was first used for soil charcoal research by Sanford and others (1985) in the Venezuelan Amazon. Despite the name of the device, it does not have a helical shaft commonly associated with augers, but a simple 8-cm diameter cylindrical bucket that allows recovery of soil increments without mixing. The device is pushed down while rotating to collect samples in successive 10-cm increments (0–10 cm, 10–20 cm, and so forth), or 5-cm increments in soils that are difficult to core. Quart-size, zipper-top plastic bags are a convenient size for holding 10-cm soil increments after extrusion from the corer, which can be accomplished by inverting the corer and stepping on the handle.

LABORATORY METHODS

Upon return to the laboratory, charcoal particles are separated from soil by wet-sieving. Soaking samples in water overnight facilitates the sieving process. A simple way to soak the samples is to add water to the plastic bags in which you collected them, and then very gently knead the bag to initiate the disaggregation of the soil sample. Two-pound plastic coffee cans make ideal containers to hold sample bags with water added (one bag per can); the cans keep the bags from falling over and spilling their contents on lab counters and contain leaks if they occur.

In our work in the Appalachian region, we have not found it necessary to use chemical pretreatments before sieving charcoal. Some researchers have used dispersants such as sodium hexametaphosphate to make sieving easier (e.g., Titiz and Sanford 2007). Because of the desirability of obtaining radiocarbon dates on charcoal particles, we recommend against the use of water softeners or dispersants sold for home use, as these may include carbon-containing compounds that might be absorbed by charcoal particles and affect radiocarbon analyses.

Following disaggregation, we wet-sieve soil samples for charcoal analysis using 8-inch diameter sieves with openings of 2 mm. We have selected this sieve size because it will capture particles large enough that it is possible to obtain a radiocarbon date on the individual piece of charcoal, and because fragments of charcoal of this size are potentially identifiable to species or genus. Smaller mesh sizes may be appropriate if large charcoal is sparse and the documentation of the presence or absence of smaller particles is important in the study design. We sieve our samples by holding the sieves under a tap-water faucet. Care should be taken that the force of the flowing water is not so strong that it breaks charcoal or pushes sediment or charcoal over the edge of the sieve. If many samples are to be sieved, the person doing the sieving will be more comfortable if supports are used to hold the sieve. A dishpan should be placed under the sieve to catch heavier sediment to avoid clogging sink drains or filling sink traps.

Charcoal can be distinguished from other materials retained on sieves by its dark black color and sheen and by the way it will usually fracture if a dissecting needle is pressed against it. Dry charcoal will usually leave a streak when gently rubbed on paper, but this test is not recommended if you plan to obtain a radiocarbon date on a specimen, as paper fibers may be transferred to the charcoal that can affect the date obtained. Charcoal can be picked from the surface of the sieve using fine forceps (place wet sieve on a plate or tray to catch drips), or the sieve and its contents can be inverted onto a dish for sorting. A dissecting scope, visor with magnifying lenses, or magnifying light make this task easier. We wash charcoal particles with distilled water before placing them in vials, aiming to remove as much loose soil as possible. We use 20-mL glass scintillation vials that we first treat in a furnace at 550 °C for 1 hour to burn off any organic contaminants. After the scintillation vials have fully cooled, they can be labeled with black marking

pens. We use Sharpie®-brand permanent markers with fine points for labeling the sides of glass vials. Extra-fine point markers are good for labeling vial lids, but do not use them on glass vials as the ink will be too thin and will fade over time. We then dry particles at 90 °C overnight in a laboratory oven and subsequently weigh them. We recommend quantifying macroscopic soil charcoal by dry mass.

Following separation from soils and drying, charcoal particles are selected for radiocarbon dating. The material dated depends on the research question. We generally favor getting dates on individual charcoal particles. Hammond and others (2006) obtained radiocarbon dates on randomly selected particles. We have dated from different horizons, with the assumption that different horizons might be of different ages, but from our own work and that of Fesenmyer and Christensen (2010) we now know that depth in the soil profile is a poor predictor of the age of charcoal in soils of Appalachian forests (fig. 2). This is a consequence of the mixing of soil due to various physical and biological factors. Unlike lake sediment sequences, in which ages of charcoal or other components can be estimated from radiocarbon dates that bracket the materials, determining the ages of past fires based on charcoal in Appalachian soils require the dating of large numbers of individual charcoal particles.

Researchers working with soil charcoal obtain radiocarbon dates by submitting samples to one of several private and university laboratories. The standard price for radiocarbon dates obtained using the AMS (accelerator mass spectrometry) method is $500–$600/date. AMS ^{14}C dating allows the dating of individual charcoal fragments with masses of 5–10 mg and sometimes less, depending on the final C in the sample. The charge for AMS radiocarbon analysis includes determination of the ratio of the stable isotopes of ^{13}C and ^{12}C, used to correct for natural isotopic fractionation. Some laboratories offer discounts to researchers with funding from the National Science Foundation or other Federal agencies: ask!

Radiocarbon ages can be converted to estimates of calibrated calendar years using one of several calibration programs. The CALIB program developed by Stuiver and Reimer (1993) has gone through several updates; the latest version is available on the Internet (http://calib.qub.ac.uk/calib/) and can be downloaded for free or used online to determine calibrated age ranges. Researchers generally report the 2-sigma calibrated age range of samples, in cal yr BP, or in cal yr CE or BCE (equivalent to AD/BC; with 0 cal yr BP equal to AD or CE 1950). There is a 95 percent chance that the true age of the charcoal particle falls within this range. Researchers also typically report a point estimate of the calibrated date, such as the weighted mean of the probability distribution function, or the median (Telford and others 2004). It is important to remember that the date is really not a single value but a probability range. Also, the radiocarbon date reflects the time that the carbon in the plant tissue was fixed by the tree, and not the date of the fire event. The time gap between carbon fixation and the occurrence of a fire is called “inbuilt age” and must be taken into account when compiling fire histories from charcoal (Gavin 2001). In the southern Appalachian region, the estimated inbuilt age is between 50 and 100 years (Fesenmyer and Christensen 2010). The inbuilt-age error needs to be added to the calibrated age range to provide a more realistic range of time during which the fire occurred. For example, if the 2-sigma calibrated age range is 610–470 cal yr BP, adding the maximum estimated inbuilt-age error above would widen this range to 710–470 cal yr BP.

Where possible, the morphological identification of macroscopic charcoal from soils provides an opportunity to identify the type of tree or shrub that burned in the fire. Charcoal fragments must be identified before dating, as the process of radiocarbon analysis is destructive. Charcoal fragments selected for morphological identification should be cut transversely with a razor blade to analyze wood anatomy. Do not use razor blades that are coated with Teflon® or rust-inhibiting oil, as these substances could add carbon contamination to the charcoal sample, and be sure to rinse the blade thoroughly with distilled water before subsequent charcoal fragments are cut. Once a clean, transverse cut has been made, identification is based on the presence and characteristics of anatomical features such as tracheids, resin canals, tyloses, rays, and growth-ring boundaries (fig. 3). Morphological identification of charcoal is best accomplished using reference specimens prepared from plant samples. A variety of methods can be used; we have created reference collections in our lab by igniting samples in crucibles in a muffle furnace (Orvis and others 2005). We have also developed collections by gathering specimens from charred trees in recent burn sites. Charcoal samples can also be identified through comparison with photographs and descriptions in various references on woody anatomy (e.g., Hoadley 1990).

CONCLUSION

Examining soil charcoal in Appalachian forests provides a way to document fires that occurred long before the first written records or tree-ring chronologies. Soil charcoal records are coarse in comparison to dendrochronological records of fire and, because of soil mixing, require many more radiocarbon dates than charcoal records from lake and wetland sediments. However, soil charcoal records provide site-specific evidence of fire that can be useful for forest management and for understanding the long-term development of forest stands. The taxonomic identification

of dated charcoal particles provides a way to reconstruct the vegetation that burned as well as the timing of past fires. Soil charcoal studies can also contribute to a better understanding of the carbon cycle and of the role of carbonized wood as a charcoal sink in Appalachian forests.

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Figure 1—Using the single root auger to collect soil charcoal in Great Smoky Mountains National Park. The photo on the right shows the extrusion of a 10-cm core increment into a labeled plastic bag. (Photos by Matthew Valente)

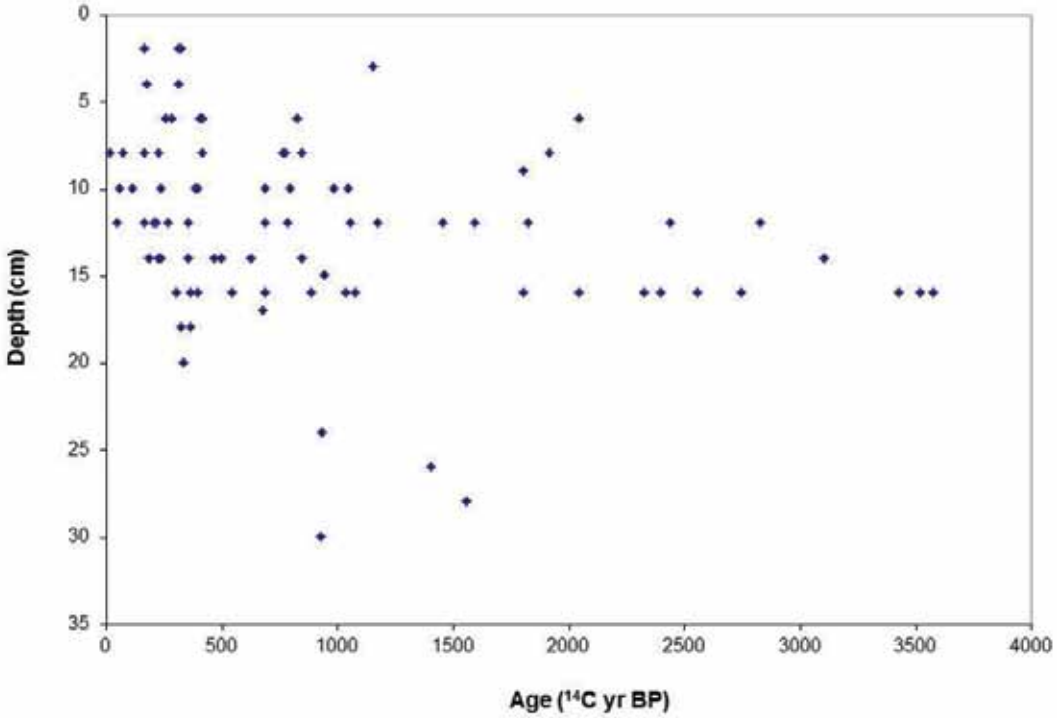

Figure 2—Radiocarbon ages of 81 charcoal fragments plotted against depth of recovery in Nantahala National Forest, from the study of Fesenmyer and Christensen (2010); data available at http://www.esapubs.org/archive/ecol/E091/049/appendix-A.htm. [Date accessed: February 1, 2011].

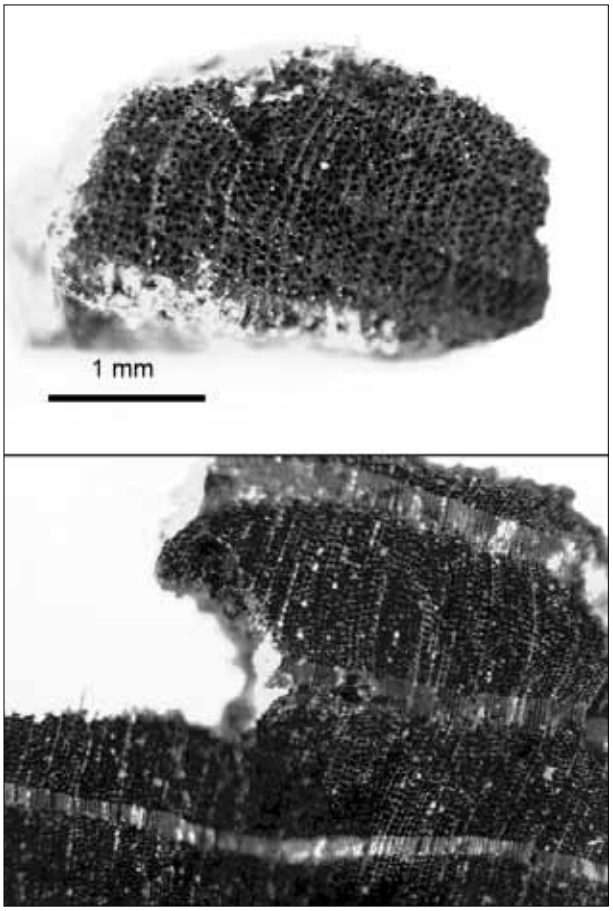

Figure 3—Macroscopic charcoal from study sites in Great Smoky Mountains National Park. The upper photograph shows charcoal from red maple (*Acer rubrum* L.). Anatomical features that allow identification of red maple are narrow rays that are approximately the same width as the widest pores, absence of tyloses, and diffuse-porous wood. The lower photograph shows charcoal from a southern yellow pine (*Pinus*, diploxylon group). Anatomical features that allow identification of southern yellow pine are tracheids, resin canals, and pronounced earlywood to latewood transition at ring boundaries. The wood anatomy of the diploxylon pines that grow in the southern Appalachians is too similar to allow differentiation of charcoal specimens to species. The scale bar is approximate and is for both photographs. (Photos by Chris Underwood)